Advanced Ion Microscopy Focus Topic
Room 104A - Session HI-WeA

10 Years of GFIS Microscopy

Moderators: Gregor Hlawacek, Helmholtz-Zentrum Dresden Rossendorf, Germany, Richard Livengood, Intel Corporation

2:20pm **HI-WeA1 A Spectacular Collision of Entrepreneurial Spirit and a Doomed Technology… Transforming the Impossible into the Helium Ion Microscope***, **Bill Ward,*** Entrepreneur, Scientist, Inventor, and Consultant **INVITED**

More than 50 years of research by many of the top physicists in our field culminated in a final effort to resolve the formidable problem of stabilizing the Gas Field Ion Source. It was the collective opinion that several of the physical problems were unsolvable before our efforts began at Micrion nearly 20 years ago.

Our inventive process, the scientific breakthroughs and the many failures are presented in story-like fashion to explain how we successfully created the brightest ion source known to mankind. The human side of our first ten years of research which led to the successful creation of the Single Atom Ion Source and the Helium Ion Microscope is discussed. How the Entrepreneurial Spirit played a significant role in solving the final technical issues will be presented.

This perspective is offered with the hope of inspiring others to dream beyond their perceived limits.

3:00pm **HI-WeA3 Recent Developments of the Gas Field Ion Source***, **John A. Notte,*** Carl Zeiss Microscopy, LLC

Since the commercial introduction of the Gas Field Ion Source in 2006, there has been a steady progression in both the GFIS technology and the GFIS applications. The majority of the technological developments were motivated by the goal of making the helium beam more stable and more versatile. This includes improvements to the high voltage stability, cryogenic system, the gun vacuum, and gas purity. Other changes were adopted to simplify the installation process and make the facilities requirements less demanding. Other efforts made the instrument more compatible and consistent with the larger family of Zeiss instruments.

Simultaneously, there have been changes to the GFIS *applications,* largely driven by insightful customers wanting to exploit this unique beam. This gradual change is reflected in the marketing literature which promoted the original instrument as a microscope (e.g. the "ORION Helium Ion Microscope"), whereas we now emphasize the descendent product as an instrument for nanofabrication (e.g. the "ORION NanoFab"). The new applications include lithography, nanoscale milling, structural modification, beam induced chemistry, transmission imaging, and various analytical methods. The latest NanoFab includes automated scripting, to allow for mass production and parametric investigations.

In support of these new applications, there is a desire to operate the GFIS with gas species other than helium to produce, for example a focused neon beam. The operation of the GFIS with neon introduces challenges not present with helium. And many of the newest developments of the GFIS are aimed towards making the neon operation more reliable. The changes include improvements to the base vacuum, column, the gas manifold, and gas purity. Other changes are operational, helping to define the circumstances where neon can operate most successfully.

This talk will review the evolution of the ORION product family over the last 10 years. Special attention will be given to the most recent efforts undertaken to produce a stable neon ion beam.

3:20pm **HI-WeA4 Monte-Carlo Simulations of Ion Beam Milling in Compound Targets***, **Kyle Mahady,*** *P.D. Rack,* University of Tennessee*; S. Tan, R.H. Livengood, Y. Greenzweig, A. Raveh,* Intel Corporation

In this talk, we will present an experimental and simulation based study of the evolution of nanostructures resulting from ion beam sputtering. We have updated our Monte-Carlo based code EnvizION, which simulates the sputtering and milling process for a variety of target compositions and structures. The computational efficiency of the updated code permits the simulation of more detailed physics in larger scale problems, with doses involving millions of ions and targets with hundreds of millions of atoms. In particular, we can now simulate milling in compound targets, as well as replacement of target atoms with impinging ions of single atom targets. We study the development of nanostructures in compound and single atom targets, and the evolution of the composition in the near surface region. Simulations of targets composed of SiO_2 demonstrate preferential sputtering of oxygen, and associated enrichment of Si in the near surface region. For ion beams in targets composed of crystalline Si, we study the replacement of target Si atoms, and relate the amorphization of the the Si with the energy implanted due to the beam. Simulation results are compared with experiments.

4:20pm **HI-WeA7 Characterization of Structural Changes During HIM and SEM Imaging of Organic Films***, **Shinichi Ogawa,*** National Institute of Advanced Industrial Science and Technology (AIST), Japan*; T. Ohashi, S. Oyama,* Nissan Chemical Industries, Ltd.

Acrylic organic films of 100 nm thick were spin coated on Si substrates, and then a helium ion or electron beam was irradiated at a dose of $2X10^{15}$ at 30 kV and $1x10^{17}/cm^2$ at 0.7 kV of an adequate observation condition, respectively.Morphological and structural changes after the beam irradiations were characterized by AFM, IR, Raman, and TOF-SIMS. Although the helium beam energy is higher, the combination of the lower helium ion current and the longer range of the ions means that the power density to the organic materials is nearly a factor of 10^3 lower with the 30 keV helium ion beam than it is with the 0.7 keV electron beam [1]. One might therefore expect the materials to shrink less and be less damaged during HIM imaging than during SEM imaging.

