Advanced Ion Microscopy Focus Topic
Room 7 & 8 - Session HI-WeA

Emerging Ion Sources and Optics

Moderator: John A. Notte, Carl Zeiss Microscopy

2:20pm **HI-WeA-1 COLDFIB – The New FIB Source from Laser Cooled Atoms**, *E Verzeroli,* ***Anne Delobbe****, M Viteau,* Orsay Physics, France*; D Comparat,* CNRS Lac Orsay, France*; A Houel, M Reveillard,* Orsay Physics, France **INVITED**

Charged particle beams of controlled energy and strong focusing are widely used tools in industry and science. Focused Ion Beam (FIB) column combine with a Scanning Electron Microscope (SEM) provide full control of nanofabrication or nanolithography processes. Ion energy can be varied typically in the 1–30KeV range, with an energy-dependent resolution attaining the nanometer range. State-of-the-art FIBs commercially available are based mainly on plasma, liquid metal tip or helium ion sources for large, intermediate, and low currents, respectively. Despite the very high technological level of the available machines, research of new ion sources allowing even higher resolution and a wider choice of atomic or molecular ions for new and demanding application is very active.

As an example, the world of electronic components evolves regularly towards the miniaturization by integrating a number of transistors more and more important. The dimensions being smaller and smaller (technology 10 nm, 7 nm even 5 nm), nowadays the instruments of analysis used, like the conventional FIB, reach their limit. Thus it's necessary to realize a technological breakthrough to be able to observe, analyze and modify components and structures on the scale of the nanometer.

Our new system, COLDFIB, wants to take up this challenge of the nanomanufacturing by the coupling of two high technologies: the laser cooling of atoms, and manipulation of charged particles.

Very innovative, this industrial solution, based on a source of ions obtained from atoms laser cooled and ionized, will allow realizing ions beam in the unequalled performances, to reach engraving's sizes of some nanometers. This new technology offers a resolution, for example at 5KeV, 10 times better than the LMIS one, and reaches the nanometer at 30keV *(Figure 1)*.

We'll present in this talk the integration on the SEM-FIB TESCAN instrument. In addition to the experimental[1] part and performances[2] will also show some first applications.

[1] L. Kime, et al., *High-flux monochromatic ion and electron beams based on laser-cooled atoms*, Phys. Rev. A 88, 033424 (2013)

[2] M. Viteau, et al., *Ion microscopy based on laser-cooled cesium atoms*, Ultramicroscopy (2016)

3:00pm **HI-WeA-3 FIB Platform Employing a Low-Temperature Ion Source**, ***Adam Steele****, A Schwarzkopf,* zeroK NanoTech*; J McClelland,* National Institute of Standards and Technology*; B Knuffman,* zeroK NanoTech

We present a demonstration of a new high-performance ion source retrofitted to a commercial FIB platform. Spot sizes as small as (2.1 ± 0.2) nm (one standard deviation) are observed with a 10 keV, 1.0 pA beam. Brightness values as high as (2.4 ± 0.1) × 10^7 A m^{-2} sr^{-1} eV^{-1} are observed near 8 pA [1]. The measured peak brightness is over 24 times higher than the highest brightness observed in a Ga liquid metal ion source (LMIS); the spot size obtained by operating our source at 10keV is significantly smaller than the spot size achievable with the replaced LMIS operating at 40 keV.

The FIB platform utilizes a Low Temperature Ion Source (LoTIS). As previously described [2], this source is composed of a several discrete stages that collect, compress, cool and finally photoionize a cesium atomic beam. High brightness and small spot sizes are achieved owing to the extremely low (10 uK) temperatures that may be achieved in the neutral atomic beam prior to photoionization. The atomic beam transmits over 5×10^{10} atoms s^{-1}, which would be equivalent to an ion beam with over 8 nA if ionized completely; extraction of currents up to 5 nA have been demonstrated to date.

We will present a description of the Cs^+ LoTIS-FIB system, together with an examination of the brightness and spot size measurement methodology at beam currents up to a nanoampere. Images acquired using the system will also be shown. Finally, we will describe outcomes of some preliminary milling, gas assisted etching and deposition experiments performed with the system.

[1] A. V. Steele, A. Schwarzkopf, J. J. McClelland, and B. Knuffman. *Nano Futures*. **1**, 015XXX (2017). (to be published 5/2017)

[2] B. Knuffman, A. V. Steele, and J. J. McClelland. *J. Appl. Phys*. **114**, 044303 (2013).

3:20pm **HI-WeA-4 Focused Cs Ion Beam Nanomachining and Material Interaction Characterization for Semiconductor Applications**, ***Richard Livengood****, R Hallstein, S Tan,* Intel Corporation, USA*; Y Greenzweig, Y Drezner, A Raveh,* Intel Corporation, Israel*; A Steele, B Knuffman, A Schwarzkopf,* zeroK NanoTech, USA

Focused ion beam Nanomachining is used extensively in semiconductor materials and circuit analysis applications. Applications range from using ion beams for large area machining for de-process sample for metrology and defect analysis, to high precision nanomachining to access device circuits [1,2]. There have been many different focused ion beam technologies developed and refined over the last 30 years to perform this type of machining. The two primary focused beam-source technologies used today are: Gallium Liquid Metal Ion source (LMIS) for micro and nanomachining applications [3,4]; and 2) Xenon plasma-cusp ion sources used for bulk material micro-machining in packages interconnects, TSV's, and backend metal layers [5,6]. More recently, the neon and nitrogen (N2) gas field ion sources (GFIS) have also been introduced to enable very small, high precession Nanomachining for circuit rewiring and mask defect repairs respectively [7,8]. Another emerging ion beam / source technology are cold beams (base on ionization of atoms cooled to sub kelvin temperatures, which gives them very low energy spread) [9]. Two such emerging sources are cesium based cold beam sources under development by ZeroK Nanotech Inc. and TOH (Tescan Orsay-Physics Holdings) [10,11].

