

Thursday Afternoon, October 22, 2015

Helium Ion Microscopy Focus Topic
Room: 211B - Session HI+AS+NS-ThA

Imaging and Milling with He and Ne Ion Beams

Moderator: Richard Livengood, Intel Corporation, John A. Notte, Carl Zeiss Microscopy

2:20pm **HI+AS+NS-ThA1 Focused Ion Beam Circuit Edit in the Nano-Device Age: A Search for the Ultimate Nano-Ion Beam, Shida Tan, Intel Corporation** **INVITED**

Evolution of the IC process technology continues to increase the challenge of circuit edit with smaller critical device dimensions, thinner process layers, densely packed structures, and complex device routing and design architecture. In this paper, the general approach employed, challenges encountered, and results acquired in neon application development using Zeiss NanoFAB (noble GFIS) platform for circuit edit will be presented. The merits and limitations of applying a Ne^+ beam in high precision circuit edit applications will be shared with the audience.

3:00pm **HI+AS+NS-ThA3 Electrical Nano-Patterning of Graphene Film by Helium Ion Beam Irradiation, Shinichi Ogawa, T. Iijima, Y. Naitou, AIST, Japan**

The helium ion microscopy is a unique technology for observation of soft materials such as low-k materials and photo resist patterns for LSI fabrication [1] and for nm order etch patterning. Graphene, a two-dimensional sheet of carbon atoms [2], is a promising channel material for next-generation transistors, and we have shown an on-off gating of current through a graphene nano-ribbon which was etched down by the helium ion beam using the helium ion microscope [3] and by controlling electrical properties of the graphene films themselves by the nano-scale helium ion irradiations generating defects [4]. This study precisely shows a nano-scale direct electrical patterning of the graphene film from a point of view of the irradiation conditions.

Helium ion beams of 0.3 nm diameter were 500 X 500 nm² regions scanned on single-layer graphene films on Si/SiO₂ substrates at doses of 0.62 - 10 10¹⁶ cm⁻² at 30 kV. A spatial resolution of the helium ion microscope patterning on the single-layer graphene was investigated by fabricating nano-ribbons of lines of 100 - 5 nm width. Dynamic force microscopy and scanning capacitance microscopy measurements revealed that helium ion irradiated regions appeared as depressed about 1 nm in topography image at above conditions, while a darker image was acquired at 2.0 10¹⁶ cm⁻² than those at less dose conditions which means transition to dielectric from metal occurred at the higher dose. A spatial resolution of the helium ion microscope patterning non-monotonically depended on the dosage of the helium ions. Increasing the dose to 5.0 10¹⁶ cm⁻² improved the spatial resolution to several tens of nanometers. However, doses of more than 1.0 10¹⁷ cm⁻² degraded the patterning characteristics.

Those results are discussed precisely changing irradiated beam conditions followed by structural analyses by such as CS-TEM.

[1] S. Ogawa, et al., Jpn. J. Appl. Phys., 49 (2010) 04DB12, [2] K. Novoselov, et al., Science 306, 666 (2004), [3] S. Nakaharai, et al., Appl. Phys. Express 5 015101 (2012), [4] S. Nakaharai, et al., 2012 IEEE International Electron Devices Meeting (IEDM), Technical Digest p.72 (2012), [5] Y. Naitou, et al., Appl. Phys. Lett. 106, 033103 (2015)

3:20pm **HI+AS+NS-ThA4 Nanopores in Silicon Nitride Membranes, Graphene and CNM: Milling and Imaging Techniques at the Helium Ion Microscope, Daniel Enmrich, E. Marschewski, Bielefeld University, Germany, A. Nadzeyka, F. Nouvertné, Raith GmbH, Germany, A. Götzhäuser, A. Beyer, Bielefeld University, Germany**

The Helium Ion Microscope (HIM) is a focussed ion beam system which can be used for both imaging and milling. In the low dose regime, the HIM operates as a microscope; high doses enable material modification and sputtering. Compared to conventional focussed ion beams (FIB), the HIM offers a very small focal spot size down to 0.35 nm and a strongly localized sputter interaction with the material. We employ the HIM for milling nanopores in free standing membranes, such as 30 nm thick Silicon Nitride, Graphene and 1 nm thick carbon nanomembranes (CNM) made from aromatic self-assembled monolayers by electron-induced cross-linking. HIM is also used for the inspection of pores. The smallest He⁺-milled nanopores have a diameter of about 3 nm in all investigated membranes. The He⁺ beam thus overcomes the resolution limit of conventional FIB tools as we show in a comparison with a high resolution Ga-FIB. Different strategies for the characterization of pores with the HIM will be discussed.

