Advanced Ion Microscopy Focus Topic
Room: 7 & 8 - Session HI+BI+NS+TR-ThM

Advanced Ion Microscopy Applications
Moderators: Armin Golzhauser, Bielefeld University, Germany, Olga Ovchinikova, Oak Ridge National Laboratory

8:00am **HI+BI+NS+TR-ThM1 Scanning Helium Atom Microscopy: Imaging with a Deft Touch**, ***Paul Dastoor***, University of Newcastle, Australia **INVITED**

Delicate structures (such as biological samples, organic films for polymer electronics and adsorbate layers) suffer degradation under the energetic probes of traditional microscopies. Furthermore, the charged nature of these probes presents difficulties when imaging with electric or magnetic fields, or for insulating materials where the addition of a conductive coating is not desirable. Scanning helium microscopy is able to image such structures completely non-destructively by taking advantage of a neutral helium beam as a chemically, electrically, and magnetically inert probe of the sample surface. Here, we present scanning helium micrographs demonstrating image contrast arising from a range of mechanisms including, for the first time, chemical contrast observed from a series of metal-semiconductor interfaces [1]. The ability of neutral helium microscopy to distinguish between materials without the risk of damage makes it ideal for investigating a wide range of systems.

1. M. Barr, A. Fahy, J. Martens, A.P. Jardine, D.J. Ward, J. Ellis, W. Allison & P.C. Dastoor, “Unlocking new contrast in a scanning helium microscope”, *Nature Communications*, **7**, 10189, (2016).

8:40am **HI+BI+NS+TR-ThM3 Biofilm Structure of Geobacter Sulfurreducens by Helium Ion Microscopy**, ***Alex Belianinov***, Oak Ridge National Laboratory, *M. Halsted, M.J. Burch,* Oak Ridge National Laboratory, *S. Kim, S. Retterer*, Oak Ridge National Laboratory

Microbial communities form biofilms on material surfaces in a multitude of ecosystems, from the root hairs of a plant to the human gut. The hallmarks of an established biofilm include (1) the attachment of microbial cells to a surface, (2) production of extracellular polymeric substance, (EPS) (3) a complex structure or “architecture,” and (4) the ability to exchange genetic information between cells. [1] Geobacter sulfurreducens forms unique, electrically conductive biofilms, a property that can be exploited in production and design of microbial fuel cells. In this work, examine biofilm formation, and biofilm properties of Geobacter sulfurreducens using a Scanning Electron Microscope (SEM) as well as a Helium Ion Microscope (HIM).

SEM is a high-resolution imaging technique used for characterization of a broad variety of materials. However, in order to image highly insulating, soft biological materials, the samples must be coated for charge compensation. These (typically) metallic coatings create a homogenous surface and may cloak true biological behavior and material contrast in the micrograph. In the case of Geobacter sulfurreducens, metal coating precludes detailed investigation of microbial attachment, presence of EPS, and fine surface details that may elucidate the mechanisms behind architecture formation and genetic material exchange.

Recently introduced HIM, offers more flexibility in investigating biological samples, as highly insulating sample can be imaged sui generis, without the use of a conductive coating. [2] This opens new pathways to capturing high resolution spatial details of biofilm formation and biofilm properties. Furthermore, high-resolution HIM imaging reveals true surface details of Geobacter sulfurreducens, such as flagella or pilin typically inaccessible by SEM. Finally, the effects of different sample preparation strategies for SEM and HIM will be illustrated and discussed.

