Wednesday Afternoon, October 21, 2015

Helium Ion Microscopy Focus Topic
Room: 210F - Session HI-WeA

GFIS Based Nanostructuring
Moderator: Shinichi Ogawa, AIST, Tom Wirtz, Luxembourg Institute of Science and Technology (LIST)

2:20pm **HI-WeA1 Nitrogen and Helium Gas Field Ion Source for Nanofabrication**, ***Marek Schmidt***, Japan Advanced Institute of Science and Technology, Japan, *K. Nagahara, O. Takechi, M. Akabori,* Japan Advanced Institute of Science and Technology, *A. Yasaka,* Hitachi High-Technologies Corporation, *T. Shimoda, H. Mizuta*, Japan Advanced Institute of Science and Technology

We report on the status and application of the gas field ion source (GFIS) focused ion beam (FIB) nanofabrication system [1] located at the Japan Advanced Institute of Science and Technology (JAIST). The atomic emission tip is biased against the extractor, generating high electric field strengths at the tip apex leading to ionization of the source gas. The ions are then accelerated to a typical energy of 25 Kv, focused, and scanned over the device under test (DUT). Such beams can be used to mill the DUT or generate images by simultaneous detection of the secondary electrons by the Everhart-Thornley detector. The GFIS-FIB employs a newly designed emission tip technology to use nitrogen ($N_2 \rightarrow N_2^+$) as a high sputter-yield source gas. Other ion species, such as helium and hydrogen, can be generated with the tip technology as well. The latter has been demonstrated to suppress the sub-surface damage formation [2], but is not currently in use at JAIST. Carbon is available for deposition, while iodine, xenon difluoride (XeF_2) and H_2O can be used for gas assisted milling.

Our current efforts involve carving graphene into sub-10-nm wide ribbons and electrically separating gold electrodes on top of graphene with the goal of realizing graphene tunnel field effect transistors. We also work on fabrication of graphene resonators based on suspended graphene that are narrowed down after the resist-based fabrication and hydrofluoric acid release. The second main area is the preparation of nanoimprint lithography (NIL) masters. Line-and-space pattern with a half pitch of 15 nm were milled into quartz substrates by the ion beam and will be applied to the fabrication of cutting-edge semiconductor devices. Lastly, we use the nitrogen ion beam for quantum point contact fabrication (QPC) in high-in-content InGaAs [3]. The size of the QPCs is ~30 nm × 150 nm, and the QPCs show step-like structures of $2e^2/h$ without magnetic field and of e^2/h and $3e^2/h$ with magnetic fields. Additionally, we will show the unique material contrast observed in nitrogen ion excited secondary electron images of graphene-based structures, and talk about low-energy operation, where 15 nm resolution has been achieved with a 5 Kv nitrogen beam. Such low energy beams will be used for defect generation and doping of graphene and other materials.

This work was partially supported by Kakenhi No. 25220904 from JSPS and the Center of Innovation Program from Japan Science and Technology Agency.

[1] F. Aramaki *et al.*, in *Proc. SPIE 8441*, 84410D, Yokohama, Japan, 2012.

[2] F. Aramaki *et al.*, in *Proc. SPIE 7969*, 79691C, San Jose, USA, 2011.

[3] M. Akabori *et al.*, *Jpn. J. Appl. Phys.*, **53**, 118002, 2014.

2:40pm **HI-WeA2 Helium-Ion Milling of Gold Nanoantennas: Toward Plasmonics with Nanometer Precision**, ***André Beyer***, *H. Vieker,* Bielefeld University, Germany, *H. Kollmann,* Oldenburg University, Germany, *X. Piao, N. Park,* Seoul National University, Korea, *M. Silies, C. Lienau,* Oldenburg University, Germany, *A. Gölzhäuser*, Bielefeld University, Germany

Plasmonic nanoantennas are versatile tools for coherently controlling and directing light on the nanoscale. For these antennas, current fabrication techniques such as electron beam lithography (EBL) or focused ion beam (FIB) milling with Ga^+-ions routinely achieve feature sizes in the 10 nm range. However, they suffer increasingly from inherent limitations when a precision of single nanometers down to atomic length scales is required, where exciting quantum mechanical effects are expected to affect the nanoantenna optics. Here, we demonstrate that a combined approach of Ga^+-FIB and milling-based He^+-ion lithography (HIL) for the fabrication of nanoantennas offers to readily overcome some of these limitations (1). Gold bowtie antennas with 6 nm gap size were fabricated with single-nanometer accuracy and high reproducibility. Using third harmonic (TH) spectroscopy, we find a substantial enhancement of the nonlinear emission intensity of single HIL-antennas compared to those produced by state-of-the-art gallium-based milling. Moreover, HIL-antennas show a vastly improved polarization contrast. This superior nonlinear performance of HIL-derived plasmonic structures is an excellent testimonial to the application of He^+-ion beam milling for ultrahigh precision nanofabrication, which in turn can be viewed as a stepping stone to mastering quantum optical investigations in the near-field.