In particular, we compare the feasibility of the ion generated secondary electron signal to the He⁺ transmission signal.

4:00pm **HI+AS+NS-ThA6 Application of Focused Helium Ion Beams for Direct-write Lithography of Superconducting Electronics, Shane Cybart, University of California San Diego** **INVITED**

The 1986 discovery of high transition temperature (high- T_c) superconductivity in copper-oxide materials set in motion an intense research effort to develop superconducting electronics functioning in the range of liquid nitrogen temperatures (77 K). Scientists and engineers soon after discovered that these materials were much more complicated than initially imagined. Anisotropic electrical properties and a very short superconducting coherence length seriously narrowed or eliminated the possibility of using classical superconducting electronic structures. These new materials demanded novel device architectures that proved very difficult to realize. Nearly three decades have passed and progress in high- T_c superconducting devices has been very slow because process control at the sub ten nanometer scale is required to make high quality, reproducible Josephson junctions: the basic building block of superconducting electronics. Recent advances in gas field focused helium ion beams provide a new and promising approach for direct-write lithography of high- T_c materials for the realization of predictable and scalable high- T_c electronics. In this work, we demonstrate fabrication of a - b plane superconducting Josephson tunnel junctions for YBa₂Cu₃O_{7- δ} (YBCO) by utilizing a focused helium ion beam to create a narrow (~nm) in-plane tunnel barrier between two superconducting electrodes. The key to this method is that YBCO is sensitive to point defects in the crystal lattice caused by ion irradiation. Increasing irradiation levels has the effects of increasing resistivity and reducing the superconducting transition temperature. At very high irradiation levels YBCO becomes insulating and no longer superconducts. Test samples were written with ion fluence ranging between 10¹⁴ and 10¹⁸ He⁺/cm². In between these two extremes we were able to determine doses that could create very high-quality Josephson junctions with both metallic and insulating barriers. The current-voltage (I-V) characteristics for lower doses show nearly ideal Josephson junction behavior with a zero voltage supercurrent that oscillated in magnetic field as expected for the Josephson effect. At much higher doses (I-V) exhibited insulator behavior. Using ac techniques we measured the differential conductance (dI/dV) in this regime which revealed the YBCO superconducting energy gap near 33 mV. This feature is a result of quasi particle tunneling which provides strong evidence that we have created an insulating barrier less than 2 nm wide. These results demonstrate the unique ability of focused helium ion beams for maskless direct write lithography of oxide tunnel barriers for electronic devices.

4:40pm **HI+AS+NS-ThA8 A Novel Efficient Approach for Investigating the Ion Implantation Effect on Small Volume Copper, Zhang-Jie Wang, Xi'an Jiaotong University, China, F.I. Allen, University of California, Berkeley, Z.W. Shan, Xi'an Jiaotong University, China, P. Hosemann, University of California, Berkeley**

Ion implantation has been used for decades to investigate the response of materials to radiation damage. Understanding the effect of He in materials is a key aspect in the optimization of fusion, fast reactor and spallation sources suffering from high He/dpa (displacements per atom) ratios. The traditional large-area He implantation techniques are rather materials-constrained and time consuming, thus limiting systematic studies. The work presented here utilizes the Zeiss ORION NanoFab instrument which deploys He and Ne ion beams in combination with a Ga ion source to quickly and efficiently manufacture nanostructures and then perform direct He implantation in selected areas of interest. Demonstrated in single crystal and copper nanotwinned structures, the systematic study of He bubble lattice distribution and twin structure evolution under different implanting doses and dose rates is achieved in a fast and efficient manner. We also utilize the combined Ga-He beam system to increase sample throughput to manufacture nanopillars and implant with He in the same chamber. Each pillar was subsequently tested using a JEOL 3010 TEM equipped with a Hysitron P195 nanomechanical testing system. The results show that the resistance of deformation twinning in single crystal Cu and twin boundary migration in nanotwinned Cu are both significantly improved for increasing He doses up to 1x10¹⁸He⁺/cm². The novel technique presented here makes it feasible and efficient to evaluate He ion damage and its effect on small volume materials.