As part of Intel's due diligence to identify break through ion beam technologies to keep pace with semiconductor scaling requirements and help identify novel analytical applications, Intel has recently been analyzing the attributes of cesium for semiconductor applications [12]. In this paper, we will discuss the attribute requirements for various semiconductor applications and publish early cesium beam machining performance attributes - based on joint characterization experiments performed by Intel and ZeroK Nanotech on cesium LoTIS focused ion beam using ZeroK's proof of concept test platform. Analysis will include preliminary characterization results for material sputter rates, beam induced etching, and other Nanomachining attributes.

4:20pm **HI-WeA-7 Spectroscopy in the Focused Ion Beam**, ***Robert Hull****,* Rensselaer Polytechnic Institute*; H Parvaneh,* Global Foundries **INVITED**

We review spectroscopic methods in the focused ion beam (FIB), and introduce the coupled Auger Electron Spectroscopy (AES) – FIB technique. While FIB tomography has become a widely-used method for exploring 3D structure of materials over length scales ranging from tens of nm to tens of µm, complementary high resolution and high sensitivity spectroscopic methods are lacking. Secondary ion mass spectroscopy (SIMS) methods are limited by low ionization yields using conventional Ga^+ liquid metal ion source (LMIS) species and/or by low detector transmission factors. The anticipated advantage of coupling AES to the FIB is that Auger electron yields per incident ion can be in the few percent range depending on the experimental conditions, improving on Ga^+ ionization yields by several orders of magnitude for many elements. We have integrated an Orsay Physics Cobra mass-selecting FIB column into a PHI VersaProbe X-Ray Photoelectron Spectroscopy (XPS) system, successfully aligning the focal points of the FIB and of the detector/analyzer optics with the necessary precision in 3D dimensions. Using primary ions with different masses (e.g. from an Au-Si alloy source), we can control the relative proportions of the Auger transitions from the atoms of the target sample rather than from backscattered/implanted atoms from the primary beam. We have studied a set of elemental targets, with strong Auger peaks observed from each. For some elements (e.g. Mg, Al and Si), additional extremely sharp peaks are observed, superimposed on the standard Auger peaks. These are due to Auger emission from atoms that have been sputtered from the surface before the inner shell vacancy is filled. The occurrence of these free atom peaks in a subset of the samples can be understood in terms of the substantially longer vacancy state lifetimes in the core levels of some elements, allowing the target atom to escape from the surface field before

Auger decay happens. For example, we observe average Auger yields of 0.06 for Cr and 0.09 for Al per incident 60 keV Si_2^+ ion. Coupled with the estimated transmission factors and solid angular detection range of the XPS hemispherical analyser employed, this translates into detection of atomic concentrations of order 0.1-1.0 % within a (50 nm)^3 voxel. These figures of merit will be compared (favorably) to other spectroscopic methods available in the FIB.

5:00pm **HI-WeA-9 Spark-discharge Coupled Laser Multicharged Ion Implantation and Deposition System**, ***Md Haider Shaim***, *M Rahman, O Balki, H Elsayed-Ali,* Old Dominion University

Multicharged ions are generated by a Nd:YAG laser (λ = 1064 nm, τ = 7 ns, pulse energy ≤175 mJ) ablation of aluminum and boron targets in an ultrahigh vacuum. Time-of-flight and electrostatic retarding field ion energy analyzers are used to detect the laser-generated ions. Spark discharge coupling to the laser ion source enhances ion generation along with generating higher charge states than observed with the laser source alone. The spark discharge electrodes are located in front of the target and is triggered by the laser plasma. For an Al target with the laser source alone, the total ion charge delivered to a Faraday cup located 1.4 m away from the source is 1.0 nC with charge state up to Al^{3+}. When the spark amplification stage is used (0.1 μF capacitor charged to 5.0 kV), the total charge increases by a factor of ~9 with up to Al^{6+} observed. The spark discharge increases the multicharged ion generation without increasing target ablation, which solely results from the laser pulse. An electrostatic cylindrical ion deflector is used for analysis and selection of charges with a specific energy-to-charge ratio. A three-electrode cylindrical einzel lens is used to focus the ion beam. A minimum ion beam diameter of ~1.5 mm was obtained. A high-voltage pulse applied to a set of two parallel deflecting plates is used for the pickup of ions with different charge states according to their time-of-flight. Fully stripped B ions with 150 eV per charge are obtained with the laser alone. These ions are used for shallow implantation without further acceleration. Al multicharged ion generation from femtosecond laser (λ = 800 nm, τ = 100 fs, pulse energy ≤1 mJ) ablation is also studied. Production of Al ions up to Al^{6+} is observed with the laser alone. Compared to nanosecond laser ablation, multicharged ion generation by femtosecond laser ablation require significantly lower laser fluence and generates higher charge states and more energetic ions.

Tribology Focus Topic
Room 10 - Session TR+AS+HI+NS+SS-WeA

Molecular Origins of Friction

Moderators: J. David Schall, Oakland University, Paul Sheehan, U.S. Naval Research Laboratory

2:20pm **TR+AS+HI+NS+SS-WeA-1 On the Stochastic Nature of Bonding in Contact: Simulations of Indentation and Sliding of DLC Tips on Diamond Surfaces**, ***J. David Schall***, Oakland University*; R Bernal,* University of Texas at Dallas*; Z Miline,* University of Pennsylvania*; P Chen, P Tsai, Y Jeng,* National Chung Cheng University, Taiwan, Republic of China*; K Turner, R Carpick,* University of Pennsylvania*; J Harrison,* United States Naval Academy

Contact at the nanoscale has important implications in the use of tip-based nanomanufacturing, data storage, and imaging with scanning probes. Tip wear reduces predictability in manufacturing and image quality such scanning probe applications. As a means to reduce wear, hard wear-resistant materials such as diamond-like carbon (DLC), ultra nanocrystalline diamond (UNCD) and amorphous carbon (a-C:H) have been used to coat scanning probe tips. Understanding of the behavior of these materials in contact is critically important towards their successful application. Recent studies using a TEM nanoindenter showed significant variation in both pull-in and pull-off forces obtained during the repeated indentation of a DLC tip on to a diamond surface. Furthermore, the pull-in and pull-off forces do not appear to be correlated. In this presentation, results from molecular dynamics simulations of a DLC tip on hydrogen terminated diamond surfaces will presented. The tip was constructed with a structure and geometry similar to that of the tip used in experiment. The resulting simulations show that the pull-in force appears to be dominated by long range dispersion forces while the pull-off force is determined by the number of covalent bonds formed during the contact. For a given surface, the formation of bonds during indention appears to be a stochastic process with multiple indents of the same tip at the surface same location showing a widely varying number of bonds formed. It is therefore hypothesized that the variation in pull-off forces observed in experiment are also related to the number of bonds formed across the tip-surface interface.

