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.

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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

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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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