AFM showed less decrease in the film thickness in a case of the ion than the electron. It indicated that the electrons might damage the film heavier than the helium ions. IR showed larger signal intensity decrease with less peak broadening and OH system remained in a case of the electrons, while it was completely destroyed in the helium ions case. Raman showed more amorphous carbon in the helium ions irradiated film, which was probably formed by destruction of CH system. Those results mean that helium ions irradiations brought about less surface morphological transformation while it resulted in larger chemical change in a deeper region of the film than the electron irradiations. This phenomenon is probably because heavier helium ions with higher energy came into deeper than electrons to the organic film cutting chains of the organic material with amorphous. TOF-SIMS showed the similar results for larger decrease of signal intensities of CH and CHO systems by the electron irradiation. Those results mean there were a lot of trade-off between irradiations of helium ions and electrons. Based on the above results with optimization, cross sections of filling of the organic materials into trenches were imaged by the HIM and SEM. As described, helium ions damaged the organic materials heavier in depth direction than electrons, while it kept original surface morphology with less transformation or shrink, so imaging of the filled organic materials into trenches by the HIM presumably shows more realistic than the SEM imaging.

T. Iijima and S. Migita are acknowledged for the usage of helium ion microscope at AIST SCR station.

References

[1] S. Ogawa, et al., "Helium ion secondary electron mode microscopy for interconnect material Imaging", Jpn. J. Appl. Phys. 49 04DB12 (2010)

4:40pm **HI-WeA8 Laser-Assisted Focused Helium and Neon Beam Induced Processing***, M.G. Stanford,* The University of Tennessee Knoxville*; S. Tan, R.H. Livengood,* Intel Corporation*; B.B. Lewis,* University of Tennessee Knoxville*; J.D. Fowlkes,* Center for Nanophase Materials Sciences, Oak Ridge National Lab*; **Philip D. Rack,*** The University of Tennessee Knoxville

He^+ and Ne^+ ion beam induced nanoscale processing has been extensively studied as an alternative ion source to the standard liquid Ga^+ source. While superior imaging and nanomachining resolution has been achieved, perhaps the Achilles heel of higher exposure dose nanomachining operations is the cumulative damage that occurs beneath the region of interest. To this end, we have developed a laser-assisted focused He^+ and Ne^+ beam induced process in which a pulsed laser photothermally facilitates the helium diffusion, and for instance silicon interstitial/vacancy recombination, and thus inhibits the amorphization and the nanobubble formation. Futhermore, we have recently studied gas-assisted and laser-gas(XeF_2)-assisted He^+ etching and have realized both reduced swelling as well as enhanced etch rates for titanium thin films. We will overview the processing parameters and the ion/photon/reactive gas fluxes that lead to both damage mitigation as well as laser- and gas-assisted etching.

5:00pm **HI-WeA9 Imaging and Lithography of Two-Dimensional Nanostructures with Helium Ions***, **André Beyer**,* Bielefeld University, Germany **INVITED**

In my talk, I will give an overview about imaging and local milling of two-dimensional nanostructures with helium ion microscopy (HIM). In particular, carbon nanomembranes (CNMs), graphene and biological cell membranes will be discussed. CNMs are made by a combination of molecular self-assembly, radiation-induced cross-linking and the detachment of the cross-linked monolayer from its substrate. Although free-standing CNMs cannot be imaged by light microscopy, charged particle techniques can visualize them. However, CNMs are electrically insulating, which makes them sensitive to charging. The same is true for biological cell membranes. I will demonstrate that HIM is particularly well suited for imaging such insulating membranes due to its efficient charge compensation tool. In particular, I will discuss the effects of sample charging, imaging of multilayers and imaging artefacts for CNMs as a model system. Furthermore, I will show that the focused helium ion beam of the HIM can be utilized to create nanopores with diameters down to 1.3 nm in insulating as well as conducting membranes. An analysis of the nanopore growth behaviour allows determination of the profile of the helium ion beam.

5:40pm **HI-WeA11 High Resolution Elemental Imaging on the Helium Ion Microscope***, **David Dowsett**, J.-N. Audinot, F. Vollnhals, T. Wirtz,* Luxembourg Institute of Science and Technology (LIST), Luxembourg

The Helium Ion Microscope (HIM) has become an ideal tool for imaging and nano-patterning [1]. Imaging with helium ions leads to resolutions of 0.5 nm for secondary electron (SE) based imaging, while structures with sub 20 nm feature sizes may be rapidly patterned using Ne. Despite these advantages, the analysis capability of the instrument is currently limited. At beam energies of 35 keV helium or neon ions do not lead to the emission of characteristic X-rays from a sample. While some compositional information can be obtained from back scattered helium [2], identifying elemental information is more difficult. Secondary Ion Mass Spectrometry (SIMS) is a powerful ion beam based technique for analysing surfaces capable of high sensitivity and high mass resolution. 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). The typical interaction volume for SIMS 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 is limited only by fundamental considerations [3-4] and not, as is currently the case on commercial SIMS instruments, the probe size.

We have developed a prototype SIMS spectrometer specifically adapted to the Zeiss ORION NanoFab. Notably the instrument is capable of producing elemental SIMS maps with lateral resolution limited only by the fundamental interaction between the primary beam and the sample. All elements/isotopes and small clusters with masses up to 500 amu are detectable with a mass resolution M/ΔM greater than 400 and parallel detection of 4 mass channels (Figure 1).

The prospect of adding SIMS to the HIM yields not just a powerful analytical capability, but opens the way for in-situ correlative imaging combining high resolution SE images with elemental and isotopic ratio maps from SIMS [5]. This approach allows SE images of exactly the same zone analysed with SIMS to be acquired easily and rapidly. Figure 2 shows a combined SE-SIMS image of a lithium titanate and boron nitride nanoparticle mixture. The SE image has a resolution of a few nanometres, clearly showing the structure of individual nanoparticles, while the SIMS image has a resolution of a few tens of nanometres and allows unambiguous identification of individual nanoparticles.

We will present the performance characteristics of the spectrometer along with the latest results in the field of materials science.