Supported by **The National Science Foundation and the Air Force Office of Scientific Research

2:40pm **TR+AS+HI+NS+SS-WeA-2 New Insights about the Fundamental Mechanisms of Friction of MoS_2**, ***John Curry***, Lehigh University*; M Wilson, T Babuska, M Chandross,* Sandia National Laboratories*; H Luftman, N Strandwitz, B Krick,* Lehigh University*; N Argibay,* Sandia National Laboratories

Molybdenum Disulfide (MoS_2) is a solid lubricant used widely in aerospace applications, capable of providing ultralow friction (coefficients, $\mu < 0.01$) in inert environments such as the vacuum found in outer space. Research over the years has led to the development of MoS_2 nanocomposites that are less susceptible to the effects of environment and aging. Yet, the fundamental mechanisms responsible for environmental sensitivity and degraded friction behavior remain highly debated. Tribological experiments and molecular dynamics (MD) simulations were used to understand the evolution of friction behavior for MoS_2 coatings as a function of temperature, lattice commensurability and defect density. The oxidative resistance of MoS_2 coatings prepared with differing degrees of defect density and basal orientation was also assessed via high-sensitivity low-energy ion scattering (HS-LEIS) depth profiling, X-ray photoelectron spectroscopy (XPS), tribological experiments and MD. Through these findings, we discuss new connections between atomic-scale mechanisms and macro-scale friction behavior of this class of lubricant in a wide range of environments.

3:00pm **TR+AS+HI+NS+SS-WeA-3 The Influence of Environmental Exposure and the Substrate on the Lubricating Properties of Two-Dimensional Materials**, *P Gong,* University of Calgary, Canada*; Z Ye,* Miami University*; L Yuan,* ***Philip Egberts***, University of Calgary, Canada **INVITED**

The friction reducing properties of graphene has been shown to have a number of interesting properties, such as a dependence on the number of layers of the lubricant present, an exceptional dependence on the surface adhesion properties of the underlying substrate, and environmental stability. These properties have been attributed to its low mechanical strength to out-of-plane deformation, its innate thinness, and the influence of the environment on the surface energy. In this work, the friction reducing properties of graphene are examined using experiments and simulation. In particular, the influence of surface energy and associated wettability of graphene are explored as a property that can influence the lubrication properties of graphene, but also friction hysteresis. While little to no variation in pull-off forces have been observed on varying numbers of graphene covering the surfaces in experiments, the impact of the variance of surface energy on friction, and in particular friction under humid environments, suggests that surface energy may have a stronger influence on graphene's friction reducing ability than suggested in previous work.

4:20pm **TR+AS+HI+NS+SS-WeA-7 Fundamental Understanding of Interfacial Adhesion and Tribochemistry by Ab Initio Calculations**, ***M.Clelia Righi***, University of Modena and Reggio Emilia, Italy **INVITED**

Tribologically-induced chemical modifications of surfaces interacting with lubricant additives or other molecules present in the environment surrounding the sliding media can substantially change the adhesion and friction of materials in contact. Therefore, is highly desirable to understand how they take place. However, tribochemical reactions are difficult to monitor in real-time by experiments, which leaves a gap in the atomistic understanding required for their control.

We apply *ab initio* molecular dynamics to monitor chemical reactions involving common solid lubricants, namely molybdenum disulfide, graphene/graphite and carbon-based films, interacting with water molecules in the presence of mechanical stresses. Our simulations elucidate atomistic mechanisms relevant to understand the effects humidity on the lubricating properties of these materials.[1,2]

As second issue, we investigate the tribochemistry of sulfur, phosphorus and graphene on iron and discuss the role of metal passivation in reducing the interfacial adhesion and shear strength. We generalize the result by establishing a connection between the tribological and the electronic properties of interfaces. This adds a new piece of information for the ultimate understanding of the fundamental nature of frictional forces. [3,4]

[1] P. Restuccia, G. Levita and M. C. Righi *Graphene and MoS2 interacting with water: a comparison by ab initio calculations* Carbon 107, 878 (2016).

[2] S. Kajita and M. C. Righi*A fundamental mechanism for carbon-film lubricity identified by means of ab initio molecular dynamics,* Carbon 103, 193 (2016).

[3] M. C. Righi, S. Loehlé, M. I. De Barros Bouchet, S. Mambingo-Doumbeand J. M. Martin *A comparative study on the functionality of S- and P-based lubricant additives by combined first principles and experimental analysis* RSC Advances, 6, 47753 (2016).

[4] P. Restuccia and M. C. Righi *Tribochemistry of graphene on iron and its possible role in lubrication of steel* Carbon 106, 118 (2016).

5:00pm **TR+AS+HI+NS+SS-WeA-9 Friction Between 2D Solids during Lattice Directed Sliding***, **Paul Sheehan,*** US Naval Research Laboratory*; C Lieber,* Harvard University

Nanometer-scale crystals of the 2D oxide molybdenum trioxide (MoO_3) were formed atop the transition metal dichalcogenides MoS_2 and $MoSe_2$. The MoO_3 nanocrystals are partially commensurate with the dichalcogenide substrates, being aligned only along one of the substrate's crystallographic axes. These nanocrystals can be slid only along the aligned direction and maintain their alignment with the substrate during motion. Using an AFM probe to oscillate the nanocrystals, it was found that the lateral force required to move them increased linearly with nanocrystal area. The slope of this curve, the interfacial shear strength, was significantly lower than for macroscale systems. It also depended strongly on the duration and the velocity of sliding of the crystal, suggesting a thermal activation model for the system. Finally, it was found that lower commensuration between the nanocrystal and the substrate increased the interfacial shear, a trend opposite that predicted theoretically.

