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).

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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.

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

Focused Ion Beam Technology (08:00-10:00)/Fundamentals of Helium Ion Microscopy (11:00-12:20)
Moderator: Gregor Hlawacek, Helmholtz-Zentrum Dresden - Rossendorf, Leonidas Ocola, Argonne National Laboratory

8:00am **HI+AS+SS+NS-ThM1 Ga+ Ion Beam Nanofabrication Techniques of 3D Micro- and Nano- Fluidic Devices**, ***Leonidas Ocola***, Argonne National Laboratory

Three-dimensional (3D) fluidic geometries have been fabricated in the past by using several layers of Polydimethylsiloxane (PDMS) molds or double-sided Si etch steps [1], which require highly accurate chip bonding to complete the fluid path and multiple process steps. An alternative to this method is the use of direct write ion beam micromachining as a means to fabricate key components of a microfluidic device that require variations in depth as well as variations in width. 3-D microfabrication currently is mainly constrained to excimer lasers [2-3] and therefore is inherently diffraction limited. Grey scale lithography is also used for 3D structures but has limited capability. On the other hand, ion beam micromachining can scale down below the diffraction limit with no change in the technique and almost unlimited depth bandwidth. The focused ion beam / scanning electron microscope (FIB/SEM) is a powerful tool used for sample analysis and characterization. When equipped with a sophisticated pattern generator and lithography technology it can expand its use to new applications in nano- and micro-fabrication. Ion beam micromachining is akin to electron beam lithography, where a beam of charged particles are steered to draw structures contained in a computer aid design (CAD) file. Unlike electron beam lithography, one can program arbitrary depths by manipulating the dwell time, or dose, of a particular structure. In this paper the work reported previously [4-5] has been expanded to large and complex geometries to place emphasis on the applicability of ion beam micromachining to practical microfluidic applications, such as straight 3D mixers and serpentine 3D mixers with sections as deep as 70 microns and channel widths as large as 30 microns. We have found that these devices can achieve full mixing of aqueous solutions in about an order of magnitude faster than traditional devices. The challenges encountered and overcome to fabricate these mixers will be described and the scalability of different fabrication techniques to nano-fluidics will be revisited.

References:

1. R. H. Liu et al., J. MEMS 9 (2000) 190

2. Y. Liao et al., Lab Chip, 12 (2012) 746

3. A. Ródenas et al., Proc. SPIE 8542 (2012) 854217

4. A. Imre et al., J. Vac. Sci. & Technol. B 28 (2010) 304

5. E. Palacios et al., J. Vac. Sci. Technol. B 28 (2010) C6I1

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8:20am **HI+AS+SS+NS-ThM2 Adding 3D to Conventional SEM or FIB Surface Imaging Information - *In situ* Surface Sensing and Nanoprofilometry for Focused Electron and Ion Beam Induced Processes Verification**, ***Andre Linden***, Raith America, Inc., *A. Rudzinski, M. Levermann, T. Michael,* Raith GmbH, *E. Maynicke*, RWTH Aachen

Nanopatterning processes and corresponding parameters are typically well understood for standard nanofabrication applications using resist based electron beam lithography (EBL) or FIB milling processes (e.g. for TEM lamella preparation).

Recently however, the bandwidth of nanofabrication applications for dedicated nanopatterning tools has significantly broadened and is no more limited to resist based EBL and mere, standard FIB milling tasks. Some latest generation multi-technique electron and ion beam nanolithography tools even facilitate additional *in situ* processes such as resistless focused electron or ion beam induced processes - e.g. material deposition or gas enhanced etching. The number of variable parameters for such complex processes involving e.g. new gas chemistry or ion species is nearly "infinite". Moreover, smart and flexible patterning strategies, e.g. by using loops in conjunction with various multi-directional patterning modes, have significant impact on the final nanostructure´s definition and performance, so that a straight *in situ* characterization of e.g. material deposition, milling or etching rates becomes crucial for most efficient understanding and subsequent optimization of such processes.

In contrast to elaborately using additional analytical equipment outside the vacuum and subsequently re-introducing the sample for further processing and optimization, we have implemented a distance sensitive nanomanipulator with nanoprofilometric capabilities into our professional multi-technique nanofabrication tools, which allows *in situ* characterization of nanostructures in 3D with ~10nm resolution by collecting topographic sample surface information.