(1) H. Kollmann, X. Piao, M. Esmann, S.F. Becker, D. Hou, C. Huynh, L.-O. Kautschor, G. Boesker, H. Vieker, A. Beyer, A. Gölzhäuser, N. Park, R. Vogelgesang, M. Silies, C. Lienau: Towards Plasmonics with Nanometer Precision: Nonlinear Optics of Helium-Ion Milled Gold Nanoantennas, Nano Letters 14, 4778 (2014)

3:00pm **HI-WeA3 Interactions of Focused Helium and Neon Ion-beams with Nanostructures**, ***Chung-Soo Kim***, *R.G. Hobbs, V.R. Manfrinato, A. Agarwal, K.K. Berggren,* MIT, *D. Wei*, Carl Zeiss NTS

Scaling the dimensions of materials to the nanoscale creates new opportunities and many applications. Nanomaterials display unique properties relative to their bulk counterparts due to surface/interface effects, quantum confinement/coherence, which have been investigated to understand new materials physics. Applications driven by nanostructures can benefit from their enhanced functionalities by modifying material properties and geometries via ion irradiation. Specifically in nanostructures, ion irradiation with a focused ion beam can play a critical role in modifying properties and geometries locally in those nanostructures. This local modification had already been proven to have applications in quantum optics and circuits by creating localized material modification at the nanoscale in bulk materials via focused helium ion beam (FHIB) irradiation However, local modification may occur in different ways in nanostructures. Therefore, one needs to investigate the effect of FHIB irradiation on nanostructures in order to control local modification in a desirable way and understand new physics.

In this work, we primarily study the interaction of a FHIB and nanostructures with two materials, single crystal silicon and diamond. Our experimental approach using thin vertical membranes fabricated by focused gallium ion beam enabled us to observe ion-nanostructure interaction in 3-dimension by preserving defects by ion irradiation. We have investigated new physical phenomena; (1) strain-induced volume expansion, (2) long-range ion propagation, and (3) material behavior's transition from a bulk to a small-scale where the size-dependent characteristic exists. We have explained these mechanisms between nanostructure (material, crystal orientation, and geometry) and helium ion (energy and dose). We have also extended our study to focused neon ion beam (FNIB) irradiation. We have investigated and compared the difference of ion-nanostructure interaction between neon and helium.

Furthermore, we have expanded our study to new nanofabrication method by embedding a 3D geometry on nanostructures with the consideration of a geometrical constraint, decided by crystal-to-amorphous boundary.

3:20pm **HI-WeA4 Polarization Control via He-ion Beam Induced Nanofabrication in Layered Ferroelectric Semiconductors**, ***Alex Belianinov***, *V. Iberi, A. Tselev, M,. Susner, M. McGuire, D.C. Joy, S. Jesse, A.J. Rondinone, S.V. Kalinin, O.S. Ovchinnikova*, Oak Ridge National Laboratory

Abstract

Rapid advanced in nanoscience rely on continuous improvements of manipulating matter at near atomic scales. 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 can result in sample contamination from the 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 that helium ion interaction 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) result in the controlled loss of ferrielectric domains, and growth of cylindrical nanostructures with enhanced conductivity, with material volumes scaling to the dosage of the beam. The nanostructures are oxygen rich, sulfur poor, with the copper concentration virtually unchanged as confirmed by Energy Dispersive X-ray (EDX). Scanning Electron Microscopy (SEM) image contrast as well as Scanning Microwave Microscopy (SMM) measurements suggest enhanced conductivity of the formed particle, whereas Atomic Force Microscopy (AFM) based measurements indicate that the resulting structures have lower dissipation and a lower young's modulus.

Acknowledgements

Research was supported (A. B., V. I., A.T., D. J., S. V. K., S. J., A. J. R. O. S. O) and partially conducted (AFM, HIM) 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.

4:20pm **HI-WeA7 Fabrication of Nanoscale Electronics with a Focused Helium Ion Beam**, ***Ethan Cho***, University of California, San Diego, *M. Ma,* Univeristy of California, San Diego, *C. Huynh,* Carl Zeiss Microscopy, LLC, *R.C. Dynes, S.A. Cybart*, Univeristy of California, San Diego