5:40pm **TR+AS+HI+NS+SS-WeA-11 Single Molecule Force Measurement: Mechanic and Symmetry Dependent Lateral Force***, **Yuan Zhang,*** Argonne National Laboratory*; S Khadka,* Ohio University*; B Narayanan, A Ngo,* Argonne National Laboratory*; Y Li,* Ohio University*; B Fisher, L Curtiss, S Sankaranarayanan, S Hla,* Argonne National Laboratory

Mechanical properties of molecules adsorbed on materials surfaces are increasingly vital for the applications of molecular thin films as well as for the fundamental understanding of quantum friction. Here, we employ atomic/molecular manipulation schemes to investigate mechanic and symmetry dependent lateral diffusion processes of individual molecules on various substrates using a combined scanning tunneling microscope (STM) and q+ atomic force microscopy system at low temperature and in ultrahigh vacuum environment. Lateral force measurements of individual sexiphenyl, an oligomer type molecule, exhibits an interesting friction behavior originated from the symmetry of the molecule-substrate system. We found ~160% increase in the lateral force during the diffusion of the molecule across a Ag(111) surface depending on the long molecular axis alignment on the surface. A comparison of the shape and symmetry dependent molecular diffusion process is realized by manipulating cobalt-porphyrin molecules on aAu(111) surface. The results are in full agreement with molecular dynamic simulations. In addition, we will also discuss atomic and molecular level force measurement using an STM tip, which opens simultaneous electronic, structural and force measurements at the single atom and molecule level.

Advanced Ion Microscopy Focus Topic
Room 7 & 8 - Session HI+BI+NS+TR-ThM

Advanced Ion Microscopy Applications

Moderators: Armin Golzhauser, Bielefeld University, Germany, Olga Ovchinikova, Oak Ridge National Laboratory

8:00am **HI+BI+NS+TR-ThM-1 Scanning Helium Atom Microscopy: Imaging with a Deft Touch***, **Paul Dastoor,*** University of Newcastle, Australia **INVITED**

Delicate structures (such as biological samples, organic films for polymer electronics and adsorbate layers) suffer degradation under the energetic probes of traditional microscopies. Furthermore, the charged nature of these probes presents difficulties when imaging with electric or magnetic fields, or for insulating materials where the addition of a conductive coating is not desirable. Scanning helium microscopy is able to image such structures completely non-destructively by taking advantage of a neutral helium beam as a chemically, electrically, and magnetically inert probe of the sample surface. Here, we present scanning helium micrographs demonstrating image contrast arising from a range of mechanisms including, for the first time, chemical contrast observed from a series of metal-semiconductor interfaces [1]. The ability of neutral helium microscopy to distinguish between materials without the risk of damage makes it ideal for investigating a wide range of systems.

1. M. Barr, A. Fahy, J. Martens, A.P. Jardine, D.J. Ward, J. Ellis, W. Allison & P.C. Dastoor, "Unlocking new contrast in a scanning helium microscope", *Nature Communications*, **7**, 10189, (2016).

8:40am **HI+BI+NS+TR-ThM-3 Biofilm Structure of Geobacter Sulfurreducens by Helium Ion Microscopy***, **Alex Belianinov**, M Halsted, M Burch, S Kim, S Retterer,* Oak Ridge National Laboratory

Microbial communities form biofilms on material surfaces in a multitude of ecosystems, from the root hairs of a plant to the human gut. The hallmarks of an established biofilm include (1) the attachment of microbial cells to a surface, (2) production of extracellular polymeric substance, (EPS) (3) a complex structure or "architecture," and (4) the ability to exchange genetic information between cells. [1] Geobacter sulfurreducens forms unique, electrically conductive biofilms, a property that can be exploited in production and design of microbial fuel cells. In this work, examine biofilm formation, and biofilm properties of Geobacter sulfurreducens using a Scanning Electron Microscope (SEM) as well as a Helium Ion Microscope (HIM).

SEM is a high-resolution imaging technique used for characterization of a broad variety of materials. However, in order to image highly insulating, soft biological materials, the samples must be coated for charge compensation. These (typically) metallic coatings create a homogenous surface and may cloak true biological behavior and material contrast in the micrograph. In the case of Geobacter sulfurreducens, metal coating precludes detailed investigation of microbial attachment, presence of EPS, and fine surface details that may elucidate the mechanisms behind architecture formation and genetic material exchange.

Recently introduced HIM, offers more flexibility in investigating biological samples, as highly insulating sample can be imaged sui generis, without the use of a conductive coating. [2] This opens new pathways to capturing high resolution spatial details of biofilm formation and biofilm properties. Furthermore, high-resolution HIM imaging reveals true surface details of Geobacter sulfurreducens, such as flagella or pilin typically inaccessible by SEM. Finally, the effects of different sample preparation strategies for SEM and HIM will be illustrated and discussed.

References:

[1] 1. Donlan, R. M. "Biofilms: Microbial Life on Surfaces." Emerging Infectious Diseases, 8(9), 881–890, 2002

[2] Joens, M. S., Huynh, C., Kasuboski, J. M., Ferranti et. al.. "Helium Ion Microscopy (HIM) for the imaging of biological samples at sub-nanometer resolution." Scientific Reports, 3(3514), 2013

Acknowledgements:

This research was supported by Oak Ridge National Laboratory's Center for Nanophase Materials Sciences (CNMS), which is sponsored by the Scientific User Facilities Division, Office of Basic Energy Sciences, U.S. Department of Energy.