References:

[1] 1. Donlan, R. M. “Biofilms: Microbial Life on Surfaces.” Emerging Infectious Diseases, 8(9), 881–890, 2002

[2] Joens, M. S., Huynh, C., Kasuboski, J. M., Ferranti et. al.. “Helium Ion Microscopy (HIM) for the imaging of biological samples at sub-nanometer resolution.” Scientific Reports, 3(3514), 2013

Acknowledgements:

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9:00am **HI+BI+NS+TR-ThM4 Channeling via Transmission He Ion Microscopy**, ***Christoph Herrmann***, Simon Fraser University, Canada, *S.A. Scott, M. Lagally,* University of Wisconsin-Madison, *K. Kavanagh*, Simon Fraser University, Canada

The spatial coherence of focussed helium (He) ion beams is significant. The He ion source is atomic size (W filament tip) and the resolution from scanning probe, ion-induced secondary electron images is sub 1 nm. Scanning transmission images with atomic resolution are theoretically predicted. We have been experimenting with a digital camera located underneath the sample stage and tilt cradle of our instrument (Zeiss Nanofab). The camera consists of an array of Si p-i-n diodes (55 µm square pixels) that allow direct detection of single He ions and atoms (20 keV - 40 keV). We have previously reported that the beam intensity profiles are uniformly distributed, as expected from the small de Broglie wavelength (80 fm), with a half angle convergence of 2 mrad.[1] At beam currents in the pA range the detector count rate was consistent with one count per He ion or atom. In this talk, we will present results that indicate planar channeling in single crystalline Si (100) membranes (25 nm - 75 nm thick). The transmission intensity as a function of position depends on the beam incidence angle, and beam energy, with random incidence profiles consistent with monte carlo scattering and range calculations (SRIM). The peak in transmission as a function of incidence angle has a half angle width of 1° at 25 kV. These results will be compared with theoretical calculations based on impact factors at low energies. Channeling experiments with other thin crystalline materials including graphite and MgO will be discussed. **Acknowledgements**: We thank Norcada Inc. (Edmonton) for supplying Si (100) 50 nm thick membranes; NSERC, CFI/BCKDF, 4DLABs for funding. [1] K.L. Kavanagh and C. Herrmann, Direct He Detection for Transmission Helium Ion Microscopy, Microsc. Microanal. submitted 2017.

9:20am **HI+BI+NS+TR-ThM5 Rapid Imaging of Nano-Porous Catalyst Particles Via Helium Ion Microscopy**, *M.J. Burch, A.V. Ievlev,* ***Holland Hysmith***, Oak Ridge National Laboratory, *K. Mahady, P.D. Rack,* University of Tennessee, *L. Luo*, ExxonMobil Chemical Company, *A. Belianinov,* Oak Ridge National Laboratory, *S. Yakovlev,* ExxonMobil Chemical Company, *O.S. Ovchinikova*, Oak Ridge National Laboratory

Porous materials are some of the most important modern day material systems, as the pore structure defines many materials applications and functionality. The pore structure of catalyst precursor particles, in particular, is of great importance to the catalyst community, as this pore structure dictates the efficiency and efficacy of grown polymers. However, despite the importance of these materials systems, there are few techniques to analyze pore size and structure. The most common technique is gas absorption, where the amount of gas absorbed and desorbed from a known amount of material is tracked and the average pore volume and size can be extracted. However, the technique is heavily dependent on sample quality and which fitting model is used to calculate volume and size. In addition, the technique is quite slow, where generally at most a single sample can be analyzed a day.

In this work, we demonstrate a novel technique to directly image and quantify pore size in nano-porous catalyst precursor particles via helium ion microscopy. We demonstrate the technique by directly imaging the surface pore structure of SiO_2 precursor catalyst particles with helium ion microscopy. Using modern day data analytics, we created an automated routine to extract pore size and distributions. We show that our HIM based technique shows comparable data to the industry standard gas absorption technique, within a 5 percent difference between the techniques of a known porous samples.

Further, to determine the effect of the helium beam on the surface of the SiO_2 particles, we simulate the beam interaction between porous SiO_2 particles and the helium beam. At low ion doses the surface modification by the ion beam is quite negligible, where at higher ion doses, significant surface modification is observed.

In conclusion, we’ve demonstrated a novel technique to directly visualize and quantify nano-pore size and structure in SiO_2 that yields complimentary data to gas absorption.

