## Advanced Ion Microscopy Focus Topic
## Room 203B - Session HI-WeA

### Novel Beam Induced Material Engineering & Nano-Patterning

**Moderators:** Armin Gölzhäuser, Bielefeld University, Germany, Olga Ovchinnikova, Oak Ridge National Laboratory

2:20pm **HI-WeA1 Delving into the Finer Details of Helium FIBID*, Frances Allen,*** University of California, Berkeley **INVITED**

Focused ion beam induced deposition (FIBID) of gaseous precursors enables localized template-free additive lithography at the nanoscale and is used in a range of applications, such as circuit edit in semiconductor engineering and the prototyping of nanodevices. To date, the majority of FIBID has used the gallium focused ion beam generated by the long-established liquid-metal ion source. However, with the development of the atomically sharp gas field-ionization source (GFIS) and subsequent emergence of the Helium Ion Microscope, FIBID using focused helium ion beams is increasingly of interest. The enhanced spatial resolution of helium FIBID over gallium FIBID and ability to deposit insulators free from gallium contamination are key areas of benefit.

The internal structure, composition, and overall shape of FIBID nanostructures and the influence of the deposition parameters thereon provide clues as to the growth mechanisms involved. Ultimately, the goal is to use this information to facilitate tunable FIBID in order to obtain nanostructures with a specific set of properties for a given application. I will present insights gleaned from scanning transmission electron microscopy (STEM) analysis of helium-FIBID nanostructures, where x-ray energy-dispersive spectrometry (XEDS) and new methods in "4DSTEM" diffraction are applied to obtain elemental compositions and grain orientation maps at the nanoscale with high sensitivity. The results are compared with those obtained for neon FIBID (the neon beam also generated by the GFIS source) and benchmarked against results from gallium FIBID. Several unique applications of helium FIBID drawing on the particular characteristics of helium-FIBID nanostructures will be discussed.

3:00pm **HI-WeA3 Anderson Localization of Graphene by Helium Ion Irradiation***, Y. Naitou,* ***Shinichi Ogawa,*** National Institute of Advanced Industrial Science and Technology (AIST), Japan **INVITED**

Graphene has been the subject of intensive research for its unique physical properties. Recently, tuning the electrical properties of graphene by irradiating it with an ion beam or exposing it to a reactive gas atmosphere has been of great interest[1][2]. The basic idea is to generate defects by using accelerated ion beam bombardment or reactive gas treatment and then to introduce localized states around the charge neutral point of graphene. Such localized states govern the transport properties of graphene, and highly defective graphene as a transition into a two-dimensional Anderson insulator is theoretically predicted[3].

Irradiation of a single-layer graphene (SLG) with accelerated helium ions ($He^+$) by helium ion microscopy (HIM) controllably generates defect distributions, which create a charge carrier scattering source within the SLG. We report direct experimental observation of metal-insulator transition in SLG on $SiO_2$/Si substrates induced by Anderson localization. This transition was investigated using scanning capacitance microscopy by monitoring the $He^+$ dose conditions on the SLG. The experimental data show that a defect density of more than ~1.2% induced Anderson localization. We also investigated the localization length by determining patterned placement of the defects and estimated the length to be several dozen nanometers—no fewer than 20 nm and no more than 50 nm. These findings provide valuable insight for direct-patterning and designing graphene-based nanostructures using HIM. Further detail will be presented[4][5].

References

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4:20pm **HI-WeA7 The Frontiers of Focused Ion Beam in Semiconductor Applications*, Shida Tan,*** Intel Corporation **INVITED**

The semiconductor performance scaling or "Moore's Law" has completely transformed the face of the planet and our daily life in the past half a century. This innovation trend continues through a combination of the transistor density scaling, heterogeneous integration, and architectural breakthroughs. These smaller critical device dimensions, thinner process layers, densely packed structures, complex device routing, and design architecture pose challenges to the focused ion beam (FIB) technology, which is used broadly in the entire product development cycle from the fabrication process to the final product debug and failure analysis. In this paper, we will talk about the unique advantages and applications of alternative ion beam in the areas of circuit edit, failure analysis, fault isolation, yield analysis, and mask repair. Trade-offs between various beam parameters to enable successful recipe implementation, challenges of the existing technologies, and the requirements for future instrumentation development will be discussed.