9:00am **HI+BI+NS+TR-ThM-4 Channeling via Transmission He Ion Microscopy***, **Christoph Herrmann,*** Simon Fraser University, Canada*; S Scott, M Lagally,* University of Wisconsin-Madison*; K Kavanagh,* Simon Fraser University, Canada

The spatial coherence of focussed helium (He) ion beams is significant. The He ion source is atomic size (W filament tip) and the resolution from scanning probe, ion-induced secondary electron images is sub 1 nm. Scanning transmission images with atomic resolution are theoretically predicted. We have been experimenting with a digital camera located underneath the sample stage and tilt cradle of our instrument (Zeiss Nanofab). The camera consists of an array of Si p-i-n diodes (55 µm square pixels) that allow direct detection of single He ions and atoms (20 keV - 40 keV). We have previously reported that the beam intensity profiles are uniformly distributed, as expected from the small de Broglie wavelength (80 fm), with a half angle convergence of 2 mrad.[1] At beam currents in the pA range the detector count rate was consistent with one count per He ion or atom. In this talk, we will present results that indicate planar channeling in single crystalline Si (100) membranes (25 nm - 75 nm thick). The transmission intensity as a function of position depends on the beam incidence angle, and beam energy, with random incidence profiles consistent with monte carlo scattering and range calculations (SRIM). The peak in transmission as a function of incidence angle has a half angle width of 1° at 25 kV. These results will be compared with theoretical calculations based on impact factors at low energies. Channeling experiments with other thin crystalline materials including graphite and MgO will be discussed. **Acknowledgements**: We thank Norcada Inc. (Edmonton) for supplying Si (100) 50 nm thick membranes; NSERC, CFI/BCKDF, 4DLABs for funding. [1] K.L. Kavanagh and C. Herrmann, Direct He Detection for Transmission Helium Ion Microscopy, Microsc. Microanal. submitted 2017.

9:20am **HI+BI+NS+TR-ThM-5 Rapid Imaging of Nano-Porous Catalyst Particles Via Helium Ion Microscopy***, M Burch, A Ievlev, **Holland Hysmith,*** Oak Ridge National Laboratory*; K Mahady, P Rack,* University of Tennessee*; L Luo,* ExxonMobil Chemical Company*; A Belianinov,* Oak Ridge National Laboratory*; S Yakovlev,* ExxonMobil Chemical Company*; O Ovchinikova,* Oak Ridge National Laboratory

Porous materials are some of the most important modern day material systems, as the pore structure defines many materials applications and functionality. The pore structure of catalyst precursor particles, in particular, is of great importance to the catalyst community, as this pore structure dictates the efficiency and efficacy of grown polymers. However, despite the importance of these materials systems, there are few techniques to analyze pore size and structure. The most common technique is gas absorption, where the amount of gas absorbed and desorbed from a known amount of material is tracked and the average pore volume and size can be extracted. However, the technique is heavily dependent on sample quality and which fitting model is used to calculate volume and size. In addition, the technique is quite slow, where generally at most a single sample can be analyzed a day.

In this work, we demonstrate a novel technique to directly image and quantify pore size in nano-porous catalyst precursor particles via helium ion microscopy. We demonstrate the technique by directly imaging the surface pore structure of $Si\text{-}O_2$ precursor catalyst particles with helium ion microscopy. Using modern day data analytics, we created an automated routine to extract pore size and distributions. We show that our HIM based technique shows comparable data to the industry standard gas absorption technique, within a 5 percent difference between the techniques of a known porous samples.

Further, to determine the effect of the helium beam on the surface of the SiO_{-2} particles, we simulate the beam interaction between porous SiO_{2-} particles and the helium beam. At low ion doses the surface modification by the ion beam is quite negligible, where at higher ion doses, significant surface modification is observed.

In conclusion, we've demonstrated a novel technique to directly visualize and quantify nano-pore size and structure in SiO_2 that yields complimentary data to gas absorption.

Acknowledgements

This work was conducted at the Center for Nanophase Materials Sciences, which is a Department of Energy (DOE) Office of Science User Facility. The users acknowledge the ExxonMobil Chemical Company for funding.

9:40am **HI+BI+NS+TR-ThM-6 Ion Beam Induced Current Measurements of Solar Cells with Helium Ion Microscopy**, *A Belianinov, S Kim,* ***Ryan Cannon,*** *M Burch, S Jesse, O Ovchinnikova,* Oak Ridge National Laboratory

The scanning electron microscope (SEM) is a versatile high-resolution microscopy tool, and perhaps the most widely used imaging platform across many engineering and scientific fields [1]. Within the last decade, another microscopy technique based on a gaseous field ionization source, utilizing Helium and Neon ions has been introduced [2]. While the popularity of the SEM is hardly challenged by the Helium Ion Microscopy (HIM), there are instances when imaging with ions offers significant advantage as opposed to imaging with electrons. In principle, both HIM and the SEM share many similarities, for example, a HIM operating at 40 keV will generate ions with velocity comparable to SEM operating at 5 keV. However, due to much higher stopping power of ions, as compared to electrons, ion based secondary electron (iSE) will be higher. Also, as a result, there is little ion backscattering, and consequently, the concentration of the ion-generated iSE2 (additional secondary electron generated by SE interaction within the material) is usually insignificant.

In this work, we exploit small interaction volumes in the HIM, and take advantage of the lower iSE2 yield, and positively charged helium ions to map ion beam induced current (IBIC) in solar cell materials. Similar studies, using electrons, have visualized induced current profiles at grain profiles in polycrystalline solar cells, and in silicon [3, 4]. Furthermore, broad ion sources have been utilized in conjunction with scanning probe systems in the past to map out current changes in FinFETs [5]. We are interested in utilizing the HIM to map current at the nanoscale near p-n junctions in CdTe to elucidate differences in contrast captured by the ion beam induced current, as opposed to the electron beam induced current. These findings will illustrate the peculiarities of ionic transport in these solar cell materials, and will evaluate the HIM technology as a potential quality control tool.

References:

[1] David C Joy, Helium Ion Microscopy: Principles and Applications, First ed. Springer, New York USA, Heidelberg Germany, Dordrecht Netherlands, London United Kingdom, 2013.