Advanced Ion Microscopy Focus Topic
Room 104A - Session HI+NS-ThM

Fundamentals of Ion Beam Microscopy

Moderators: Armin Gölzhäuser, Bielefeld University, Germany, Philip D. Rack, The University of Tennessee Knoxville

8:00am **HI+NS-ThM1 Generation of Hydrogen Beams using Single Atom and Trimer Nanotips**, ***Radovan Urban,*** University of Alberta and The National Institute for Nanotechnology, Canada*; K. Nova,* University of Alberta, Canada*; M. Salomons,* National Institute for Nanotechnology, Canada*; R.A. Wolkow,* University of Alberta and The National Institute for Nanotechnology, Canada*; J.L. Pitters,* National Institute for Nanotechnology, Canada

Hydrogen ion beams have been discussed as useful for scanning ion microscopy (SIM) due to their low mass and low sputtering rates. However, hydrogen ion beams are known to occur as mixtures of H^+, H_2^+ and H_3^+ depending on the electric field strength. There is some evidence that various tip orientations contribute differently to the ratios of the ions and also that site-specific regions also affect the gas species but it has not been clearly determined. Understanding the relationship between the field strength dependence, tip shape, and apex termination with specific hydrogen ion creation is therefore critical to prepare pure hydrogen ion beams of a single species. We employed W and Ir to prepare atomically sharp nanotips with various atomic arrangements at the very apex to compare the ratios of H^+, H_2^+ and H_3^+.

The experimental setup included a custom field ion microscope (FIM) operating in ultrahigh vacuum (base pressure <$5x10^{-11}$ Torr). The tip was mounted on a heating loop wire for degassing and could was cooled with a liquid helium flow cryostat. Nanotips were prepared from tungsten single crystal W(111) wire and polycrystalline Ir wire using the field assisted chemical etching method. A magnetic field of ~1T was generated using two permanent magnets mounted between the extractor and the micro-channel plate.

The hydrogen beam composition from a single atom W(111) and Ir nanotips at different applied tip voltages was recorded and analyzed. At low voltages the H_2^+ beam dominates. As the voltage is increased, H_3^+ is also observed until it dominates at larger voltages. In this manner, a particular species can be selected depending on the operating voltage. Furthermore, comparing the hydrogen beam composition between W(111) single atom tip and trimer structure reveal important differences. For trimer nanotip, H_2^+ becomes a significant species and equals the H_3^+ current. However, in the case SAT, H_3^+ becomes the only contribution to ion current at higher voltages resulting in pure H_3^+ beam suitable for imaging.

Relative ratios of H^+, H_2^+ and H_3^+ were studied as a function of tip material (tungsten and iridium), applied voltage, and tip apex structure (single atom and trimer nanotips). We have determined that the tip structure and apex termination for both tungsten and iridium nanotips play important roles in the production of hydrogen ion beams. It has been found that single atom tip at high tip voltages produces nearly pure H_3^+ beam.

8:20am **HI+NS-ThM2 High-brightness Xenon Gas Field Ion Source from a Single-Atom Tip**, ***Ing-Shouh Hwang,*** Institute of Physics, Academia Sinica, Taipei, Taiwan, Taiwan, Republic of China*; W.T. Chang, W.C. Lai, P.-C. Li,* Institute of Physics, Academia Sinica, Taipei, Taiwan*; T.Y. Fu,* Department of Physics, National Taiwan Normal University, Taipei, Taiwan*; T.T. Tsong,* Institute of Physics, Academia Sinica, Taipei, Taiwan

Current focused ion beam systems are mainly equipped with liquid metal ion sources (LIMSs). Even though LMISs are very reliable in operation, their relatively large source size and high energy spread limit the current density. In contrast, gas field ion sources(GFISs) can reach higher beam currents at smaller beam diameters because of their atomic-scale source size and a small energy spread (<1 eV). Since 2006, Zeiss Orion helium ion microscope (HIM) has demonstrated superior performance with a spatial resolution better than 0.5 nm [1]. To expand the application of GFISs, it is essential to develop GFISs of various ion species, particularly, ions of high mass.

Here we present Xe-GFIS emitted from a noble metal covered W(111) single-atom tip (SAT) [2,3]. This type of SATs are thermally and chemically stable, and high-brightness helium, neon, argon, hydrogen, oxygen, and nitrogen GFISs have been generated [4,5]. The Xe-GFIS also exhibits a very narrow beam with a half opening angle of ~0.5°. The ion current stability is good (instability ~2%). The reduced brightness of Xe-GFIS is measured to be 1.3 x 10^8 $Am^{-2}sr^{-1}V^{-1}$ at the gas pressure of 10^{-4} torr, 3 orders of magnitude higher than that of Ga-LMIS and several orders of magnitude higher than that of Xe magnetically enhanced inductively coupled plasma ion source (5.4x10^3 $Am^{-2}sr^{-1}V^{-1}$) [6]. In principle, the brightness of the Xe-GFIS can be further enhanced at a higher gas pressure or by using an emitter of a larger radius. The operation temperature can be ~200 K, which is much higher than the cryogenic temperature required for HIM. Thus Xe-GFIS-FIB would be easier to implement than HIM and may become a powerful tool for nanoscale milling and secondary ion mass spectroscopy.

[1] Ward, B.W., J.A. Notte, and N.P. Economou, J. Vac. Sci. & Technol. B 24, 2871 (2006).

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[3] H.-S. Kuo, I.-S. Hwang, T.-Y. Fu, J. Y. Wu, C. C. Chang & T. T. Tsong, Nano Lett. 4 2379 (2004)

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[6] N. S. Smith et al., J. Vac. Technol. B 24, 2902 (2006).