First results of direct *in situ* growth rate determination of focused electron beam induced material deposition (FEBID) for process calibration as well as 3D surface topographic information of challenging milling applications will be presented.

8:40am **HI+AS+SS+NS-ThM3 Nanofabrication Using Gas-Assisted Focused Ion Beams**, ***Chad Rue***, FEI Company **INVITED**

A brief introduction to gas-assisted etching for Focused Ion Beams (FIBs) is given, including typical chemical precursors for various applications, and appropriate beam control parameters such as pixel overlap, dwell time, and refresh time. These factors are reasonably well-understood for pattern dimensions that are large compared to the size of the ion beam. However, for applications such as nanofabrication, which require high milling precision over small areas, the limiting size of the ion beam and its associated activated volume begin to influence the milling performance. The remainder of the discussion will focus on the relatively-unexplored regime in which the size of the pattern dimension is comparable to the size of the ion beam itself. The influence of various beam control parameters, particularly refresh time, becomes critically important to the milled profile of the desired structure. Redeposition effects, peripheral erosion, and mill rate trends are discussed. Operating tips and tricks are described, including the use of drift compensation strategies. The minimum physically achievable via size is examined. For a 10 pA Ga^+ beam at 30 keV, used to mill a via in a SiO_2 substrate with XeF_2-assist, the minimum achievable via size (FWHM) is found to be 50 ± 10 nm, and is relatively independent of depth or aspect ratio. Implications for nanofabrication are discussed and examples are shown.

9:20am **HI+AS+SS+NS-ThM5 The Psychology and Applications of a Bipolar Plasma Focused Ion Beam**, ***Rod Boswell***, ANU, Australia, *N. Smith, P. Tesch, N. Martin*, Oregon Physics

A new high brightness ion source has been developed using bi-polar power supplies that can be used with either positive or negative ions. This has involved a redesign of the plasma source and the acceleration optics to allow high currents to be focused with an energy of up to 30kV. We expect to make significant advances in Ultra High Resolution SIMS with a negative oxygen beam; a second application is the milling of structures in glass with a O- beam, such as a microfluidic set of channels. At the higher voltages mentioned above, it should be possible to cut cross sections of Through Silicon Vias in glass substrates. The challenges encountered in creating and extracting the negative ions will be discussed along with some performance and application data.

9:40am **HI+AS+SS+NS-ThM6 Advanced FIB Applications with New Ion Species and Large Area Capabilities**, ***Sven Bauerdick***, *L. Bruchhaus,* Raith GmbH, Germany, *J. Fridmann,* Raith America, Inc., *P. Mazarov, A. Nadzeyka, R. Jede*, Raith GmbH, Germany

Focused ion beam (FIB) systems are applied to a wide range of applications in R&D nanofabrication, both for creating functional devices as well as for preparing sample imaging and analysis. With different ion species on one hand and very sophisticated patterning approaches on the other hand it is possible to improve results and provide solutions for more advanced applications. Here we show and discuss the capabilities of Ga and new ion species like Au or Si with high resolution, long-term stability and easy handling, which is combined with an instrument design enabling large area or elongated patterns by write field stitching or truly continuous writing, respectively.

The type of ion defines the nature of the interaction mechanism with the sample and has significant consequences on the resulting nanostructures or samples. Therefore, we have extended the FIB technology towards the delivery of multiple ion species selectable into a nanometer-scale focused ion beam by employing a liquid metal alloy ion source (LMAIS). A mass separation filter is incorporated into the column to allow for fast and easy switching between different ions. The respective capabilities of mainly Ga, Au and Si have been investigated (resolution, milling rate, imaging, implantation) and according results and applications will be presented.

Moreover we investigated, optimized and tested milling approaches for pattern (write field) stitching and for truly continuous patterning based on precise stage movement while milling/ cutting with the ion beam. An improved beam pattern needs to mimic the looping strategy of conventional milling, so that grooves with defined depth, steep sidewalls and minimum re-deposition can be achieved. This combination of functionality enables applications like nanofabrication of micro-fluidic mixers, zone plates, large area gratings, or wafer-level nanopore devices as well as sample investigation e.g. imaging, X-sectioning and preparation in an automated way. Examples for new nanofabrication techniques like large area hard masking by implantation, both for reducing and increasing the rate in standard etching processes, or seamless direct milling of nano-fluidic channels over cm's will be discussed.