Since the invention of gas field ion source focused ion beams, researchers have had access to ion sources from inert gases. In particular, the focused helium ion beam (FHB) has a sub-nanometer beam spot that allows the direct patterning of nanometer scale devices without contamination. However, preparing samples with direct patterning is time consuming, especially when milling away large volumes of material. Here we demonstrate patterning a wide range of materials through disordering the crystal structure that only requires a dose orders of magnitude less than the milling dose. Patterning graphene has been difficult due to edge formation in the reactive ion etching step. Using a lower dose to perturb and disorder the carbon atoms would locally change the characteristics of graphene without destroying the integrity of the film. For materials that are hard to grow as a multilayer such as the iron based superconductor FeAs, the properties of conventional stacked Josephson junctions are limited by the film quality. With a FHB we can induce disorder in a very narrow region that suppresses the critical temperature (T_C) within this region to serve as the junction barrier. By removing the multilayer-processing the fabrication steps of these in-plane junctions are greatly simplified, and higher quality junctions are produced. Using disorder to create a lower Tc barrier also works for more metallic superconductors like MgB_2. For materials that undergo a superconductor-insulator transition, as for example cuprates, we demonstrate insulating in-plane tunnel junctions and nano wires. This technique also works for magnetic manganites that are sensitive to disorder. These materials have interesting characteristics at the interface between a multiferroic and magnetic material. Extensive care during the processing phase is required to preserve the interfacial properties. We demonstrate that the interface remains intact after a junction is fabricated with FHB. Most of the materials described above are extremely sensitive to processing, but by only locally altering the properties we eliminate interfacial problems and degradation due to heat generated in removing the material. Direct patterning using disorder induced from the FHB pushes the feature size truly to the nano meter scale, simplifies the fabrication both in time and number of steps, and preserves material integrity throughout the process. This FHB has the potential to be a general technology, and therefore allows us to engineer and study the fundamental physics of a variety of phenomena.

4:40pm **HI-WeA8 Creating and Imaging Nanosized Magnets using HIM and TEM Holography**, ***Gregor Hlawacek***, Helmholtz-Zentrum Dresden - Rossendorf, Germany, *F. Röder,* TU Dresden, *R. Bali, S. Wintz, R. Hübner, L. Bischoff,* Helmholtz Zentrum Dresden Rossendorf, *H. Lichte,* TU Dresden, *K. Potzger, J. Lindner, J. Fassbender*, Helmholtz Zentrum Dresden Rossendorf

Besides imaging, gas field ion source (GFIS) based microscopes [1] are used for materials modification. This usually is based on the use of high fluence to either mill the sample material or implant Nobel gas ions into the target

material [2].

Here, we present a novel route utilizing a Helium Ion Microscope (HIM) to form nano-sized magnets of arbitrary shape using very low fluences ($6x10^{14}$ cm^{-2}) of 20 keV-25 keV Neon ions. The fine Neon beam available in the HIM is used to locally switch 40 nm thin $Fe_{60}Al_{40}$ films from the well ordered paramagnetic B2 structure into the ferromagnetic A2 structure [3,4]. Planar structures potentially useful for applications such as spin valves or other spin--transport devices have been formed this way. Kerr Microscopy and off-axis TEM holography has been used to analyse the resulting magnetic nano-structures. Results on the energy depended depth of magnetization as well as on the lateral definition of the magnetic structures due to scattering are presented.

FR and HL gratefully acknowledge funding from the European Union Seventh Framework Programme under Grant Agreement 312483 - ESTEEM2 (Integrated Infrastructure Initiative - I3).

1. Hlawacek, G., Veligura, V., van Gastel, R. & Poelsema, B. Helium ion microscopy. J. Vac. Sci. Technol. B Microelectron. Nanom. Struct. 32, 020801 (2014).

2. Veligura, V. et al. Digging gold: keV He+ ion interaction with Au. Beilstein J. Nanotechnol. 4, 453–460 (2013).

3. Bali, R. et al. Printing nearly-discrete magnetic patterns using chemical disorder induced ferromagnetism. Nano Lett. 14, 435–41 (2014).

4. F. Röder, et al. Direct Depth- and Lateral- Imaging of Nanoscale Magnets Generated by Ion Impact, submitted (2015).

5:00pm **HI-WeA9 Understanding Device Functionality in CVD-grown $MoSe_2$ Laterally Tuned with a Focused Helium Ion Beam**, ***Vighter Iberi***, *M.-W. Lin, X. Li, A. Ievlev, S. Jesse, S.V. Kalinin, A.J. Rondinone, D.C. Joy, K. Xiao, O.S. Ovchinnikova*, Oak Ridge National Laboratory

The scalability of electronic and information technology devices depends on the ability to tune layered materials. With the recent development of CVD-growth processes for high quality 2-dimensional materials, large scale fabrication has become routine. Monolayer molybdenum diselenide (MoSe2) has become a highly attractive candidate in the fabrication of functional electronic and optoelectronic devices due to its high electron mobility. However, critical is the structuring and functional tuning of these materials, as currently being done for semiconductors. Here, we will discuss the use of focused helium ion beams in tailoring the functionality of MoSe2 electronic devices with nanometer precision. Using a helium ion beam under high dosing allows for milling and structuring of MoSe2 devices with nanometer precision and prevents ion implantation and resist contamination effects. For lower helium ion doses we are able to tune the mobility as ascertained by local transport measurements. The nature of the associated properties of this material were explored using a combination of aberration-corrected scanning transmission electron microscopy (STEM), scanning probe microscopy (SPM) and optical spectroscopy techniques that provided insight into local mechanical, electromechanical, chemical and atomic structure properties of these devices and elucidate the effect of ion beam dose on device performance. Future perspective and scalability of this approach to device fabrication will also be discussed.

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