8:40am **HI+NS-ThM3 New Ion Source for Nanofabrication and Microscopy**, ***Adam Steele,*** *B. Knuffman, A. Schwarzkopf,* zeroK NanoTech Corporation*; J.J. McClelland,* National Institute of Standards and Technology (NIST) **INVITED**

Performance measurements from a recently constructed focused ion beam (FIB) prototype that employs a new ion source technology will be presented. The performance of any FIB system, and hence the tasks to which it is best suited, are typically determined by its ion source. The high brightness and low energy spread of the Low Temperature Ion Source (LoTIS) employed here has the potential to enable significantly smaller focal spot sizes across a range of beam currents and beam energies in an optimized FIB.

The LoTIS consists of a laser-cooled atomic beam of cesium which is compressed and then photoionized within a volume of a few cubic micrometers. A uniform electric field is applied to form an ion beam. The micro-kelvin temperature of the neutral atoms results in a Cs^+ beam with a low intrinsic transverse velocity spread, yielding low emittance. The small energy spread is determined in this source by the finite spatial extent over which ions are created in a uniform electric field of approximately 10^5 V/m. Previous measurements have shown was shown that LoTIS can achieve a brightness in excess of 1×10^7 A m^{-2} sr^{-1} eV^{-1} and an energy spread less than 0.34 eV [1].

This brightness and energy spread imply that, when coupled to an optimized ion acceleration and focusing column, a d_{50} spot size of 1 nm should be achievable at 1 pA. The source has also achieved total currents over 5 nA, albeit at a reduced brightness. Among other benefits, these source characteristics are expected to enable a FIB with better nanomachining performance and reduced subsurface damage.

The presentation will also briefly discuss FIB equipped with a similar Li^+ ion source technology that offers a unique capability to site specifically deposit lithium into target substrates.

[1] B Knuffman, AV Steele, and JJ McClelland. *J. Appl. Phys*. **114**, 4 (2013).

9:20am **HI+NS-ThM5 Recent Liquid Metal Ion Source developments for Improving Focused Ion Beam Machines**, ***Jacques Gierak,*** LPN-CNRS, Route de Nozay France*; L. Bischoff,* Helmholtz-Zentrum Dresden-Rossendorf, Institute of Ion Beam Physics and Materials Research, Germany*; P. Mazarov, L. Bruchhaus,* Raith GmbH, Germany*; P. Lozano, C. Perez Martinez,* Massachusetts Institute of Technology **INVITED**

Nowadays Focused Ion Beams (FIBs) machines have become very important tools capable of fulfilling many challenges ranging from micro- to nanofabrication. These tools are widely used both for industrial[1] and emerging nanosciences applications[2].

Traditionally FIB technology has been mainly based on gallium Liquid Metal Ions Source. The very high brightness, long lifespan, small source size of the gallium LMIS, and its easy handling, remain its chief and most decisive advantages, but some weaknesses are also well known that inhibit improvements in the resolution of LMIS-based FIB. Therefore progress on ion sources operational characteristics remains very desirable.

In this presentation we will first summarize our recent efforts aiming at optimizing gallium LMIS "needle type" within a dedicated environment for stable operation at lowest possible emission currents. This effort and the important performance gains[3], we will detail, are a firm evidence that progresses can still be expected from this technology.

We will then review and detail the advantages of Liquid Metal Alloy Ion Sources (LMAIS) that represent a promising alternative to expand the already remarkable application field of FIB machines. Incoming ion species are found to influence significantly the properties of FIB-patterned nanostructures, in particular their electrical, optical, magnetic, and mechanical properties. A selection of the best suited elements transported in a focused ion beam will open new nanofabrication routes. In this presentation we will explain how nearly half of the elements of the periodic table can be made available to the FIB technology as a result of continuous research in this area during the last forty years[4] and how, in our opinion, nanotechnology can now take benefit of these.

Finally we will introduce Ionic Liquid Ion Sources (ILIS) that are capable to produce ion beams through field-evaporation from room temperature molten-salts[5]. The possibility of extracting both positive and negative ions having a composition that can be tuned by the selection of the polarity, the liquid chemical composition, the ion emission current and the ion landing energies represents a formidable perspective for FIB technology.

In conclusion we will summarize our vision on the future of FIB technology with improved performances, versatility and on the science frontiers it might help to push.

11:00am **HI+NS-ThM10 Elucidating the Directed Nanoscale Transformations when Building with Ions in Liquid**, *A. Ievlev, V. Iberi, J. Jakowski, M.J. Burch, H. Hysmith, A. Belianinov, R.R. Unocic,* ***Olga Ovchinnikova,*** Oak Ridge National Laboratory

In-situ direct writing by ion beams from solutions opens a pathway for resistless fabrication of nanostructures with higher purity than standard gas phase deposition approaches like IBID. In particular the use of the helium ions with the opposite charge and shorter mean free path offers the potential for the localization of the reaction zone on the single digit nanometer scale. However, to fully control the interaction of the ion beam with the liquid to allow for single digit fabrication a comprehensive understanding of the radiolytic process as well as role of secondary iSE generated in solution has to be developed. Here we will present our results on the visualizing nanoparticle nucleation and growth parameters through data analytics on acquired in-situ growth movies and correlate these results to a fully encompassing time-dependent quantum dynamical simulation that takes into account both quantum and classical interactions. Additionally, with optimized instrument parameters and solution chemistry we are able to demonstrate writing of platinum structures from liquid (beam induced electroplating) in a platinum chloride solution using helium ions with sub-10 nm resolution. Furthermore, we will discuss opportunities for using in situ flow cell technology for understating of diffusion processes as they relate to direct writing with ions in solution.