11:00am **HI+AS+SS+NS-ThM10 SIMS on the Helium Ion Microscope : a Powerful Tool for High-resolution High-sensitivity Nano-Analytics**, ***Tom Wirtz**, D. Dowsett,* Luxembourg Institute of Science and Technology (LIST), Luxembourg, *S. Sijbrandij*, Carl Zeiss Microscopy **INVITED**

While the ORION Helium Ion Microscope NanoFab has become an ideal high resolution imaging and nanofabrication tool, its analysis capability is currently limited. By contrast, Secondary Ion Mass Spectrometry (SIMS) is an extremely powerful technique for analysing surfaces owing in particular to its excellent sensitivity, high dynamic range, very high mass resolution and ability to differentiate between isotopes. The combination of He/Ne microscopy and SIMS would not just offer the prospect of obtaining SIMS information limited only by the size of the probe-sample interaction (~10 nm) but also of directly correlating such SIMS images with high resolution (0.5 nm) secondary electron images of the same zone taken at the same time. We have therefore investigated the feasibility of combining SIMS with Helium Ion Microscopy from a fundamental and instrumental point of view.

In order to reach good detection limits when probing very small voxels in imaging applications, the ionization probability of the sputtered atoms and molecules needs to be maximized. When using He^+ and Ne^+ bombardment, the intrinsic yields are low compared to the ones found in conventional SIMS. However, the yields may be drastically increased by using reactive gas flooding during analysis, namely O_2 flooding for positive secondary ions and Cs flooding for negative secondary ions. Our results show that both negative and positive ion yields obtained with He^+ and Ne^+ bombardment may be increased by up to 4 orders of magnitude when using such reactive gas flooding. This optimization of secondary ion yields leads to detection limits varying from 10^{-3} to 10^{-6} for a lateral resolution between 10 nm and 100 nm.

The prototype instrument we developed during this feasibility study contains extraction optics allowing the emitted secondary ions to be extracted with a maximized efficiency and without negatively impacting the focusing of the incoming He^+ or Ne^+ ion beam (broadening or distortion of the ion beam due to the electric fields). These extraction optics are coupled to a specially designed compact high-performance magnetic sector double focusing mass spectrometer that we developed for the purpose of HIM-SIMS. The specifications of this mass spectrometer include high mass resolution with optimized transmission ($M/\Delta M > 1000$ at 100% transmission or $M/\Delta M > 3000$ at 50% transmission), full mass range (H-U) and parallel detection of several masses.

The results are very encouraging and the prospects of performing SIMS on the Helium Ion Microscope are very interesting. In this paper we will present the main findings of our feasibility study, including fundamental, instrumental and application aspects.

11:40am **HI+AS+SS+NS-ThM12 Nanometer TOF-RBS and TOF-SIMS in a Helium/Neon Ion Microscope**, ***Nico Klingner**, R. Heller, G. Hlawacek, S. Facsko, J. von Borany*, Helmholtz-Zentrum Dresden - Rossendorf, Germany

Helium ion microscopes (HIM) have become powerful imaging devices within the last decade. Their excellent lateral resolution down to 0.3 nm and their high field of depth make them a unique tool in surface imaging [1]. So far the the analytical capabilities of a HIM are rather limited or need complex detection setups. In addition we will discuss major challenges and physical limitations of ion beam analysis in the HIM.

We will present a new and relatively easy to implement method for ion beam analysis in the HIM by means of time of flight spectrometry to obtain elemental information from the sample. We will demonstrate the flexibility and applicability of the method to image samples with target mass contrast, to analyze the target compositions, and to measure depth profiles of films with few tens of nm thickness.

Pulsing the primary helium or neon ion beam and measuring the time of flight of ejected particles allows to obtain the energy of the backscattered particles as well as the mass of the ionized, sputtered target atoms. This has been achieved by chopping the primary ion beam down to pulse widths of 18 ns by use of the built-in beam blanker and a customized plug-on beam blanking electronics. The secondary particles are detected by means of a multi channel plate mounted on a flange of the HIM.