[2] Gölzhäuser, A. and Hlawacek, G., Helium Ion Microscopy. Springer International Publishing. 2016

[3] Donolato, C., Journal of Applied Physics, 54 (3), 1314-1322, 1983

[4] Chen, J., et. al., Journal of Applied Physics, 96(10), 5490-5495, 2004

[5] Manfredotti, C., et.al., Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment, 380(1-2), 136-140, 1996

11:00am **HI+BI+NS+TR-ThM-10 Writing Magnetic Domains with a Helium Ion Microscope**, ***Daniel Emmrich,*** Bielefeld University, Germany*; A Gaul, D Holzinger, A Ehresmann,* University of Kassel, Germany*; F Karimian, M Klug, J McCord,* Kiel University, Germany*; A Beyer, A Gölzhäuser,* Bielefeld University, Germany

Microscopes based on gas field ion sources offer surface-sensitive, high resolution imaging and state of the art nano-machining.[1] It was further shown that light ions like helium or neon enable a modification of the magnetic properties, e.g., turning thin films from paramagnetic to ferromagnetic state, without significant sputtering.[2]

In this work, two-dimensional ion bombardment induced magnetic patterning (IBMP)[3] is demonstrated with a helium ion microscope to create magnetic domains in an exchange biased thin film system. Such a system consists of a thin ferromagnetic layer coupled to an underlying antiferromagnet. Low dose helium ion irradiation at an energy of 15 keV in an external magnetic field leads to a new, remanent magnetization direction, determined by the external magnetic field. By subsequently patterning the sample in differently orientated external magnetic fields, complex magnetic domain patterns such as chiral structures can be written. Based on magnetic force microscopy and optical Kerr microscopy, we will discuss the achievable resolution as well as the shapes of different artificial magnetic domains.

[1]G. Hlawacek and A. Gölzhäuser (eds),Helium Ion Microscopy (Springer International Publishing, Switzerland, 2016.).

[2]F. Roder, G. Hlawacek, S. Wintz, R. Hubner, L. Bischoff, H. Lichte, K. Potzger, J. Lindner, J. Fassbender, and R. Bali, Scientific reports 5, 16786 (2015).

[3]A. Gaul, S. Hankemeier, D. Holzinger, N.D. Müglich, P. Staeck, R. Frömter, H.P. Oepen, and A. Ehresmann, Journal of Applied Physics 120, 33902 (2016).

11:20am **HI+BI+NS+TR-ThM-11 Characterisation of Nanomaterials on the Helium Ion Microscope using Correlative Secondary Electron and Mass Filtered Secondary Ion Imaging**, *J Audinot, D Dowsett, F Vollnhals, T Wirtz,* Luxembourg Institute of Science and Technology (LIST), Luxembourg*;* ***John A. Notte,*** Carl Zeiss Microscopy, LLC

In order to add nano-analytical capabilities to the Helium Ion Microscope, we have developed a Secondary Ion Mass Spectrometry (SIMS) system specifically designed for the Zeiss ORION NanoFab [1-3]. SIMS is based on the generation and identification of characteristic secondary ions by irradiation with a primary ion beam (in this case helium or neon). It is an extremely powerful technique for analysing surfaces owing in particular to its excellent sensitivity (detection limits down to the ppb are possible, so that SIMS can be used to detect both major and trace elements), high dynamic range (a same signal can be followed over several orders of magnitude), high mass resolution and ability to differentiate between isotopes.

In SIMS, the typical interaction volume between the impinging ion beam and the sample is around 10 nm in the lateral direction. As the probe size in the HIM is substantially smaller (both for He and Ne), the lateral resolution on the integrated HIM-SIMS is limited only by fundamental considerations and not, as is currently the case on commercial SIMS instruments, the probe size [4,5]. We have demonstrated that our instrument is capable of producing elemental SIMS maps with lateral resolutions down to 12 nm [3-5].

Furthermore, HIM-SIMS opens the way for in-situ correlative imaging combining high resolution SE images with elemental and isotopic ratio maps from SIMS [4,5]. This approach allows SE images of exactly the same zone analysed with SIMS to be acquired easily and rapidly, followed by a fusion between the SE and SIMS data sets.

In this talk, we will present a number of examples taken from various fields of materials science (battery materials, solar cells, micro-electronics, coatings) and life science (nanoparticles in creams and biological tissues) to show the powerful correlative microscopy possibilities enabled by the integrated HIM-SIMS instrument.

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Advanced Ion Microscopy Focus Topic
Room 7 & 8 - Session HI+NS+TR-ThA

Novel Beam Induced Surface Analysis and Nano-Patterning

Moderators: Anne Delobbe, Orsay Physics, Shinichi Ogawa, National Institute of Advanced Industrial Science and Technology (AIST)

2:20pm **HI+NS+TR-ThA-1 Multimodal Chemical Imaging of Nanoscale Interfacial Phenomena on a Combined HIM-SIMS Platform***, **Olga Ovchinnikova,*** Oak Ridge National Laboratory **INVITED**

The key to advancing energy materials is to understand and control the structure and chemistry at interfaces. However, significant gaps hamper chemical characterization available to study and fully comprehend interfaces and dynamic processes; partly due to the lack of breadth of necessary information, as well as its scattered nature among a multitude of necessary measurement platforms. Multimodal chemical imaging transcends existing analytical capabilities for nanometer scale spatially resolved interfacial studies, through a unique merger of advanced helium ion microscopy (HIM) and secondary ion mass spectrometry (SIMS) techniques. In this talk I will discuss how to visualize material transformations at interfaces, to correlate these changes with chemical composition, and to distil key performance-centric material parameters using a multimodal chemical imaging approach on a combined HIM-SIMS system. Particular attention will be focused on how to use the HIM-SIMS to study the role of ionic migration on the photovoltaic performance, or alternatively whether the ionic migration plays a positive or negative role in determining superior photovoltaic performance in organic-inorganic perovskites (HOIPs). I will discuss how synthesizing perovskite on custom substrates effect active chemical agents in materials and understand how interfaces in materials affect the local chemistry, specifically, key energy related parameters such as electron and ion motion and their re-distribution. Overall, multimodal chemical imaging on a combined HIM-SIMS platform offers the potential to unlock the mystery of active interface formation through intertwining data analytics, nanoscale elemental characterization, with imaging; to better grasp the physical properties of materials and the mechanistic physics-chemistry interplay behind their properties.

