Helium Ion Microscopy Focus Topic
Room: 203 A - Session HI-ThA

Imaging and Lithography with Helium Ions
Moderator: G. Hlawacek, University of Twente, Netherlands

2:00pm **HI-ThA1 Imaging of Biological Cells and Carbon Nanomembranes with Helium Ion Microscopy**, ***A. Beyer***, Bielefeld University, Germany **INVITED**

In my talk, I will present a helium-ion microscopy (HIM) study of biological cells and carbon nanomembranes (CNMs). The cells were imaged without conductive coating and the attainable high resolution allowed imaging of extremely small features at the cell surface. Charging of these specimens was effectively compensated by the electron flood gun.

HIM is also a very efficient imaging tool for characterizing CNMs which exclusively consist of surface-near atoms. These 1 nm thick membranes yield a high secondary electron signal, provided that charging is absent. This condition is fulfilled by choosing a suitable beam current or employing the electron flood gun.

Aspects of helium ion beam lithography will also be discussed. In particular, I will show the fabrication of patterned CNMs by local cross-linking of aromatic self-assembled monolayers with helium ions.

2:40pm **HI-ThA3 Patterning of Sub-10 nm Optical Apertures on Single Crystal Metallic Films with the Helium Ion Microscope**, ***D. Pickard***, Unaffiliated, *H.F. Hao, V. Viswanathan,* National University of Singapore, *M. Bosman,* IMRE , A*STAR, *J. Dorfmüller, H. Giessen,* University of Stuttgart, Germany, *A.S. Yusuf, Z.K. Ai, Y. Wang, M. Mahmoudi*, National University of Singapore **INVITED**

Metallic nanostructures, resonant at optical frequencies, provide controlled enhancement and concentration of electromagnetic energy in the near-field. One example is the enhanced transmission and field localization through sub-wavelength C-apertures on thin metallic films, where transmission gains of 6x and field enhancements of 550x have been reported by others. [1] [#_ftn1]-[2] [#_ftn2] Typically, the critical dimensions of optical apertures are on the order of tens of nanometers (for low-order structures in the near-IR). These dimensions are accessible with conventional focused gallium ion beam patterning, and this has traditionally been the technique used for fabrication. However, for patterning dimensions smaller than 30 nm (typical of visible and ultraviolet structures, or higher order resonant structures), gallium based systems have not performed as successfully. The most critical shortcomings of Ga+ patterning in this regime are the degradation of the fine structure by etching with the beam's tail, and the shift in the optical characteristics or quenching of the resonant metal's properties due to gallium implantation. gallium implantation [3] [#_ftn1].

We have employed the Helium Ion Microscope to directly pattern high order, sub-10 nm optical fractal apertures (free of implanted metal impurities) through optically thick, polycrystalline metallic films and single crystal metal nanoplatelets. Our experimental measurements of the near-field mode profiles with electron energy loss spectroscopy (EELS) demonstrate tight field confinement in multiple modes as predicted by FDTD simulations. This has resulted in extremely high fidelity, optically-active resonant structures (down to 10 nm critical dimension). Controlled fabrication of structures on this size scale opens fascinating prospects for engineering complex multi-modal structures which were previously unrealizable by other techniques. We report our investigations in this arena and detail a variety of novel structures that are now accessible with this technique.

[1] X.L. Shi, L. Hesselink, J. Opt. Soc. Am. B **21**, 13 (2004)

[1] B. Lee, I.M. Lee, S. Kim, D. Ho Oh, L. Hesselink, J. Mod. Optic. **57**, 19 (2010)

[1] J.B. Leen, P. Hansen, Y.T. Cheng, L. Hesselink, OptLett **33**, 23 (2008)

3:40pm **HI-ThA6 Characterization of 2D Materials by using Scanning Helium Ion Microscopy**, ***H.X. Guo***, *J.H. Gao, D. Fujita*, National Institute for Materials Science, Japan

Two dimension(2D) materials, such as graphene or hexagonal boron nitride (h-BN), have layer structures which are different from bulk materials [1]. Normally, different layers of the 2D materials were combined by a weak band compared with the interlayer chemical bands. This makes the 2D materials special in physical and chemical properties such as optical properties or band structures. Many methods have been applied to research 2D materials, such as Raman microscopy, scanning probe microscopy, transmission electron microscopy and others.