Acknowledgements

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

11:20am **HI+NS-ThM11 Determination of an Upper Limit of Ionization Probability during SIMS Experiments using Laser Post-ionization**, ***Nicholas Popczun,*** *L. Breuer,* Pennsylvania State University*; A. Wucher,* University of Duisburg-Essen, Germany*; N. Winograd,* Pennsylvania State University

The prospect of secondary ion mass spectrometry (SIMS) as a method of molecular imaging and molecular depth profiling of organic materials has grown with the implementation of polyatomic primary ion sources. These sources increase the total sputter yield and reduce chemical damage, creating a phenomena where the rate of damage removed by the primary ion beam exceeds the rate of damage created. Improving the sensitivity for molecular imaging and molecular depth profiling further relies on increasing the secondary ion yield of the molecular species. The most obvious suggestion to accomplish this is to increase the ionization probability, which has been estimated to be as low as 10^{-7} for atomic primary ion sources. Our lab has developed a method of directly measuring the total ionized and neutral sputtered molecular species located in the same volume sensitive to extraction. This measurement is accomplished by rastering a mid-IR femtosecond pulse for laser post ionization (LPI) of secondary neutral molecules in a two-dimensional plane perpendicular to the direction of laser propagation. Here, we apply this technique for the first time to organic molecules coronene and guanine. Two-dimensional representations of the spatial distribution of neutral molecular species sputtered by C_{60}^+ bombardment are presented. Correction for undersampling of the laser volume and subsequent photofragmentation yields an upper limit for the ionization probability for each molecule. In general, this work provides a visual representation of the spatial distribution of sputtered, organic neutral molecules, delivering additional information for the improvement post-ionization techniques.

11:40am **HI+NS-ThM12 Studying Gas Cluster Ion Beam Sputter Yields and Surface Topography in the Helium Ion Microscope**, ***Anders Barlow,*** *N. Sano, J.F. Portoles, P.J. Cumpson,* Newcastle University, UK

The applications of ion beams in surface analysis are large and clear in the recent literature. In our multi-user facility most projects benefit from the use of an ion beam processing step, whether for cleaning or oxide removal prior to chemical analysis, or for sputter depth profiling through an interface or layer. In our facility we have access to a number of different ion beams: argon monoatomic and gas cluster ion beams (GCIB), C_{60} ion beams, and on our ORION NanoFab, helium/neon ion beams and a gallium focussed ion beam (FIB). These beams serve numerous purposes, from cleaning of surfaces prior to chemical analysis in X-ray photoelectron spectroscopy (XPS) and time-of-flight secondary ion mass spectrometry (ToF-SIMS), to depth profiling in these techniques, to imaging and FIB milling. In all cases however, the ion beam is interacting with the surface under analysis, and this interaction needs to be studied and well-understood. In chemical analysis such as XPS and ToF-SIMS, this requires knowledge of damage mechanisms that impact the reported chemistry from the technique [1]. Understanding how the ion beam can generate nanoscale topography that directly affects the measurements is paramount [2,3].

We are applying helium ion microscopy (HIM) to studying how the ion beams on our instruments change the surfaces we are analysing. The ultra-high resolution of the HIM allows us to see nanoscale topography on surfaces with new-found sharpness at very high magnification, elucidating the mechanisms behind topography formation during treatment. We have investigated the GCIB etching of indium phosphide (InP) using 8keV Ar_{300} clusters (i.e. 300 Ar atoms per cluster). InP is known to generate significant topography following ion beam irradiation. We observe with a spectacular level of clarity the mechanisms behind topography formation, surpassing other commonly used imaging techniques such as scanning probe and scanning electron microscopy. We can also relate the stages of nanotopography growth with total ion beam dose, from a single GCIB etch crater. With this new technique we can more confidently relate the results we obtain from XPS and ToF-SIMS with the topography we observe in the HIM.

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Advanced Ion Microscopy Focus Topic
Room 104A - Session HI+MI+NS-ThA

Ion Beam Based Imaging and Nanofabrication

Moderators: Jacques Gierak, LPN-CNRS, Shinichi Ogawa, AIST, Japan

2:20pm **HI+MI+NS-ThA1 Mask Repair Technology using Gas Field Ion Source**, ***Anto Yasaka***, *F. Aramaki, T. Kozakai, O. Matsuda,* Hitachi High-Tech Science Corporation, Japan **INVITED**

We developed a new ion beam based mask repair system using a gas field ion source (GFIS). For conventional photomasks, nitrogen ions were used to repair defects, while hydrogen ions were used for EUVL masks. We evaluated the performance of the mask repar system on MoSi based phase shift masks and EUV masks. The results demonstrates that GFIS technology is a reliable solution of repairing defects on high end photomasks for 1Xnm generation and beyond.

3:00pm **HI+MI+NS-ThA3 Application of an Advanced Bi Cluster LMIS for TOF-SIMS Analysis at the Nano-scale**, *F. Kollmer, W. Paul, D. Rading, R. Moellers,* ION-TOF GmbH, Germany*; N.J. Havercroft,* ION-TOF USA*; E. Niehuis,* ***Julia Zakel,*** ION-TOF GmbH, Germany

In recent years, the application of cluster primary ions has become standard for all kinds of TOF-SIMS applications. Organic surfaces, in particular, benefit from the cluster bombardment due to a more efficient emission of molecular species compared to mono-atomic bombardment. However, the ultimate spot size so far has been obtained by Ga based liquid metal ion sources. In our contribution we will show that a Bi based cluster LMIS has the potential to outperform the established Ga LMIS even in terms of TOF-SIMS imaging at the highest lateral resolution.