5:00pm **HI-WeA9 2D Materials Under Ion Irradiation: In-situ Experiments and the Role of the Substrate*, Gregor Hlawacek,*** *S. Kretschmer,* Helmholtz Zentrum Dresden-Rossendorf, Germany*; M. Maslov,* Moscow Institute of Physics and Technology*; S. Ghaderzadeh, M. Ghorbani-Asl, A.V. Krasheninnikov,* Helmholtz Zentrum Dresden-Rossendorf, Germany

Helium ion Microscopy (HIM) is frequently used for the fabrication of 2D nanostructures in graphene, $MoS_2$ and other materials. While some of the experiments are carried out with freestanding materials most of the work is done on supported material. While the defect production is understood for the former case, it is not fully understood in the latter setup. We used a combination of analytical potential molecular dynamics and Monte Carlo simulations to elucidate the role of the different damage channels, namely primary ions, backscattered atoms and sputtered substrate atoms.

Using this approach we looked at the defect production by helium and neon ions in $MoS_2$ and graphene supported by $SiO_2$ at typical energies used in HIM. We show that depending on ion species and energy defect production for supported 2D materials can be dominated by sputtered atoms from the support, rather than direct damage induced by the primary ion beam. We also evaluated the consequences of these additional damage mechanisms on the achievable lateral resolution for HIM based defect engineering and nano-fabrication in 2D materials. The obtained results agree well with experimental results obtained by in-situ and ex-situ characterization of defects in graphene and $MoS_2$.

5:20pm **HI-WeA10 Sample Heating Effects from Light Ions in Thin Films*, John A. Notte,*** *B.B. Lewis,* Carl Zeiss Microscopy, LLC

The term "FIB Renaissance" has been applied to the recent period of ion source development which has brought forth many new species suitable for focused ion beam (FIB) instruments. Several of the new species are relatively light ions, including hydrogen, helium, lithium, and neon, which are appreciably lighter than the prevailing gallium FIB – by a factor of 3 or more. At the conventional energies (5 to 30 keV) these ions species interact with the sample differently, and warrant a reconsideration of the established understanding which is largely founded on the traditional gallium FIB.

The most marked distinction of these light ions is the ratio of electronic stopping power compared to nuclear stopping power. For example, for a 30 keV helium ion, the nuclear stopping power can be a decade lower than its electronic stopping power. While for 30 keV gallium, the nuclear stopping power is a decade higher than the electronic stopping power. Consequential to this, near the surface the light ions remain relatively collimated because $M_{ion} >> M_{elec}$, making angular deflections necessarily small. As the light ions gradually penetrate deeper, they lose their energy, and the electronic stopping power is correspondingly reduced until the nuclear stopping power dominates. Here, large angular deflections become dominant, and the majority of the lattice damage takes place at these greater depths for light ions. For the special case of thin films, nuclear stopping might never become predominant for light ions.

The heat transfer mechanisms are even more drastically different when comparing light ions to heavier ions. First, by virtue of their large penetration depth, the light ions have a larger volume in which their energy is dissipated – reducing the corresponding temperature rise. But more significantly, the light ions lose most of their energy through excitations to the electrons. These excited electrons have characteristic mean free paths which can be relatively long, providing an effective pathway for energy transfer to a much larger volume. Whereas for nuclear stopping power, the ion's energy is transferred to the lattice much more locally. And since nuclear stopping is predominant for heavy ions, the energy is necessarily deposited locally, giving rise to appreciably higher temperature. Further, for the special case of thin films, the temperature rise from light ions is further reduced. Lastly, a special case of low beam currents is considered, where the time interval between ion arrivals may sometimes be longer than the time scale for thermal relaxation. This gives rise to non-overlapping temperature spikes which can be independent of probe current.

5:40pm **HI-WeA11 Helium Ion Direct Write Patterning of Superconducting Electronics**, ***Shane Cybart**, E.Y. Cho, H. Li,* UC Riverside; *Y. Naitou, S. Ogawa,* National Institute of Advanced Industrial Science and Technology (AIST), Japan

We report the fabrication of nanoscale Josephson junctions in 25 nm thick YBa2Cu3O7 thin films. Our approach utilizes a finely focused gas field ion source from a helium ion microscope to directly modify the material on the nanometer scale to convert irradiated regions of the film into insulators. In this manner, the film remains intact and no material is milled or removed. We will present results of how the critical dimension beam affects the electrical properties. Furthermore we reflect on the potential of this method for future device applications in superconducting computing.

We acknowledge T. Iijima and Y. Morita for the usage of the HIM at SCR station of AIST.