Acknowledgements

This work was conducted at the Center for Nanophase Materials Sciences, which is a Department of Energy (DOE) Office of Science User Facility. The users acknowledge the ExxonMobil Chemical Company for funding.

9:40am **HI+BI+NS+TR-ThM6 Ion Beam Induced Current Measurements of Solar Cells with Helium Ion Microscopy**, *A. Belianinov, S. Kim,* ***Ryan Cannon****, M.J. Burch, S. Jesse, O.S. Ovchinnikova*, Oak Ridge National Laboratory

The scanning electron microscope (SEM) is a versatile high-resolution microscopy tool, and perhaps the most widely used imaging platform across many engineering and scientific fields [1]. Within the last decade, another microscopy technique based on a gaseous field ionization source, utilizing Helium and Neon ions has been introduced [2]. While the popularity of the SEM is hardly challenged by the Helium Ion Microscopy (HIM), there are instances when imaging with ions offers significant advantage as opposed to imaging with electrons. In principle, both HIM and the SEM share many similarities, for example, a HIM operating at 40 keV will generate ions with velocity comparable to SEM operating at 5 keV. However, due to much higher stopping power of ions, as compared to electrons, ion based secondary electron (iSE) will be higher. Also, as a result, there is little ion backscattering, and consequently, the concentration of the ion-generated iSE2 (additional secondary electron generated by SE interaction within the material) is usually insignificant.

In this work, we exploit small interaction volumes in the HIM, and take advantage of the lower iSE2 yield, and positively charged helium ions to map ion beam induced current (IBIC) in solar cell materials. Similar studies, using electrons, have visualized induced current profiles at grain profiles in polycrystalline solar cells, and in silicon [3, 4]. Furthermore, broad ion sources have been utilized in conjunction with scanning probe systems in the past to map out current changes in FinFETs [5]. We are interested in utilizing the HIM to map current at the nanoscale near p-n junctions in CdTe to elucidate differences in contrast captured by the ion beam induced current, as opposed to the electron beam induced current. These findings will illustrate the peculiarities of ionic transport in these solar cell materials, and will evaluate the HIM technology as a potential quality control tool.

References:

[1] David C Joy, Helium Ion Microscopy: Principles and Applications, First ed. Springer, New York USA, Heidelberg Germany, Dordrecht Netherlands, London United Kingdom, 2013.

[2] Gölzhäuser, A. and Hlawacek, G., Helium Ion Microscopy. Springer International Publishing. 2016

[3] Donolato, C., Journal of Applied Physics, 54 (3), 1314-1322, 1983

[4] Chen, J., et. al., Journal of Applied Physics, 96(10), 5490-5495, 2004

[5] Manfredotti, C., et.al., Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment, 380(1-2), 136-140, 1996

11:00am **HI+BI+NS+TR-ThM10 Writing Magnetic Domains with a Helium Ion Microscope**, ***Daniel Emmrich***, Bielefeld University, Germany, *A. Gaul, D. Holzinger, A. Ehresmann,* University of Kassel, Germany, *F. Karimian, M. Klug, J. McCord,* Kiel University, Germany, *A. Beyer, A. Gölzhäuser*, Bielefeld University, Germany

Microscopes based on gas field ion sources offer surface-sensitive, high resolution imaging and state of the art nano-machining.[1] It was further shown that light ions like helium or neon enable a modification of the magnetic properties, e.g., turning thin films from paramagnetic to ferromagnetic state, without significant sputtering.[2]

In this work, two-dimensional ion bombardment induced magnetic patterning (IBMP)[3] is demonstrated with a helium ion microscope to create magnetic domains in an exchange biased thin film system. Such a system consists of a thin ferromagnetic layer coupled to an underlying antiferromagnet. Low dose helium ion irradiation at an energy of 15 keV in an external magnetic field leads to a new, remanent magnetization direction, determined by the external magnetic field. By subsequently patterning the sample in differently orientated external magnetic fields, complex magnetic domain patterns such as chiral structures can be written. Based on magnetic force microscopy and optical Kerr microscopy, we will discuss the achievable resolution as well as the shapes of different artificial magnetic domains.