We will discuss fundamental emission properties such as energy spread and virtual source size for the main species of a Bi cluster LMIS. Via a consistent optimisation of emission parameters and an adaption of the ion-optical column, a lateral resolution in the 20 nm range can be achieved. At this scale it seems that we are approaching the physical limits since not only the primary ion beam spot size, but also the size of the sputter cascade as well as the signal intensity limit the obtainable useful lateral resolution. Further progress requires the combination of the SIMS data with complimentary imaging techniques of higher lateral resolution or sophisticated sample preparation methods such as bevelling of the surface region with an FIB column.

In this respect we will show that a combined TOF-SIMS Scanning Probe Microscopy (SPM) provides the required information on the nanometer level. Moreover, information on surface topography and other physical properties of the scanned surface area can be obtained in-situ. The investigated samples include inorganic reference samples, alloys, biological samples, hybrid sample systems and thin films.

3:20pm **HI+MI+NS-ThA4 Nanoscale Imaging and Characterization of Interface Driven Assembly of Soft Materials via He-Ion Beam Microscopy**, ***Matthew Burch***, *A. Belianinov, D. Chang, Y. Luo, K. Hong, O.S. Ovchinikova,* Oak Ridge National Laboratory

The ability to directly image and characterize nanoscale structures and features of soft materials is key to understanding the role growth, interfaces and extrinsic stimuli have on the functionality of these materials. In particular, the arrangement and architecture of bottlebrush block copolymer systems is of interest, as material properties depend greatly on the organization and interfaces of these polymers during growth. However, due to the insulating nature of these materials, directly imaging surface features at the nanoscale using traditional electron microscopy based techniques is challenging. Alternatively, He-ion beam microscopy (HIM) has been developed to the level where it can now characterize and directly image the nanoscale surface features of these soft materials directly.

In this work, He-Ion microscopy is utilized to investigate the nanoscale structures of copolymer systems. In particular, the ordered periodic structures of bottle brush copolymer thin film systems are investigated to understand how different substrates and growth conditions impact the final periodic lamella and domain structures. Of particular interest is how the interface driven separation leads to different short range molecular and long rage surface ordering. Furthermore, we will discuss how surface ordering of the copolymers effects the functionality of the material by correlating HIM imaging results with local probing of electromechanical and electrochemical using scanning probe microscopy.

Acknowledgements

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

4:00pm **HI+MI+NS-ThA6 Advances in *Ex Situ* Lift Out and Manipulation Techniques for FIB Applications**, ***Lucille Giannuzzi***, EXpressLO LLC

The focused ion beam (FIB) *ex situ* lift out (EXLO) technique was the first lift out technique developed for transmission electron microscopy (TEM), surface science, and other site specific analysis of materials [1,2]. EXLO is well known for its ease, speed, and reproducibility, and is perfectly suited for manipulation of thick or electron transparent thin specimens for site specific microscopy or analytical characterization. EXLO is also perfectly suited for manipulation of electron transparent specimens to MEMS carrier devices used for *in situ* TEM holders. Micromanipulation techniques also aid in specimen preparation for particulates and fibers that require subsequent FIB milling. A review of EXLO and advances of the technique using a new slotted grid specimen carrier will be presented. This new grid negates the need for a carbon film specimen support and allows for additional specimen FIB milling or other post processing after manipulation [3].

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[2] F.A. Stevie et al., *Surf. Interface Anal.* 31 (2001) 345–351.

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4:20pm **HI+MI+NS-ThA7 Helium Ion Microscopy Imaging of Carbon Nanofoams from Hydrothermal Carbonization of Sucrose**, ***Natalie Frese***, Bielefeld University, Germany*; S.T. Mitchell, A. Bowers, K. Sattler,* University of Hawaii*; A. Gölzhäuser,* Bielefeld University, Germany

Carbon nanofoam is considered as potential hydrogen storage material as well as cathode material for metal-air batteries. It is known that carbon nanofoam contains both sp^2- and sp^3-bonded carbon atoms. However, there is still a lack of knowledge about the atomic structure of this material. In this work, different types of carbon nanofoams were produced by low-temperature hydrothermal processing of carbon precursor materials. It was found that the produced foams have a low density and are uniform in their appearance. Helium-ion microscopy, X-ray photoelectron spectroscopy and Raman spectroscopy were used to characterize the foam samples. The results show good consistency between the micro- and nanostructure as well as the elemental composition. We conclude that hydrothermal processing of carbon precursor materials is an effective method to produce high-quality carbon nanofoams of graphitic nature.

4:40pm **HI+MI+NS-ThA8 Nanofabrication Limits in Layered Ferroelectric Semiconductors via He-ion Beam**, ***Alexei Belianinov***, *A. Ievlev, V. Iberi, H. Hysmith, M.A. Susner, M. McGuire, S. Jesse, S.V. Kalinin, O.S. Ovchinnikova,* Oak Ridge National Laboratory

Manipulating matter at progressively finer and ultimately atomic scales enables new functionality and effectively drives nanoscience. Currently, well understood, robust resist-based lithography, carries the brunt of nanofabrication, however local electron, ion and physical probe methods are improving as well, driven largely in part of their ability to fabricate without multi-step preparation processes that contaminate the sample with processing resists and solvents. Furthermore probe based methods extend beyond nanofabrication to nanomanipulation and imaging, vital ingredients to rapid transition to testing and manufacturing of layered 2D heterostructured devices.