## Advanced Ion Microscopy Focus Topic
## Room 203B - Session HI+AS-ThM

### Advanced Ion Microscopy & Surface Analysis

**Moderators:** Gregor Hlawacek, Helmholtz Zentrum Dresden-Rossendorf, Germany, Shida Tan, Intel Corporation

8:00am **HI+AS-ThM1 Pushing the Limits: Secondary Ion Mass Spectrometry with Helium Ion Microscopy*, Alex Belianinov,*** Oak Ridge National Laboratory*; S. Kim,* Pusan National University, South Korea*; M. Lorenz,* University of Tennessee Knoxville*; A.V. Ievlev, A. Trofimov, O.S. Ovchinnikova,* Oak Ridge National Laboratory **INVITED**

Material functionality is defined by structure and chemistry often at micro- and nano-scale. The effects are coupled; however, few methods exist that can simultaneously map both. This presents a challenge for material scientists. Functional materials are continuously increasing in complexity, and the number of studies devoted to designing new materials often overwhelms characterization capacity. Recently, attention has been devoted to offer hardware and software solutions in chemical imaging, where a blend of *in-situ* and *ex-situ* techniques are used to capture and describe material behavior using combinatorial data. However, many of the emerging techniques need to be carefully validated and contrasted against existing approaches.

This talk will cover the performance of the recently developed combinatorial Helium Ion Microscopy (HIM) and Secondary Ion Mass Spectrometry (SIMS) tool on a wide variety of conductive and insulating samples. While the HIM imaging and milling performance to explore the physical structure has been repeatedly demonstrated, questions on the effect, quality, and resolution of a Neon beam SIMS remain. Ion yields, chemical resolution, and charge compensation results and strategies will be presented and discussed.

**Acknowledgement**

This research was conducted at the Center for Nanophase Materials Sciences, which is a DOE Office of Science User Facility.

8:40am **HI+AS-ThM3 When HIM meets SIMS*, Tom Wirtz,*** Luxembourg Institute of Science and Technology (LIST), Luxembourg*; O. De Castro, J. Lovric,* Luxembourg Institute of Science and Technology (LIST)*; J.-N. Audinot,* Luxembourg Institute of Science and Technology (LIST), Luxembourg

In 2015, we first presented a Secondary Ion Mass Spectrometry (SIMS) system which we specifically developed for the Zeiss ORION NanoFab Helium Ion Microscope (HIM) [1]. This SIMS system is based on (i) specifically designed secondary ion extraction optics coupled with post-acceleration transfer optics, providing maximized extraction efficiency while keeping a finely focussed primary ion beam for highest lateral resolution, (ii) a compact floating double focusing magnetic sector mass spectrometer allowing operation in the DC mode at full transmission (and hence avoiding duty cycles like in TOF systems that either lead to very long acquisition times or, for a same acquisition time, intrinsically limit the sensitivity) and (iii) a specific detection system allowing the detection of several masses in parallel. We have demonstrated that our instrument is capable of producing (i) mass spectra with high mass resolution, (ii) very local depth profiles and (iii) elemental SIMS maps with lateral resolutions down to 12 nm [1-5]. Furthermore, HIM-SIMS opens the way for in-situ correlative imaging combining high resolution SE images with elemental and isotopic ratio maps from SIMS [2,3,6]. This approach allows SE images of exactly the same zone analysed with SIMS to be acquired easily and rapidly, followed by a fusion between the SE and SIMS data sets [6]. Moreover, thanks to its depth profiling capability of the SIMS add-on, it is now possible to follow the chemical composition in real time during nano-patterning in the HIM for applications such as end-pointing.

Here, we will review the instrument performance and present a number of examples taken from various fields of applications, with a special emphasis on 3D reconstructions in materials science (battery materials, solar cells, micro-electronics) and on correlative HIM-SIMS bioimaging.

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9:00am **HI+AS-ThM4 Deciphering Chemical Nature of Ferroelastic Twin Domain in $MAPbI_3$ perovskite by Helium Ion Microscopy Secondary Ion Mass Spectrometry*, Yongtao Liu,*** University of Tennessee*; L. Collins,* Oak Ridge National Laboratory*; R. Proksch,* Asylum Research an Oxford Instruments Company*; S. Kim,* Oak Ridge National Laboratory*; B.R. Watson,* University of Tennessee*; B.L. Doughty,* Oak Ridge National Laboratory*; T.R. Calhoun, M. Ahmadi,* University of Tennessee*; A.V. Ievlev, S. Jesse, S. Retterer, A. Belianinov, K. Xiao, J. Huang, B.G. Sumpter, S.V. Kalinin,* Oak Ridge National Laboratory*; B.H. Hu,* University of Tennessee*; O.S. Ovchinnikova,* Center for Nanophase Materials Sciences, Oak Ridge National Laboratory