Acknowledgements

This work was conducted at the Center for Nanophase Materials Sciences, which is a Department of Energy (DOE) Office of Science User Facility

3:00pm **HI+NS+TR-ThA-3 Characterizing Surface Immobilized Antibodies using ToF-SIMS and Multivariate Analysis***, N Welch,* CSIRO Manufacturing, Australia*; R Madiona,* La Trobe University, Australia*; J Scoble, B Muir,* CSIRO Manufacturing, Australia*; **Paul Pigram,*** La Trobe University, Australia

Antibody attachment, orientation and function at the solid interface are critical for the sensitive detection of biomolecules during immunoassays. Correctly oriented antibodies with solution-facing antigen binding regions have improved antigen capture in comparison with randomly oriented antibodies. Direct characterization of oriented proteins with surface analysis methods still remains a challenge. Surface sensitive techniques, however, such as Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) provide information-rich data that can be used to probe antibody attachment, orientation, denaturation and related characteristics.

Diethylene glycol dimethyl ether plasma polymers (DGpp) functionalized with chromium (DGpp+Cr) have improved immunoassay performance that is indicative of preferential antibody orientation. ToF-SIMS data from proteolytic fragments of anti-EGFR antibody bound to DGpp and DGpp+Cr have been used to construct artificial neural network (ANN) and principal component analysis (PCA) models indicative of correctly oriented systems. Whole antibody samples (IgG) tested against each of the models indicate preferential antibody orientation on DGpp+Cr. Cross-reference between ANN and PCA models yield 20 mass fragments associated with $F(ab')_2$ region representing correct orientation, and 23 mass fragments associated with the Fc region representing incorrect orientation. The mass fragments have been compared with amino acid fragments and amino acid composition in $F(ab')_2$ and Fc regions. A ratio of the sum of the ToF-SIMS ion intensities from the $F(ab')_2$ fragments to the Fc fragments demonstrated a 50% increase in intensity for IgG on DGpp+Cr as compared to DGpp.

This systematic data analysis methodology offers new opportunities for the investigation of antibody orientation on a range of substrates. It also yields good results for the characterization of antibody denaturation and for determining limits of detection.

4:00pm **HI+NS+TR-ThA-6 Single-nanometer Functional Graphene Devices Patterned with Helium Ion Beam***, **Hiroshi Mizuta,*** *M Schmidt, T Kanzaki,* Japan Advanced Institute of Science and Technology (JAIST), Japan*; S Ogawa,* National Institute of Advanced Industrial Science and Technology (AIST), Japan*; M Muruganathan,* Japan Advanced Institute of Science and Technology (JAIST), Japan **INVITED**

The bombardment of specimen by accelerated ions causes implantation and surface sputtering. The latter can be employed to create structures with sub-10 nm dimensions. This precision is demonstrated in electrically contacted and suspended graphene layers by a Helium ion microscope (HIM). 5 nm wide monolayer graphene, suspended above a pore and milled by HIM, had been demonstrated [1]. However, the physical properties of such a ribbon cannot be investigated in such a device architecture due to lack of electrical contacts. Recently, we demonstrated a ~6 nm wide suspended GNR and reported on the room temperature electrical characteristics. A wide range of drain current, at which current suppression occurs, has been observed [2]. However, that device was based on exfoliated grapheme, which makes it necessary to individually design the structures, and the yield is typically small.

Here, we report on the large-array processing of 100 nm wide monolayer CVD based suspended graphene structures by HIM. The structures are prepared by electron-beam lithography and thin-film processing. Before HIM milling, the graphene is released by buffered hydrofluoric acid etching and critical point drying. Annealing in H_2/Ar atmosphere is used to remove the resist contamination.

We will first discuss results of fabricating suspended GNRs with constriction milled with HIM (30 keV acceleration voltage, $1.1x10^{18}$ ion/cm^2). The GNRs with sub-10-nm constriction are successfully patterned, and the electrical conduction is measured as function of temperature. We will discuss the milling results and electrical characterization in detail along with their potential impact on the performance of graphene-NEMS-based single-molecular detection [3]. We will then report on the recent progress in preparing arrays of nanopores in graphene by HIM. 9x9 arrays with pitch of ~9 nm are successfully realized with a dose of $6.5x10^{18}$ ions/cm^2. Arrays of pores spanning a complete suspended ribbon with a pitch of ~18 nm are demonstrated as well, and we will discuss the impact of such periodic structure on the electrical and thermal (phononic) transport for nanoscale heat phonon engineering.

Acknowledgements: T. Iijima is acknowledged for the usage of the HIM at AIST SCR Station**.** This research was supported through the Grant-in-Aid for Scientific Research KAKENHI 25220904, 16K13650, 16K18090 from JSPS and COI program of the Japan Science Technology Agency.

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5:00pm **HI+NS+TR-ThA-9 Monte Carlo Simulation Study of Gas Assisted Focused Ion Beam Induced Etching***, **Kyle Mahady,*** *P Rack,* University of Tennessee*; S Tan,* Intel Corporation*; Y Greenzweig,* Intel Corporation, Israel*; R Livengood,* Intel Corporation*; A Raveh,* Intel Corporation, Israel

We present a simulation study of focused ion beam etching using a gas assist. The use of a precursor gas greatly enhances material removal rate when compared to ion beam sputtering, enabling features such as valleys to be etched with lower ion doses, and consequently less damage to the substrate. The basis of our study is a Monte Carlo based code for focused ion beam milling, which simulates the cumulative removal of material due to sputtering, and secondary electron emission, for various target compositions and structures. In this talk, we describe the gas assisted etching portion of the code, which simulates monolayer adsorption of XeF_2 to a SiO_2 substrate, and the reactions between adsorbed gas and surface atoms which lead to volatilization and material removal. We study the effect of etching parameters such as beam current and gas flux on the shape of etched valleys, and the influence of ion species such as Ne^+ and Ga^+, to characterize the underlying limitations on etching resolution. Simulations are compared against experimental results, for validation and to understand experimentally observed features.