In this work we demonstrate chemical and physical changes induced by a helium ion beam in a Helium Ion Microscope (HIM) with the surface of bulk copper indium thiophosphate (CITP) $CuM_{III}P_2X_6$ (M = Cr, In; X= S, Se) library of compounds of varying copper concentration; from 4–100%. Physical changes in micro- and nano-fabrication are explored via Atomic Force Microscopy, (AFM), and chemical changes are probed by Secondary Ion Mass Spectrometry, (SIMS). Our work illustrates controlled loss of ferrielectric domains, and nanostructure growth with material volumes scaling to the dosage of the helium ion beam. The nanostructures are oxygen rich, sulfur poor, with the copper concentration virtually unchanged. Effects of varying copper concentration on the quality of the fabricated nanostructures, as well as the differences in their chemical make-up will be discussed.

Acknowledgements

Research was supported (A. B., V. I., A. I., H. H., S. J. S. V. K. O. S. O) and partially conducted (AFM, HIM, SIMS) at the Center for Nanophase Materials Sciences, which is sponsored at Oak Ridge National Laboratory by the Scientific User Facilities Division, Office of Basic Energy Sciences, US Department of Energy. This work was also supported (M. S., M. M.) and

partially conducted (material growth) by the U.S. Department of Energy, Basic Energy Sciences, Materials Sciences and Engineering Division.

5:00pm **HI+MI+NS-ThA9 Focused Ion Beam Technology Challenges for Circuit Edit**, ***Yuval Greenzweig***, *Y. Drezner, A. Raveh,* Intel Corporation

The challenges of Circuit Edit (CE) using focused ion beam (FIB) are driven by the perpetual down-scaling of minimum features per VLSI process technology generation. The recent emergence of FIBs with much reduced probe sizes relative to Ga LMIS based tools, may provide a long-needed revitalization of FIB nanomachining capabilities such as FIB image resolution and machining acuity, necessary for nanomachining tasks such as CE. However, other requirements must go along, driving preferences of ion species, ion energies, and requirements for system performance in several areas.

Among the challenging requirements of CE is the task of milling in a controlled and planar fashion through layers of parallel metal lines with intervening dielectric, and end-pointing on a metal layer of choice. The end-pointing is based on the real-time secondary electron (SE) image during ion milling, and the requirement is leaving most of the target metal intact. If linear dimensions of features, such as minimum metal widths, reduce by a factor $\boldsymbol{\alpha}$ from one VLSI generation to the next, then maintaining quality realtime milling images, i.e., sufficient resolution and signal to noise, requires milling vertically through a layer proportional to $\boldsymbol{\alpha}^{-2}$ relative to the previous generation - same number of ions, but in a smaller pixel. On the other hand, the vertical thickness of the metals has also decreased by a, causing the etching to scale as $\boldsymbol{\alpha}^{-3}$ relative to the thinner new metal thicknesses. Previous VLSI process generation scaling factors have been approximately $\boldsymbol{\alpha}$ = 0.7, so that the severity of this challenge has been getting worse by ~3X for several generations and is now at the feasibility limit. To improve on this, SE emission and collection efficiency must improve, and in particular SE collection efficiency of normally emitted SEs, which are the bearers of the information from the bottom of these milling boxes. The figure of merit representing this challenge is the SE yield times the SE detector collection efficiency, divided by the sputter yield (or etch rate), this provides opportunity for GFIS sources.

Other challenges of the CE application which providing preferences of ion species and ion energy will be discussed.

5:20pm **HI+MI+NS-ThA10 Ion-milling of Graphene Nanostructures While Supported and Unsupported: Considerations of Graphene Contamination, Substrate Scattering and Beam Tailing**, *J. Swett,* Lockheed Martin Space Systems Company*; V. Iberi, D. Cullen,* ***Adam Rondinone****,* Oak Ridge National Laboratory

Graphene and other 2D materials offer novel characteristics and opportunities compared to traditional thin films. Common nanofabrication techniques including e-beam and nanoimprint lithography can be used to pattern atomically thin 2D systems but the multi-step processes they utilize result in exposure of the film to solvents and resists, and hence degradation of the material's novel electronic properties. Herein we demonstrate that helium and neon-ion milling are effective tools for the creation of very fine features with arbitrary geometries in supported and unsupported graphene, to include conductive structures, arrays of pores, and engineered defects. Properties of graphene, including contamination levels, play an important role in determining millability, as do instrumental parameters such as beam tailing and substrate scattering.

Acknowledgement

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

5:40pm **HI+MI+NS-ThA11 Interaction of Gas Field Ionized Nitrogen with Silicon**, ***Marek Schmidt***, *Y. Oshima, L.T. Anh, X. Zhang, T. Kanzaki, M. Akabori,* Japan Advanced Institute of Science and Technology, Japan*; A. Yasaka,* Hitachi High-Tech Science Corporation, Japan*; M. Muruganathan, T. Shimoda, H. Mizuta,* Japan Advanced Institute of Science and Technology, Japan

A larger number of gas molecules (among them helium, nitrogen and neon) can be ionized by the gas field ion source (GFIS) and used as projectiles in focused ion beam (FIB) systems. Among them, the nitrogen stands out as it forms a very strong covalent bond. It is not yet fully understood how this N_2 molecule behaves during field ionization and sample interaction, i.e. if and when the bond is broken. Previously, it has been shown that cross section studies are very useful in analyzing beam/sample interaction [1]. Here, we report scanning transmission electron microscopy (STEM) analysis of cross sections extracted from silicon bombarded with ionized N_2 molecules. The extracted implantation depths for ion energies of 25 and 16 keV are compared with theoretical values and suggest that the bond is broken during sample interaction. We use first principle molecular dynamics simulation to support this finding, in particular that the covalent bond is broken within the first few atomic layers of the impinged silicon target.