Hybrid organic-inorganic perovskites (HOIPs) have recently attracted attention due to its success in optoelectronics, largely due to power conversion efficiency, which has exceeded 20% in a short time. Recently, the appearance of twin domains in $MAPbI_3$ has been described ambiguously in a number of investigations. While all previous publications are limited in the descriptions of ferroelectric and/or ferroelastic nature, given (i) the correlation of defect chemistry and ferroelasticity, (ii) the coupling of ferroelectricity and ionic states, the chemistry of this twin domain can no longer be ignored. In earlier investigations, the twin domain size is revealed in the range of 100 nm- 400 nm, well in the detectability of helium ion microscopy secondary ion mass spectrometry (HIM-SIMS) (spatial resolution ~10 nm). Therefore, in this work, we correlate HIM-SIMS with multiple image techniques to unveil the chemical nature of the twin domain in $MAPbI_3$ perovskite.

Our scanning probe microscopy (SPM) studies indicate the variation of elasticity and energy dissipation between domains. Moreover, correlating SPM with scanning electron microscope (SEM), we observed smooth topography and twin domain contrast in SEM image, simultaneously, indicating the twinning contrast in SEM image is not due to morphology. These results allow us to suppose the chemical variation between twin domains, suggesting the need of clarifying the chemical difference between domains.

Using HIM-SIMS, which combines high-resolution imaging <0.25 nm of helium ion microscopy with the chemical sensitivity of secondary ion mass spectrometry (SIMS), we can detect ion distribution with a spatial resolution of 10 nm, allowing us to quantitatively explore the chemical composition of the twin domains (100 nm-400 nm). A HIM-SIMS using two gas field ionization sources ($He^+$ and $Ne^+$) was utilized for mass-selected chemical imaging of perovskite samples as well as identification of chemical species by spectrum collection in this study. In a positive mode measurement, $CH_3NH_3^+$ (m/z~32) chemical map shows that the $CH_3NH_3^+$concentration differs both in grains and twin domains, however, the $Pb^+$ (m/z~208) distributes uniformly. These results clarify that the chemical variation between domains originates from $CH_3NH_3^+$ segregation. For the most relevant for the optoelectronic applications of HOIPs, we have shown that this chemical variation affects HOIPs' interaction with light. Combining HIM-SIMS with multiple image techniques, this work offers insight into the fundamental behaviors of the twin domain in $MAPbI_3$, as well as a new line of investigative thought in these fascinating materials.

9:20am **HI+AS-ThM5 Helium and Neon Ion Microscopy for Microbiological Applications*, Ilari Maasilta,*** University of Jyvaskyla, Finland **INVITED**

Imaging of microbial interactions has until now been based on well-established electron microscopy methods. In this talk I review our recent drive to study microbiological samples using a helium ion microscopy (HIM). The main focus will be given on bacterial colonies and interactions between bacteria and their viruses, bacteriophages, which we imaged in situ on agar plates [1]. Other recent biological applications will also be briefly discussed. In biological imaging, HIM has advantages over traditional scanning electron microscopy with its sub-nanometer resolution, increased surface sensitivity, and the possibility to image nonconductive samples. Furthermore, by controlling the He beam dose or by using heavier Ne ions, the HIM instrument provides the possibility to mill out material in the samples, allowing for subsurface imaging and in situ sectioning.

Here, we present the first HIM-images of bacterial colonies and phage–bacterium interactions are presented at different stages of the infection as they occur on an agar culture. The feasibility of neon and helium milling is also demonstrated to reveal the subsurface structures of bacterial colonies on agar substrate, and in some cases also structure inside individual bacteria after cross-sectioning. The study concludes that HIM offers great opportunities to advance the studies of microbial imaging, in particular in the area of interaction of viruses with cells, or interaction of cells with biological surfaces.

