Advanced Ion Microscopy and Ion Beam Nano-engineering Focus Topic
Room B231-232 - Session HI+NS-ThA
Emerging Ion Sources, Optics, and Applications & Flash Session

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

2:20pm **HI+NS-ThA-1 Cold Atom Ion Sources**, ***Jabez McClelland***, *J Gardner, W McGehee,* National Institute of Standards and Technology (NIST)*; A Schwarzkopf, B Knuffman, A Steele,* zeroK NanoTech Corp. **INVITED**

Ionization of laser-cooled atoms has emerged as a new approach to creating high brightness ion sources for applications such as focused ion beam (FIB) microscopy, milling, and secondary-ion mass spectrometry (SIMS). Conventional sources, such as the Ga liquid metal ion source (LMIS) or the gas field ionization source (GFIS), attain brightness by emitting from a very sharp tip. In contrast, cold atom sources attain high brightness through reducing the transverse velocity spread of the ions. With the ultracold, microkelvin-range temperatures achievable with laser cooling, the corresponding velocity spread can lead to a brightness significantly higher than typical LMIS values. Moreover, the phase-space shape of the emittance of the source – narrow in velocity, wide in space – brings new opportunities for ion optical design. For example, high currents can be obtained without the high current density present in sharp tip sources. This can result in reduced Coulomb effects, such as increased emittance and broadened energy spread (Boersch effect). Other advantages of this type of source include insensitivity to contamination, access to new ionic species, inherent isotopic purity, and fine control over emission, down to the single ion level. To date, sources have been demonstrated with Cr,[1] Li,[2] Rb,[3] and Cs[4,5] ions, realizing novel species and nanometer-scale spot sizes. In this talk I will review progress in the field and discuss recent developments in Li ion sources and applications.

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3:00pm **HI+NS-ThA-3 Silicon Lithiation by Direct-writing with a Focused Li^+-ion Beam**, *W McGehee,* ***Evgheni Strelcov***, *V Oleshko, C Soles, N Zhitenev, J McClelland,* National Institute of Standards and Technology (NIST)

Improving the performance of Li-ion batteries requires understanding and controlling nanoscale ion transport at the level of interfaces, grain boundaries and defects. While in the last decades a range of electron and scanning probe microscopy techniques have been developed for probing local transport, no reliable method exists for quantitative and controllable nanoscale lithiation. Moreover, wet-cell electrochemical lithiation is significantly complicated by electrolyte decomposition, formation of solid-electrolyte interfacial (SEI) layer and parasitic reactions running in parallel to lithium insertion.

Building on our previous work,[1] here we introduce a new method of direct-write quantitative lithiation of battery-relevant materials in vacuo, in the absence of SEI or liquid electrolyte. To benchmark the technique, we use a focused, several keV Li^+-ion beam to inject lithium into 35-nm thick crystalline Si membranes with a sub-micron lateral precision. The lithiated regions, undergoing morphological, structural, chemical and functional transformations, were characterized with a combination of electron and scanning probe microscopy techniques. We observed saturation of interstitial lithium in the silicon membrane at ≈ 10 % dopant number density and spill-over of excess lithium onto the membrane's surface. The implanted Li^+ remains electrochemically active, and the spill-over effect can possibly be avoided by cooling the sample. The presented method is especially useful for probing non-equilibrium and low-concentration phases of lithiated materials that form because of incomplete lithium extraction or during initial states of pristine anode lithiation. Focused ion beam lithiation will enable controlled studies and improved understanding of Li^+ ion interaction with local defect structures and interfaces in electrode and solid-electrolyte materials.

E.S. acknowledges support under the Cooperative Research Agreement between the University of Maryland and the National Institute of Standards and Technology Center for Nanoscale Science and Technology, Award 70NANB14H209, through the University of Maryland.

W.R.M. and E.S. contributed equally.

1. Takeuchi, S.; McGehee, W. R.; Schaefer, J. L.; Wilson, T. M.; Twedt, K. A.; Chang, E. H.; Soles, C. L.; Oleshko, V. P.; McClelland, J. J. *Journal of The Electrochemical Society* **2016,** 163, (6), A1010-A1012.

3:20pm **HI+NS-ThA-4 A New FIB for Deterministic Single Ion Implantation**, ***Nathan Cassidy***, UK National Ion Beam Centre, University of Surrey, UK*; D Cox,* Advanced Technology Institute, University of Surrey, UK*; R Webb,* UK National Ion Beam Centre, University of Surrey, UK*; B Murdin,* Advanced Technology Institute, University of Surrey, UK*; P Blenkinsopp, I Brown,* Ionoptika Ltd., UK*; R Curry,* The Photon Science Institute, University of Manchester, UK

Single isolated dopant atoms implanted into solid state devices have been shown to be a viable architecture for quantum technologies. Ion implantation provides many advantages as a manufacturing method for such devices, such as speed and scalability, however controlling the number of implanted ions with single-ion precision poses a significant challenge. In this paper we will present a new instrument designed for the deterministic implantation of single ions with high precision.