5:00pm **HI+AS+NS-ThA9 Helium Ion Microscopy Analysis of Itokawa Asteroid Particles Obtained from Hayabusa Mission, Vaithiyalingam Shuthanandan**, Pacific Northwest National Laboratory, *R.C. Ogliore, K. Nagashima*, University of Hawai'i at Manoa

Particles returned from the S-type asteroid Itokawa by JAXA's Hayabusa mission show evidence of space weathering features. These features can be very small (<1 μm in size) and very shallow (within a 100 nm of the surface). The smallest space-weathering surface features and textures are difficult to resolve by field-emission SEM (FEG-SEM). In order to see these effects, we have used Helium ion microscopy (HIM). Two Hayabusa particles: RB-QD04-0062 (“#62”): 40 μm , olivine and plagioclase) and RB-QD04-0091 (“#91”): 43 μm , olivine and plagioclase) were imaged using HIM. The particles were removed from their glass slides with a Sutter micromanipulator and tungsten needle and then stuck on a SEM stub with a thin layer of Post-It note glue. The stub was coated with ~6 nm of carbon for electrical conductivity. Helium ion microscopy images of the surfaces of two Hayabusa grains revealed diverse space-weathering features on scales from several nm to several μm . Both Hayabusa particles show variable surface textures, a variety of splash melt features, adhering grains, and small holes. Two porous particles, with structures reminiscent of chondritic-porous interplanetary dust particles, were found adhering to the surface of the Hayabusa grains (a 1.2 μm object on #91, a 350 nm object on #62). Much of the surface of #62 was covered with small bumps 25–100 nm in size, whereas other regions were free of small bumps. A large, 6 μm quenched melt splash feature was found on the surface of #62. On the other hand, faces of #91 showed multiple concoidal fractures and splash melt droplets and pancakes, but fewer and smaller surface bumps compared to #62. For comparison purposes a 30 μm grain of lunar soil, which had a much higher density of sub- μm splash melt features than the Hayabusa grains, was also imaged. No obvious impact craters (holes with raised rims) were found on this surface. The variations in surface textures indicate that the grains of Itokawa asteroid experienced a complex history of fracturing and exposure to space-weathering processes on the surface of asteroid Itokawa.

5:20pm **HI+AS+NS-ThA10 Multi-Beam Ion Microscopy and Nanofabrication at UC Berkeley**, *Frances Allen*, UC Berkeley, Lawrence Berkeley National Laboratory (LBNL), Biomolecular Nanotechnology Center/QB3, *P. Lum*, Biomolecular Nanotechnology Center/QB3, *T.C. Pekin*, UC Berkeley and LBNL, *Z.J. Wang*, UC Berkeley and Xi'an Jiaotong University, Republic of China, *R. Thayer*, UC Berkeley, *J. Hong*, UC Berkeley and LBNL, *A.A. Omrani*, UC Berkeley, *M.F. Crommie*, *J. Bokor*, UC Berkeley and LBNL, *N.H. Patel*, UC Berkeley, *A.M. Minor*, UC Berkeley and LBNL, *P. Hosemann*, UC Berkeley

A Zeiss Orion NanoFab Helium Ion Microscope (HIM) has recently been installed at UC Berkeley in the Biomolecular Nanotechnology Center. The specialized gas-field ion source is operated using He or Ne gas and a separate column with a liquid-metal ion source is used to generate a Ga^+ beam. Thus, the advanced imaging and nanofabrication capabilities of the HIM using He^+ and Ne^+ can be combined with the bulk milling capability of Ga^+ enabling a range of imaging and nanofabrication modalities all in one tool.

We will present a selection of initial results from the microscope, highlighting the versatility of this multi-beam instrument and a close collaboration with Transmission Electron Microscopy (TEM) facilities at the National Center for Electron Microscopy in the Molecular Foundry of Lawrence Berkeley National Laboratory for the characterization of NanoFab-fabricated electron-transparent specimens. For example, using Ne^+ and Ga^+ beams and subsequent TEM analysis we explore the effect of Ne^+ milling versus conventional Ga^+ milling of TEM specimens focusing on aluminum alloys and their nanomechanical properties. We use the He^+ beam to selectively implant Ga^+ -milled nanopillars for TEM investigation of He-bubble superlattices and *in situ* TEM nanomechanical testing for a range of implantation doses. Further applications combining fine milling with high-resolution HIM imaging include the fabrication of magnetic multilayer tunnel junction island structures down to a diameter of <10nm, fabrication of MoS_2 nanoribbons, and site-specific cross-sectioning of scales from the wing of the *Junonia coenia* butterfly species for investigations of the development of nanostructures responsible for structural color. In many applications the use of the electron flood-gun for charge compensation when imaging insulating specimens is a crucial component.

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