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11:00am **HI+AS-ThM10 Characterization of Soot Particles by Helium Ion Microscopy**, ***André Beyer****, D. Emmrich, M. Salamanca, L. Ruwe, H. Vieker, K. Kohse-Höinghaus, A. Gölzhäuser,* Bielefeld University, Germany

Complementary techniques for the characterization of soot particles are needed to gain insight into their formation processes. In this contribution, we focus on Helium Ion Microscopy (HIM) which allows high contrast imaging of soot particles with sizes down to 2 nm. Soot formation was realized with well-defined model flames from different fuel compositions. The particles were sampled on silicon substrates at different positons within the flame which allows choosing the particles degree of maturity. Large numbers of particles were recorded with a single HIM image in a relatively short time. A number of such images were combined to obtain meaningful particle size distributions. In addition, the following geometric properties of soot particles were evaluated: sphericity, circularity, and fractal dimension. Comparison with other experimental techniques as well as theoretical model calculations demonstrate the strength of the HIM characterization method [1-3].

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11:20am **HI+AS-ThM11 Development of a Surface Science Spectra Submission Form for Low Energy Ion Scattering (LEIS)**, *M.R. Linford,* ***Tahereh Gholian Avval****,* Brigham Young University*; H.H. Brongersma, T. Grehl,* IONTOF GmbH, Germany

Historically, Surface Science Spectra (SSS) has been an important archive for X-ray photoelectron spectroscopy (XPS), Auger electron spectroscopy (AES), and time-of-flight secondary ion mass spectrometry (ToF-SIMS) data; this detailed, peer-reviewed database now consists of thousands of submissions and spectra that represent these techniques. Thus, SSS has been and continues to be a valuable resource to the surface community. Recently, SSS has begun to expand the surface/material techniques it covers. For example, it now accepts spectroscopic ellipsometry submissions on the optical properties of materials. It is anticipated that submissions in this area will slowly increase so that SSS will become a valuable source of information in this area as well.

In this presentation we discuss the development of an SSS submission form for low energy ion scattering (LEIS). Fields in the form that will be discussed include the abstract, introduction, data, and conclusions. In an SSS submission, the provenance of the sample is carefully documented. A detailed description is also required of the equipment used and of all of its relevant operating parameters – the nature of its beams, the beam energies, the analyzer geometry, etc. The original data collected by the submitter must be supplied, and representative examples of it must be plotted.

Finally, we will show sample submissions based on this new form that should have been submitted for publication by AVS 2018. These will include LEIS submissions of $CaF_2$, SrO, and $Al_2O_3$ reference materials

11:40am **HI+AS-ThM12 Time of Flight Backscatter and Secondary Ion Mass Spectrometry in the Helium Ion Microscope**, ***Nico Klingner****, R. Heller, G. Hlawacek, J. von Borany, S. Facsko,* Helmholtz Zentrum Dresden-Rossendorf, Germany

Existing Gas Field Ion Source (GFIS) based focused ion beam (FIB) tools suffer from the lack of a well integrated analytic method that can enrich the highly detailed morphological images with materials contrast. While Helium Ion Microscopy (HIM) technology is relatively young several efforts have been made to add such an analytic capability to the technique. So far, ionoluminescence, backscattering spectrometry (BS), and secondary ion mass spectrometry (SIMS) using a magnetic sector or time of flight (TOF) setups have been demonstrated.

After a brief introduction to HIM itself and a summary of the existing approaches I will focus on our own time of flight based analytic approaches. TOF-HIM is enabled by using a fast blanking electronics to chop the primary beam into pulses with a minimal length of only 20 ns. In combination with a multichannel-plate based stop detector this enables TOF backscatter spectrometry (TOF-BS) using He ions at an energy of only 30 keV. The achieved lateral resolution is 54 nm and represents a world record for spatially resolved backscattering spectrometry.

Finally a dedicated extraction optics for positive and negative secondary ions has been designed and tested. The setup can be operated in point and shoot mode to obtain high resolution SIMS data or in imaging mode to obtain lateral resolved maps of the sample surface composition. First experiments revealed a very high relative transmission of up to 76% which is crucial to collect enough signal from nanoparticles prior to their complete removal by ion sputtering. The mass resolution of 200 is sufficient for many life science applications that rely on the isotope identification of light elements (e.g.: C, N). The lateral resolution of 8 nm has been evaluated using the knife edge method and a 75%/25% criterion which represents a world record for spatially resolved secondary ion images.