The SIMPLE (Single Ion Multi-species Positioning at Low Energy) tool, is a new focused ion beam tool in operation designed for the manufacture of quantum technologies. The tool has a 25kV LMIG set up for femtoAmp sample currents, with ultra-fast beam blanking, neutral blocking and a highly efficient secondary electron detection system. Deterministic ion implantation is achieved through extraction of single ions through fast beam blanking with low currents, ion implant detection through collection of secondary electron (SE) signal from the target and high spatial precision in ion placement.

To date we have demonstrated > 85% probability of implanting a single Bi+ ion into silicon without error, with a 20nm beam determining dopant placement precision. This surface secondary electron detection efficiency has been validated through simultaneous measurements of a transmitted electron signal, achieved by implanting through thin lamellae. The ion placement precision has been determined through imaging of ion induced damage on highly oriented pyrolytic graphite (HOPG) surfaces. Much work has taken place maximizing the detection efficiency for secondary electrons and investigating the factors which affect the SE yield.

Currently the system is running with Bi source, and there are In sources available. Alongside the development of the instrument there is also research into developing a series of liquid-metal ion sources for elements with optical and quantum applications including P, Te, Se and Cd. A second SIMPLE tool has also been installed at the UK National Ion Beam Centre, which operates with a 20kV duoplasmatron arc source, capable of 50nm spot sizes. SIMPLE #2 will initially operate with nitrogen source for the fabrication of NV centres in diamond.

4:00pm **HI+NS-ThA-6 Technology and Applications of a Plasma Ion Source with User-selectable Ion Species**, ***Gregory Schwind***, *S Kellogg, J Stiller, M Doud, C Rue, B Van Leer,* Thermo Fisher Scientific **INVITED**

The focused ion beam (FIB) has become an indispensable tool for micro- and nano-machining applications. Due to its high brightness and ease of use, the gallium liquid metal ion source (LMIS) has been the source of choice over much of the nearly four decades of FIB history. At the beginning of this decade, a new generation FIB system based on the inductively coupled plasma (ICP) ion source was brought to market, offering beam current and throughput 20 times greater than LMIS-based systems. A next generation plasma source has been developed [1], offering the option to change the ion beam species by switching the feed gas supplied to the plasma source. The ability to dynamically change ion species—for example from a noble gas such as argon to an electronegative species such as oxygen—creates new design challenges for the source, the FIB optical subsystem, and the platform as a whole. Both empirical measurements and numerical simulations were used to better understand the species-specific performance of the source design. Results show that the emission properties depend on both the ion species and the plasma density, which lead to orienting the system design around specific modes of operation optimally suited to each species, FIB current and landing energy [2].

Several new and exciting application areas are enabled by the ability to switch FIB ion species dynamically. Ion-surface interactions such as sputtering, implantation, and the creation of an amorphous damage layer depend on the ion's momentum [3], which in turn depends on ion mass. Furthermore, chemical reactivity between the incoming ion and the target surface seems to play a role in the surface modification process in some instances. Several FIB application examples illustrating these interdependencies will be shown.

[1] Sergey Gorelick and Alex De Marco, "Fabrication of glass microlenses using focused Xe beam," Opt. Express 26, 13647-13655 (2018)

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4:40pm **HI+NS-ThA-8 Neutral Helium Microscopy***, **Bodil Holst**,* University of Bergen, Norway

Neutral helium microscopy is a new imaging technique currently under development. In a neutral helium microscope a beam of neutral helium atoms is created through supersonic expansion from a nozzle and focussed onto the surface to create a scanning instrument. The resolution is determined by the beam spotsize on the surface. The neutral helium microscope has several advantages: the very low energy of the beam (less than 0.02 eV compared to several keV for helium ion or electron microscope) , charge neutrality, and inertness of the helium atoms, a potential large depth of field, and the fact that at thermal energies the helium atoms do not penetrate into any solid material. This opens the possibility, among others, for the creation of an instrument that can measure surface topology on the nanoscale, even on surfaces with high aspect ratios. The helium microscope currently exist in two configurations: The pinhole microscope and the zone plate microscope, both are covered in this paper. We begin with a series of images which demonstrate and explores the unique contrast mechanisms of the new instrument. This is followed by a general discussion of helium microscope designs and resolution.

