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].

[1] L.A. Giannuzzi et al., Mat. Res. Soc. Symp. Proc. 480 (1997) 19-27.

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

[3] L.A. Giannuzzi et al., *Microsc. Microanal*. 21 (2015) 1034-1048.

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.

[1] R. H. Livengood et. al, *Nucl. Instrum. Methods Phys. Res. Sect. Accel. Spectrometers Detect. Assoc. Equip.*, vol. 645, no. 1, pp. 136–140, Jul. 2011.

[2] F. Aramaki et. al, in *Proc. SPIE 8441*, Yokohama, Japan, 2012, vol. 8441, p. 84410D–84410D–6.

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.

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