We will show TOF-RBS and TOF-SIMS measurements for different materials, which can give complementary information. Lateral resolved TOF-SIMS allows to quickly obtain qualitative elemental mapping while the TOF-RBS gives the standard-free quantitative sample composition of regions of interest. We will also show, that with TOF-RBS depth profiling of nm-thick layers is possible.

[1] G. Hlawacek, V. Veligura, R. van Gastel, and B. Poelsema, J. Vac. Sci. Technol. B 32(2), 2014

12:00pm **HI+AS+SS+NS-ThM13 Improving Pattern Fidelity in Helium Ion Beam Lithography using Pixel Dose Optimization**, *N. Kalhor,* TU Delft, Netherlands, *W. Mulckhuyse,* TNO Technical Sciences, Netherlands, ***Paul Alkemade**,* TU Delft, Netherlands, *D. Maas*, TNO Technical Sciences, Netherlands

Scanning Helium ion beam lithography (SHIBL) with a sub-nanometer beam probe size at the sample surface is a promising technology for high-resolution lithography with high pattern density.[1] The advantages of SHIBL compared to e-beam lithography are higher sensitivity and a lower proximity effect. Remarkably, there are unique similarities in the activation response of resists to He-ions and extreme-ultraviolet (EUV) photons in EUV lithography (EUVL). Both primary beams produce low energy secondary electrons (SEs) and are not hindered by proximity effect. Recently Maas et al. experimentally demonstrated these similarities and suggested SHIBL as a promising method for pre-screening chemically amplified resists (CARs) prior to their final performance evaluation in an EUV scanner.[2]

However, unlike an EUV photon which only interacts with one resist molecule, an He-ion scatters inelastically in the resist and causes a chain of collisions with resist molecules, producing one or more SEs per collision. Also, a small dose-to-clear of 0.085 ions/nm^2 for SHIBL in a CAR was measured.[2] Hence, Maas et al. hinted at ion shot noise as a limiting factor in pattern fidelity in SHIBL.[2]

Here, we present a heuristic resist activation model for single-pixel dose SHIBL. The model employs a point-spread function (PSF) to account for all contributing factors in the resist activation. Ion shot noise impact is modeled with Poisson statistics. We show a good agreement between the model and our experimental single-pixel dose SHIBL results for line-and-space (LS) and contact hole patterns. Our model indicates pattern fidelity in sensitive CAR is not only limited by ion shot noise; instability of the He-ion source emission and post-exposure resist processing can also play important roles. Moreover, we introduce optimized-pixel-dose SHIBL to improve critical dimension uniformity (CDU), line width roughness (LWR), exposure latitude and throughput gain. In this approach, we calculate an optimum ion dose map for a given binary pattern such that the pattern's edges are exposed at the steepest part of the PSF to improve resist-pattern contrast and to minimize ion shot noise effect. Pixel dose optimization is advantageous to single-pixel exposure when the feature size is larger than the FWHM of the PSF. We discuss this by comparing our modeling results for single-pixel and optimized-pixel-dose SHIBL exposure modes for a desired LS pattern. We show that pixel-dose optimization could reduce LWR by ~45% (~1.3 nm) with a concurrent 20% dose reduction.

[1]V. Sidorkin et al., J. Vac. Sci. Technol. B **27**, L18 (2009)

[2]Maas et al., SPIE Proc. **9048**, 90482Z (2014)

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 nm^2 regions scanned on single-layer graphene films on Si/SiO_2 substrates at doses of 0.62 - 10 10^{16} 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 ionirradiated regions appeared as depressed about 1 nm in topography image at above conditions, while a darker image was acquired at 2.0 10^{16} 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 10^{16} cm^{-2} improved the spatial resolution to several tens of nanometers. However, doses of more than 1.0 10^{17} 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 Emmrich***, *E. Marschewski*, Bielefeld University, Germany, *A. Nadzeyka, F. Nouvertné,* Raith GmbH, Germany, *A. Gölzhäuser, 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 $YBa_2Cu_3O_{7-\delta}$ (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 10^{14} and 10^{18} He^+/cm^2. 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 systematical 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 PI95 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 $1x10^{18}He^+/cm^2$. 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 Shutthanandan***, 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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