[1]G. Hlawacek and A. Gölzhäuser (eds),Helium Ion Microscopy (Springer International Publishing, Switzerland, 2016.).

[2]F. Roder, G. Hlawacek, S. Wintz, R. Hubner, L. Bischoff, H. Lichte, K. Potzger, J. Lindner, J. Fassbender, and R. Bali, Scientific reports 5, 16786 (2015).

[3]A. Gaul, S. Hankemeier, D. Holzinger, N.D. Müglich, P. Staeck, R. Frömter, H.P. Oepen, and A. Ehresmann, Journal of Applied Physics 120, 33902 (2016).

11:20am **HI+BI+NS+TR-ThM11 Characterisation of Nanomaterials on the Helium Ion Microscope using Correlative Secondary Electron and Mass Filtered Secondary Ion Imaging**, *J.-N. Audinot, D.M.F. Dowsett, F. Vollnhals, T. Wirtz,* Luxembourg Institute of Science and Technology (LIST), Luxembourg, ***John A. Notte***, Carl Zeiss Microscopy, LLC

In order to add nano-analytical capabilities to the Helium Ion Microscope, we have developed a Secondary Ion Mass Spectrometry (SIMS) system specifically designed for the Zeiss ORION NanoFab [1-3]. SIMS is based on the generation and identification of characteristic secondary ions by irradiation with a primary ion beam (in this case helium or neon). It is an extremely powerful technique for analysing surfaces owing in particular to its excellent sensitivity (detection limits down to the ppb are possible, so that SIMS can be used to detect both major and trace elements), high dynamic range (a same signal can be followed over several orders of magnitude), high mass resolution and ability to differentiate between isotopes.

In SIMS, the typical interaction volume between the impinging ion beam and the sample is around 10 nm in the lateral direction. As the probe size in the HIM is substantially smaller (both for He and Ne), the lateral resolution on the integrated HIM-SIMS is limited only by fundamental considerations and not, as is currently the case on commercial SIMS instruments, the probe size [4,5]. We have demonstrated that our instrument is capable of producing elemental SIMS maps with lateral resolutions down to 12 nm [3-5].

Furthermore, HIM-SIMS opens the way for in-situ correlative imaging combining high resolution SE images with elemental and isotopic ratio maps from SIMS [4,5]. 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.

In this talk, we will present a number of examples taken from various fields of materials science (battery materials, solar cells, micro-electronics, coatings) and life science (nanoparticles in creams and biological tissues) to show the powerful correlative microscopy possibilities enabled by the integrated HIM-SIMS instrument.

[1] T. Wirtz, N. Vanhove, L. Pillatsch, D. Dowsett, S. Sijbrandij, J. Notte, Appl. Phys. Lett. 101 (4) (2012) 041601-1-041601-5

[2] L. Pillatsch, N. Vanhove, D. Dowsett, S. Sijbrandij, J. Notte, T. Wirtz, Appl. Surf. Sci. 282 (2013) 908-913

[3] T. Wirtz, D. Dowsett, P. Philipp, Helium Ion Microscopy, edited by G. Hlawacek, A. Gölzhäuser, Springer, 2017

[4] T. Wirtz, P. Philipp, J.-N. Audinot, D. Dowsett, S. Eswara, Nanotechnology 26 (2015) 434001

[5] P. Gratia, G. Grancini, J.-N. Audinot, X. Jeanbourquin, E. Mosconi, I. Zimmermann, D. Dowsett, Y. Lee, M. Grätzel, F. De Angelis, K.Sivula, T. Wirtz, M. K. Nazeeruddin, J. Am. Chem. Soc. 138 (49) (2016) 15821–15824

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