5:00pm **HI+NS-ThA-9 GaBiLi Liquid Metal Alloy Ion Sources for Advanced Nanofabrication***, P Mazarov,* RAITH GmbH, Germany*; T Richter, L Bruchhaus, W Pilz, R Jede,* Raith GmbH, Germany*; **Yang Yu**, R Schmid, J Sanabia,* Raith America, Inc.*; L Bischoff,* Helmholtz Zentrum Dresden-Rossendorf, Germany*; G Hlawacek,* Helmholtz-Zentrum Dresden Rossendorf, Germany

Nanofabrication requirements for FIB technologies are specifically demanding in terms of patterning resolution, stability and the support of new processing techniques. Additionally, the type of ion defines the nature of the interaction mechanism with the sample and thus has significant consequences on the resulting nanostructures [1]. Therefore, we have extended the technology towards the stable delivery of multiple ion species selectable into a nanometer scale focused ion beam by employing a liquid metal alloy ion source (LMAIS) [2]. This LMAIS provides single and multiple charged mono- as well as polyatomic ion species of different masses, resulting in significantly different interaction mechanisms. Nearly half of the elements of the periodic table are thus made available in the FIB technology as a result of continuous research in this area [3]. This range of ion species with different mass or charge can be beneficial for various nanofabrication applications. Recent developments could make these sources to an alternative technology feasible for nanopatterning challenges. In this contribution, the operation principle, the preparation and testing process as well as prospective domains for modern FIB applications will be presented. As an example we will introduce the GaBiLi LMAIS [4]. It enables high resolution imaging with light Li ions and sample modification with Ga or heavy polyatomic Bi clusters, all coming from one ion source. For sub-10 nm focused ion beam nanofabrication and microscopy, the GaBiLi-FIB or the AuSiGe-FIB could benefit of providing additional ion species in a mass separated FIB without changing the ion source.

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5:20pm **HI+NS-ThA-10 Focused Ion Beams in Biology: How the Helium Ion Microscope and FIB/SEMs Help Reveal Nature's Tiniest Structures***, **Annalena Wolff**,* Central Analytical Research Facility, Institute for Future Environments, Queensland University of Technology (QUT), Brisbane QLD 4000, Australia*; N Klingner,* Helmholtz Zentrum Dresden-Rossendorf, Germany*; W Thompson,* HeelionicsLLC*; Y Zhou,* Queensland University of Technology (QUT), Australia*; J Lin,* Affiliated Stomatological Hospital of Xiamen Medical College, China*; Y Peng,* CSIRO Manufacturing, Australia*; J Ramshaw,* St. Vincent's Hospital, University of Melbourne, Australia*; Y Xiao,* The Australia-China Centre for Tissue Engineering and Regenerative Medicine (ACCTERM), Queensland University of Technology, Australia

Focused Ion Beam (FIB) devices such as the Helium Ion Microscope (HIM) as well as FIB/SEMs are increasingly popular within the biological sciences in recent years. High resolution imaging of uncoated non-conductive samples with the HIM helps reveal nature's tiniest structures while the FIB/SEM allows to prepare TEM lamellae, 3D reconstruct the sample or reveal sub surface structures with nanometre precision.

This presentation shows how the HIM as well as FIB/SEMs can be used in biological sciences to reveal nature's tiniest structures. The presented work then focuses on the underlying ion-solid interactions and the effect of ion beam parameters on heating induced by ion beams. The work presented here deals with gallium ion solid interactions, however the broader results are applicable to any type of FIB including the helium ion microscope (HIM) and plasma FIBs. The interactions of gallium ions in skin were simulated using Monte Carlo methods, finite element simulations and numerical modelling for different beam parameters. The program SRIM [4] was used to obtain theoretical results which permit estimation of the ion beam induced temperature increases, using the physical principles of Fourier's law of conductive heat transfer.

The technique was tested on collagen, a soft biological material which is commonly used in biomedical applications. Collagen was chosen as a suitable test sample as it loses its fibrillary structure when denaturated by heat, permitting damage to easily be recognized. Cross-sections and TEM lamellas were prepared from non-embedded collagen with conventional FIB processing parameters as well as heat reducing FIB parameters.

The results also show that heat damage can be prevented by reducing the local dose rate and area underneath the ion beam. Using lower acceleration voltages allows the operator to select higher local dose rates (ion beam currents) and minimized processing times. A TEM comparison of a microtome prepared lamella and a FIB prepared lamella (using different heat reducing parameters) shows that the fibrillar structures can be maintained, and heat damage avoided. The approach described here can be used to determine suitable parameters for other soft materials.

The authors acknowledge scientific and technical assistance of Peter Hines, Jamie Riches, Rachel Hancock, and Ning Liu and the facilities at the Australian Microscopy & Microanalysis Research Facility (AMMRF) at the Central Analytical Research Facility (CARF), Queensland University of Technology, Brisbane, Australia.

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