12:00pm **HI+AS-ThM13 Helium and Neon Focused Ion Beam Hard Mask Lithography on Atomic Layer Deposition Films**, ***Matthew Hunt****,* California Institute of Technology*; J. Yang,* University of Texas at Austin*; S.A. Wood, O.J. Painter,* California Institute of Technology

A hard mask lithography technique has been developed wherein a helium or neon focused ion beam (FIB) is used to directly etch a pattern into a thin, atomic layer deposition (ALD) film before then transferring the pattern into the underlying material using a reactive ion etch (RIE). The technique takes advantage of small He-FIB and Ne-FIB probe sizes, capable of directly etching patterns with feature sizes on the order of 1s and 10s of nanometers, respectively, while sidestepping several negative consequences associated with direct etch, namely that low sputter rates prevent large-area patterning from being carried out efficiently, with straight sidewalls, and/or without inducing substrate swelling. An example of the technique is presented here in which (1) 4-10 nm of ALD aluminum-oxide is applied as the hard mask on a 60 nm thick film of aluminum, (2) a <10 pC/um Ne-FIB dose is used to pattern lines that barely etch through the hard mask, and (3) a $Cl_2/CH_4/H_2$ RIE is used to etch the underlying aluminum in 10s of seconds. Neon FIB writing time is reduced by a factor of 20 or more compared to directly etching through the full 60 nm aluminum film. Nanowires as thin as 25 nm are produced with straight sidewalls on 70 nm pitch. This example is being utilized to make superconducting quantum circuit components, e.g. 4 mm long nanocoil inductors that fit into a (20x20) $um^2$ area. The technique has potentially wide-ranging nanofabrication applications given its amenability to different ALD/substrate material sets and compatibility with both He- and Ne-FIB.

## Advanced Ion Microscopy Focus Topic
## Room 203B - Session HI-ThA

### Emerging Ion Sources, Optics, and Applications

**Moderators:** John A. Notte, Carl Zeiss Microscopy, LLC, Shinichi Ogawa, National Institute of Advanced Industrial Science and Technology (AIST)

2:20pm **HI-ThA1 Development of Gas Field Ionization Source using Gas with Low Ionization Energy that Enables Sample Processing and Observation***, **Shinichi Matsubara**, H. Shichi, T. Hashizume,* Hitachi, Japan **INVITED**

Practical use of a gas field ionization ion source (GFIS) has been tried for decades, but it was known difficult to realize. The GFIS has an extremely high brightness and a small source size, so that the convergence performance is excellent. As the atomic structure formation technology of the emitter tip has matured, the He - GFIS has been used in the real world scanning ion microscopes (SIMs). At first, their application was focused on the observation of the surface of samples because of its surface sensitivity, long focal depth, and a high resolution. Recently, however, Ne - GFIS has also been used for applications on fine direct fabrication which was difficult with a gallium liquid metal ion source.

We are developing GFISs which emit various kinds of ions and their own characteristics has been investigated. We are also developing ion-switching techniques which enable quick switching between fabrications and observations. With these techniques, we expect to create innovative applications difficult to realize with other technologies. In the previous studies we showed that an $H_3^+$ ion is superior for observation with a low damage. Its energy dispersion is comparable with a $He^+$ ion (0.5 eV) but the sputtering rate is expected to be smaller. We also showed that the $H_3^+$ and $Ne^+$ ions can be switched within 1 s by using a mixed gas of $H_2$ and Ne, and by changing the emitter voltage. With this technique, we can instantly switch between fabrication and observation. Regarding the Ar-GFIS which is promising for fine fabrication, we showed that the effective fabrication rate given by the product of the current and the sputtering rate is highest among the Ne, Ar and Kr. Furthermore, we have put into practical use of a photomask repairing technology using $N_2$ - GFIS.

3:00pm **HI-ThA3 Development of Scanning Helium Microscopy (SHeM)***, **Susanne Schulze**, D.J. Ward, M. Bergin, S. Lambrick, W. Allison, J. Ellis, A. Jardine,* University of Cambridge, UK

Some of the major insights in the development of modern materials have come from scanning probe, electron, and ion microscopy, with advances in resolution and sensitivity enabling new material science. While charged particle beam techniques are widely used they have the serious drawback of causing damage to sample surfaces, while scanning probe techniques are limited to relatively flat surfaces and suffer from limited scan speeds. Instead, we are pursuing a different approach, using neutral atom beams. Here we will report on recent advances and development of the scanning helium microscopy (SHeM) technique.

Since SHeM uses a neutral beam of helium atoms at very low energy (<100 meV), the technique is suitable for measuring a variety of samples including insulators, semiconductors, organic and biological species. It is particularly attractive as the approach does not require any complicated post processing techniques. We will report on recent studies on range of materials and potential new applications, including measurements performed in collaboration with colleagues at the University of Newcastle (Australia) [1,2]. A particular focus will be on describing the underlying mechanisms of contrast formation.