5:20pm **HI+NS+TR-ThA-10 Direct Write of Complex 3-Dimensional Structures with Helium Ion Microscopy**, ***Matthew Burch***, *A Ievlev,* Oak Ridge National Laboratory*; M Stanford, B Lewis,* University of Tennessee*; X Sang, S Kim, J Fowlkes,* Oak Ridge National Laboratory*; P Rack,* University of Tennessee*; R Unocic, A Belianinov, O Ovchinikova,* Oak Ridge National Laboratory

The next generation of computing, the generation which will follow the end of Moore's law, will need materials processing and interconnects that exist in 3-dimensions. This need has led to multiple investigations into the fabrication of complex free standing 3-dimensional structures onto any substrate at the nano-scale. Several techniques are currently being developed to fabricate free-standing micro- and nano- level structures including two-beam photon lithography and focused electron beam induced deposition (FEBID). Recent advancements in FEBID has led to the ability to simulate and subsequently fabricate complex 3D-platinum structures from an organometallic precursor.

In this work, we demonstrate the ability of the helium ion microscope (HIM) to fabricate complex 3-dimensional structures using focused ion beam deposition (FIBID) at scales smaller than previously demonstrated with FEBID. Using modern day image analytics we demonstrate a method we've successfully utilize to investigate and understand some of the most important structure-growth parameters with FIBID and how that parameter space impacts the size and morphology of created structures. These parameters include beam current, organometallic gas volume, dwell time, etc., and how these impact a grown structures length, width, and possible growth angles.

We further investigate the composition and crystalline nature utilizing scanning transmission electron microscopy (STEM) and electron energy loss spectroscopy (EELS) in the prescence of different carrier gasses of nitrogen and oxygen. We show, that in general the morphology and chemistry is nearly identical between the two gasses, that there is a slight difference in the apparent crystalline nature between the two flow gasses.

Finally, we demonstrate the minimum size structures currently grown with FIBID and the complex nature of the way these structures can be grown. We also demonstrate the HIM's unique ability to direct write structures repeatedly and reliably on non-conductive using the HIM's unique charge compensation mechanisms.

Acknowledgements

This work was conducted at the Center for Nanophase Materials Sciences, which is a Department of Energy (DOE) Office of Science User Facility.

Advanced Ion Microscopy Focus Topic
Room Central Hall - Session HI-ThP

Advances in Ion Microscopy Poster Session

HI-ThP-1 Sub-10 nm Width High Aspect Ratio Trench Patterning of Gold Film using Helium Ion Microscope*, Etsuo Maeda,* The University of Tokyo, Japan*; T Iijima,* National Institute of Advanced Industrial Science and Technology (AIST), Japan*; R Kometani,* The University of Tokyo, Japan*; S Migita, S Ogawa,* National Institute of Advanced Industrial Science and Technology (AIST), Japan

The helium ion microscope (HIM) realized the sub-nm level imaging with helium (He) ion beam from single tungsten atom on the top of the trimer. There are numbers of study focusing on nanostructure imaging, for example carbon nanotubes, graphene films, and self-assembled monofilms [1-3]. Through these previous studies, the advantages of HIM for imaging with high resolution and high contrast have been proved.

In our work, an HIM (Carl Zeiss ORION PLUS at AIST SCR station) was applied for sub-10 nm He ion beam etching to realize nano-gap trenches of high aspect ratio for the high sensitive surface enhanced Raman scattering (SERS) devices consisted by gold (Au) structures. To reveal the penetration depth of the He ions into the Au film with 30 kV acceleration voltage, two-body collision calculation was performed [4]. As the results of the calculation, the expected penetration depth was decided as 200 nm. Following the penetration depth results, Au films were prepared (70, 160, and 700-nm-thick) on 500-nm-thick silicon dioxide (SiO_2) layer. After a focused He ion beam irradiation (10^{20} ions/cm^2, acceleration voltage: 30kV, beam current: 2.5 pA), a cross section shape was evaluated with Z-contrast transmission electron microscopic (TEM) images.

In case of thin films (70 and 160-nm-thick), funnel shapes from beam profile were observed. The dimension of the funnel shape was ~40 nm as width and ~35 nm as depth. The narrowest gap size was 5.5 nm for 70-nm-thick Au film and 1.4 nm for 160-nm-thick Au film. The lowest estimated aspect ratio of the nano-gap trench was 24.0 with 160-nm-tchik Au film. Moreover, observed Au atom penetration depth of 160-nm-thick Au film was 20% lager than that of 70-nm-thick Au film. With 70-nm-thick Au film, the fabricated narrow gap was destroyed by recoiled He ions from an interface between the Au film and SiO_2 layer.

Unlike the thin Au films, a thick Au film (700-nm-thick) showed blistered shape in the middle of the film as expected. The fissures in the thick Au film could be related to the blistering phenomena as seen in the Si substrate. He ions which could not path through thick Au film might be gasified in the thick Au film. For the purpose of realizing the nano-gap trenches for SERS devices, we need more structural and morphological characterization to suppress these blistering phenomena.

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HI-ThP-2 Optimized *ex situ* Lift Out of FIB Prepared Specimens*, Lucille Giannuzzi,* EXpressLO LLC

Focused ion beam (FIB) microscopes may be used to prepare site specific lift out specimens for subsequent characterization by transmission electron microscopy, surface science, or other analytical techniques [1-3]. *ex situ* lift out (EXLO) exploits the physics of adhesion forces for picking up a specimen with a solid probe tip and placing or manipulating it to a suitable carrier [1-3]. In this presentation, techniques for fast, easy, and successful lift out and manipulation reproducibility rates are described. Once the specimen is lifted out to the probe, the probe can be rotated to position the specimen either on top of, or under the probe, depending on the type of carrier to be used. In addition, since the specimen is not rigidly fixed to the probe, the specimen orientation can be rotated about the probe using the carrier itself, for precise positioning in just a couple of minutes. Precise orientation of the specimen with respect to the probe provides the greatest success rates, reliability, and throughput of the manipulation process. These and other methods will be fully described and presented.

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