In this presentation, we will show our investigation of 2D materials with scanning helium ion microscopy(SHIM) and other methods. The BN nano sheets and quasi-free standing graphene were synthesized by BN and carbon segregation on surface of metallic substrate [2]. We characterized the number and morphology of the h-BN by using scanning electron microscopy(SEM) and SHIM. On the basis of the interaction between the scanning particles (electrons and helium ions) and h-BN nanosheets, we interpreted an exponential relationship between the intensities of images and the number of layers. Inelastic mean free paths (IMFP) of electrons and helium ions in h-BN nano sheets were calculated approximately. The quasi-free standing graphene on metallic substrate was characterized by scanning kelvin probe microscopy, scanning Auger microscopy, SEM and SHIM. The SHIM images of such samples show high surface sensitivity and space resolution. The advantage of different characterization were interpreted in this presentation.

[1] Mingsheng Xu, Tao Liang, Minmin Shi, and Hongzheng Chen, Chem. Rev, DOI: 10.1021/cr300263a.

[2] Mingsheng Xu, Daisuke Fujita, Hongzheng Chen, and Nobutaka Hanagata, Nanoscale, 3, 2854(2011)

4:00pm **HI-ThA7 Helium Ion Microscopy of CVD-grown Films: Transition Metals and Catalytically Active Transition Metal Oxides**, ***H. Vieker***, *A. Beyer, Z.-Y. Tian, P. Mountapmbeme Kouotou, A. El Kasmi, K. Kohse-Höinghaus, A. Gölzhäuser*, Bielefeld University, Germany

Pulsed spray evaporation – chemical vapor deposition (PSE-CVD) is a cheap and scalable route to prepare specifically engineered layers, e.g. metallic and metal oxide films. The latter type is a promising class of materials for developing new efficient catalysts. Such developments require a detailed analysis of the surface morphology which significantly affects the catalytic activity. Among other methods, we employed helium ion microscopy to investigate such films. The high resolution and the high depth of focus are very advantageous in imaging these highly corrugated surfaces. We revealed extremely small surface structures which yield new insights in the morphology of these films. In this study, changes in the morphology of metallic as well as metal oxide PSE-CVD layers by varying the deposition temperature, precursor type, pressure and composition were investigated which leads to a better understanding of the involved growth processes and the catalytic activity.

4:20pm **HI-ThA8 Helium Ion Microscopy of Blood Clot Microstructure**, ***S.A. Boden***, University of Southampton, UK, *G. Mills, P.A. Evans,* Morriston Hospital, UK, *M. Bagnall, H.N. Rutt*, University of Southampton, UK

In addition to a smaller probe size and so higher resolution imaging, a key advantage of the helium ion microscope (HIM) is the large depth-of-field (DOF) it provides, typically five times larger than that of a scanning electron microscope [1] . Here we exploit the high resolution and large DOF of the HIM in a study of how diluting blood affects the resulting blood clot microstructure.

Blood clot formation involves the polymerization of fibrinogen into fibrin, forming a fibrous mesh which binds the clot together. Clinicians are looking for better ways of determining what effect dilution has on clot formation to improve the management of fluid replacement therapy. One such method being developed is a rheological technique that measures the gel point (GP) of clotting blood and the incipient clot microstructure complexity at the gel point (the fractal dimension, D_f) [2] . In this study, HIM is used to characterize fully matured clots to demonstrate that variations in the haemorheological properties measured during clotting (D_f), as a result of diluting with isotonic saline, can be correlated with changes in the resulting microstructure of the mature clots. Demonstrating the link between D_f of the incipient clot and the resulting clot microstructure is an important step in developing D_f as a biomarker for use in management of fluid replacement therapy and potentially as a point of care test.