Many of the technological challenges associated with SHeM have now been addressed, including helium focusing, sample preparation and nanoscale manipulation, thus enabling preliminary instruments to be developed[1,2,3]. One of the remaining challenges is adequate detection of neutral atom beams, which is a particular problem due to helium's high ionization energy[3]. Applications that require time-sensitive measurements require a small ionization volume; however, when very high temporal resolution is not required, as with SHeM, very large ionizers with high detection efficiencies can be used. We will also report a recently developed detector, based on the approach recently applied to surface spin-echo experiments [4,5,6], and having the highest yet reported sensitivity for helium atoms.

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3:20pm **HI-ThA4 Fabrication of Trimer/Single Atom Tip for GFIS by Field Evaporation without Tip Heating***, **Kwang-Il Kim**,* University of Science and Technology, Republic of Korea*; Y.H. Kim, T. Ogawa,* Korea Research Institute of Standards and Science (KRISS), Republic of Korea*; S.J. Choi,* Kyungpook National University, Republic of Korea*; B. Cho, S.J. Ahn, I.-Y. Park,* Korea Research Institute of Standards and Science (KRISS), Republic of Korea

The application of the helium ion microscope (HIM) has expanded in various fields, such as nano-patterning, material science, and biology, due to its high spatial resolution for imaging and high-precision machining [1-3]. HIM realized sub-nm resolution with gas field ion sources (GFIS) which generate s ion beams from one or three topmost atoms of tips to obtain high beam current density. However, it is difficult to fabricate atomically sharp tips, such as trimer/single atom tip (TSAT), in an ultra-high vacuum (UHV) condition. TSAT can be typically fabricated by either a build-up method or field-assisted reactive gas etching method with oxygen and nitrogen [4-7]. However, these methods usually adopt resistive tip heating at about 1000 K as pre-cleaning of tip surface before the tip sharpening process. This heating leads to complex system because of a power supply circuit to provide or flow a current through a heating loop, where the tip was welded. In our study, we show that TSAT can be fabricated by field evaporation effect with an oxide layer which remains on the tip surface owing to the absence of tip heating.

As the result of this study, we could get a single crystalline field ion microscopy (FIM) image of W(111) with fabricating a TSAT by field evaporation phenomenon without tip surface cleaning by high temperature heating process. The oxide layer which remained after electrochemical etching process induces etch-like phenomenon in UHV condition without any additional gas injection. In order to analyze verify the proposed etching process, the analytical techniques of transmission electron microscope (TEM), energy filtered transmission electron microscope (EFTEM), and electron energy loss spectroscopy (EELS) were used. To compare the etching results whether the insulating layer present or not, we did additional experiment for tip heating. It was found that tungsten oxides contained in the insulating layer of the tip surface causes the etching. This method is much simpler than conventional methods because it uses only field evaporation phenomenon for fabricating TSAT. Therefore, we can simplify the equipment configuration since there is no need to heat the tip.

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4:40pm **HI-ThA8 Avoiding Amorphization Related Shape Changes of Nano-structures during Medium Fluence Ion Beam Irradiation of Semiconductor Materials***, **Xiaomo Xu**, G. Hlawacek, H.-J. Engelmann, K.-H. Heinig,* Helmholtz Zentrum Dresden-Rossendorf, Germany*; W. Möller,* Helmholtz-Zentrum Dresden-Rossendorf, Germany*; A. Gharbi,* CEA-LETI, France*; R. Tiron,* CEA-LETI, MINATEC, France*; L. Bischoff, T. Prüfer, R. Hübner, S. Facsko, J. von Borany,* Helmholtz Zentrum Dresden-Rossendorf, Germany

We present an approach to mitigate the ion beam induced damage inflicted on semiconductor nano-structures during ion beam irradiation. Nanopillars (with diameter a of 35 nm and height of 70 nm) have been irradiated with either a 50 keV $Si^+$ broad beam from an implanter or a 25 keV focused $Ne^+$ beam from a helium ion microscope (HIM) . Upon irradiation of the nanopillars at room temperature with a medium fluence (2e16 ions/$cm^2$), strong plastic deformation has been observed which hinders further device integration. This differs from predictions made by the simulations using TRI3DYN. However, irradiation at elevated temperatures with the same fluence would preserve the shape of the nanopillars.

