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.

Author Index

Bold page numbers indicate presenter