All nitrogen ion implantation was carried out in the GFIS-FIB nanofabrication system [2] located at the Japan Advanced Institute of Science and Technology. Line implantation was carried out on cleaned silicon. Following the cross section preparation STEM observation was conducted. For the 25 keV beam, an implantation depth of ~75 nm is observed, while this decreases to ~35 nm for the case of 16 keV. These values match the theoretically predicted values for the case that two nitrogen atoms are ionized with a single charge (N_2^+), and split upon impact. The splitting is also predicted by the molecular dynamics simulation we conducted.

These results help to give a clearer picture of the nitrogen ionization in a GFIS and the resulting beam. After ionization of the N_2 molecule through electron tunnelling into the atomically sharp emission tip, the ion is accelerated to the energy $E = E_0$and focused onto the sample. Upon interaction with the sample surface, the covalent bond is momentarily split. Consequently, each of the nitrogen atoms has only half of the energy $E = E_0/2$. The ion charge is dissipated in the substrate by transfer of an electron.

The help of M. Uno with the usage of the GFIS-FIB is acknowledged. The authors thank M. Ito for the help with TEM cross section preparation. This work is supported by the Center Of Innovation (COI) program of the Japan Science Technology Agency.

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6:00pm **HI+MI+NS-ThA12 Spatially Controlled Ripple Formation in the HIM using Low Voltages and High Temperatures**, ***Gregor Hlawacek***, *L. Sottili, M. Engler, S. Facsko,* Helmholtz-Zentrum Dresden Rossendorf, Germany

Ripple formation is a well known phenomenon that is observed for many materials under low energy ion bombardment. Often broad beam noble gas ion irradiation using energies of a few keV is employed to create these self-organized patterns on various metal, semiconductor and insulator surfaces. In addition to the fundamental interest in the formation and evolution of these structures they can be utilized in a number of new applications. Creating nano scale periodic roughness can be of interest for various microfluidic applications or to control friction in new MEMS and NEMS devices. However, these applications are not realized at their full potential today as the required sub micron patterning which can not easily be realized using broad beams.

Here, we present for the first time ripple patterns that have been created on the GaAs(001) surface using 5 keV Ne ions and elevated temperatures of up to 600 K in a Helium Ion Microscope (HIM). We will present the home built sample heater that can be loaded through the load lock of the Carl Zeiss Orion NanoFab and describe the influence on the device performance, as well as HIM operation at 5 keV.

The evolution of the ripple wavelength changes from 30 nm at low 1e17 Ne/cm² to 80 nm at 1e18 Ne/cm². The orientation of the ripples with respect to the shape can be changed by rotating the pattern on the surface and the influence of the geometrical constrains of the irradiated area on the ripple pattern is studied.

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

Aspects of Advanced Ion Microscopy Poster Session

HI-ThP1 Gas Field Ion Sources from Single-Atom Tips*, W.T. Chang,* *C.Y. Lin, W.C. Lai, Y.F. Yu,* Institute of Physics, Academia Sinica, Taipei, Taiwan, Taiwan, Republic of China*; T.Y. Fu,* National Taiwan Normal University, Taipei, Taiwan, Taiwan, Republic of China*; T.T. Tsong, I.S. Hwang,* Institute of Physics, Academia Sinica, Taipei, Taiwan, Taiwan, Republic of China

Focused ion beams (FIBs) have been widely used in a large number of applications, such as high-resolution scanning ion microscopy, lithography, nanofabrication, secondary ion mass spectroscopy (SIMS), serial sectioning tomography, etc. Current FIB systems have relied on the high brightness, moderate energy spread, ease-of-use, and robustness of the gallium liquid metal ion source (LMIS). One of the main drawbacks of LMIS-FIB system s is the inevitable contamination of liquid metal i n materials . To extend the applications of FIB technology, it is essential to develop high-brightness ion sources of various species because different ion beams serve different purposes. It has been well recognized that the brightest ion sources are gas field ion sources (GFISs). The virtual source size (~1 nm or smaller) and the energy spread (<1 eV) of GFISs are at least one order of magnitude smaller than those of LMISs. This implies GFIS-FIB systems can achieve a much better resolution than the current Ga-FIB systems. Another important advantage of GFIS-FIB systems is that the same emitter is capable of producing different ion beams simply by changing the gas species.

Since 2006, Zeiss Orion helium ion microscope (HIM) [1] successfully demonstrated a spatial resolution better than 0.5 nm. The HIM uses a pure tungsten trimer tip as the emitter and two ion species, He^+ and Ne^+, can be stably emitted. Here we present another type of GFIS emitters, n oble-metal covered tungsten single-atom tips (SATs), which can reliably produce a wider variety of ion species. These SATs are thermally stable and chemically inert, and thus can be generated and regenerated through annealing [2]. Helium, neon, argon, hydrogen, oxygen, nitrogen, and recently xenon ions have been emitted from this type of SATs. Light ions have the lowest sputtering rates and are beneficial for scanning ion microscopy. Heavy ions can provide a high sputtering rate, suitable for ion milling. Due to the high secondary ion yields, oxygen and xenon ion beams can be applied to SIMS. These SAT-GFISs have a half opening angle ~0.5° and a brightness several orders of magnitude higher than that of Ga-LIMS. The ion current of these SAT-GFISs are very stable (instability <5%). Since these SATs can be regenerated for more than 50 times, therefore their lifetimes are sufficiently long for most practical applications.

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Author Index

Bold page numbers indicate presenter