It is well known that a critical temperature exists for silicon above which it will recrystallize during ion beam irradiation. This prevent s the amorphization of the target material independent of the applied fluence. At high enough temperatures and not for too high flux this prevents the ion beam hammering and viscous flow of the nano-structures. These two effects are responsible for the shape change observed at low temperature. This has been observed previously mainly for swift heavy ions and energies higher than 100 keV. We used HIM and transmission electron microscopy to follow the morphological evolution of the pillars and their crystallinity. While irradiation at room temperature results in amorphization and the related destruction of the nanopillars, irradiation above 650 K preserves the crystalline nature of the pillars and prevents viscous flow. This effect has been observed previously mainly for swift heavy ions and energies higher than 100 keV. Such high-temperature irradiation, when carried out on a nanopillar with Si/SiO2/Si layer stack, would induce ion beam mixing without suffering from the plastic deformation of the nanostructure. Due to a limited mixing volume, single Si-NCs would form in a subsequent rapid thermal annealing process via Oswald ripening and serve as a basic structure of a gate-all-around single electron transistor device.

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## Advanced Ion Microscopy Focus Topic
## Room Hall B - Session HI-ThP

## Advanced Ion Microscopy Poster Session

**HI-ThP1 He+ and Ne+ Ion Beam Resolution Dependency on Beam Energy***,*
***Waqas Ali****,* Intel Corporation, USA*; S. Tan,* Intel Corporation*; R.M. Hallstein, R.H. Livengood,* Intel Corporation, USA

For several decades, Gallium (Ga+) remained an ion species of choice for circuit edit (CE) applications due to its excellent micro and nanomachining capabilities. But due to continuous device scaling, now it is becoming highly challenging to fulfill all the needs of CE with Ga+ based focused ion beam (FIB) tools. Recently Neon gas field ionization source (GFIS) has emerged as one of the most viable solutions to supplement CE requirements where a Ga+ FIB falls behind [1]. A lot of effort has gone into the beam characterization of the Neon (Ne+) and Helium (He+) beams of Orion NanoFab that is the first GFIS based commercial tool. In this paper, we present our results on resolution characterization of He+ and Ne+ beams as a function of beam energy.

He+ beam resolution characterization was done at 10, 20 and 30 kV beam energies whereas Ne+ beam resolution was characterized at 10 and 25 kV. The test was conducted on CVD graphene on TEM grid and ImageJ was used for image analysis. The lateral resolution for Helium was 0.54 ± 0.07 nm at 30 kV beam energy whereas for Neon the resolution was 2.45 ± 0.46 nm at 25 kV beam energy both with 100 fA beam currents. The unparalleled resolution specs. of Ne+ and He+ ion beams have made them attractive not only for CE but for many other applications like high resolution imaging for fault isolation, failure analysis, EUV mask repair, lithography, graphene pattering, plasmonics and biological imaging etc. [2].

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**HI-ThP2** ***Focused Cs Ion Beam-Induced Deposition and Gas Assisted Etch Characterization Results for 10nm Circuit Edit Applications****,* ***Roy Hallstein****,*
*R.H. Livengood, M.P. Ly,* Intel Corporation, USA*; Y. Greenzweig, Y. Drezner,* Intel Corporation, Israel*; B.J. Knuffman, A.V. Steele, A.B.J. Knuffman,* zeroK NanoTech

Focused Ion Beam Gas Assisted Etch (GAE) and Ion Beam Induced Deposition (IBID) are used extensively in Circuit Edit nanomachining. Historically the Gallium Focused Ion Beam (FIB) has been the primary ion source technology for Circuit Edit applications. [1, 2] More recently, the neon and nitrogen ($N_2$) gas field ion sources (GFIS) have also been introduced to enable very small, high precision nanomachining for circuit rewiring and mask defect repairs respectively. [3, 4, 5] Other emerging ion source technologies are the so-called 'cold ion' sources, which ionize atoms that have been laser-cooled to micro-kelvin temperatures. These sources have been shown to have high brightness and low energy spread, enabling small focal spot sizes. [6] Two such emerging 'cold' sources that produce cesium ion beams are under development by zeroK NanoTech Corporation and Tescan Orsay Holding. [7, 8]

As part of the due diligence to identify breakthrough ion beam technologies to keep pace with nanomachining applications scaling requirements, we have completed preliminary analysis of the attributes of cesium for Circuit Edit applications. In this paper, Proof of Concept 10nm Circuit Edit results using the zeroK Nanotech Cesium ion beam-based GAE and IBID will be presented. Preliminary results include GAE chemical etching of semiconductor materials and IBID results for dielectric and metal depositions. Finally, preliminary electrical test results of proof of concept Circuits Edits on 10nm process node will be presented.

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