HIM is used to image blood clots formed from samples diluted by isotonic saline to various degrees (0 – 60% dilution), so that the average fibril width can be measured and compared to the D_f of the sample. The large DOF of the HIM (due to its small beam convergence angle) is particularly useful when imaging blood clot microstructure because of their inherent 3D nature and high degree of surface topography. A large number of fibrils appear in focus within one image and so a large number of width measurements can be extracted. Furthermore, the large DOF allows the capture of high quality stereopairs from which the 3D structure of the fibrin network can be analyzed. In addition, the HIM enables imaging of the uncoated fibril surface at a higher resolution compared to SEM which could lead to a deeper understanding of the effects of dilution on blood clot fibril structure.

[1] B. W. Ward, J. A. Notte, and N. P. Economou, *Journal of Vacuum Science and Technology B*, vol. 24, no. 6, pp. 2871–2874, 2006.

[2] P. A. Evans, K. Hawkins, R. H. K. Morris, N. Thirumalai, R. Munro, L. Wakeman, M. J. Lawrence, and P. R. Williams, *Blood*, vol. 116, no. 17, pp. 3341–6, Oct. 2010.

4:40pm **HI-ThA9 Formation of "Ridge" like Structures for Possible Suppression of Secondary Electron Emission on Cu and Al Surfaces**, ***V. Shutthanandan***, *S. Manamdhar, M.I. Nandasiri, A. Devaraj, D.E. Perea, S.A. Thevuthasan, D.M. Asner,* Pacific Northwest National Laboratory, *D. Rubin, W.H. Hartung, Y. Li*, Cornell University

The performance of future high intensity positron and proton accelerators is likely affected by the electron cloud (EC) generated by the secondary electrons yield (SEY) created from the inner wall of vacuum chambers. One of the promising techniques for suppressing EC formation in regions with magnetic fields is the use of modified surfaces such as longitudinally grooved chamber surfaces to help suppress the escape of secondary electrons from the walls into the central volume of the vacuum chamber. However, the use of macroscopic structures in chambers increases the vacuum chamber impedance and can adversely impact a high intensity beam, particularly if the beam motion has a significant component perpendicular to the direction of the structures. A possible way to obtain the same "geometric" suppression of the electron cloud with less impact on the particle accelerator beams of interest is to prepare the vacuum chamber surfaces with microstructures produced by ion bombardment. In this project we have investigated the secondary electron yield from the ion beam modified Cu and Al surfaces, which are typically employed in high energy positron/electron circular accelerators, and correlate the yield to the chemical and structural properties of the microstructures generated by the high energy ion beam and their interfaces. "Ridge" like structures were generated by irradiating the surfaces using 1 MeV gold, copper and aluminum ions at 60 degrees or more from the normal to the surface. Modified sample surfaces were investigated using Rutherford backscattering spectrometry (RBS), X-ray photoelectron spectroscopy imaging (XPS), Helium ion microscopy (HIM), Atomic Force Microscopy (AFM), high-resolution transmission electron microscopy (HRTEM) and Atom probe tomography (APT). HIM micrographs obtained from the as implanted samples show that the surface of the implanted region underwent substantial rearrangement and formed "ridge" like structures at higher ion fluence. These "ridge" like structures are formed throughout the implanted region with an average height of 1 to 2 microns. The measured secondary electron yield from these structures will be correlated to the microstructures and the combined results will be presented.

5:20pm **HI-ThA11 Towards SIMS on the Helium Ion Microscope: Detection Limits and Experimental Results on the ORION**, *T. Wirtz*, ***D. Dowsett***, Centre de Recherche Public – Gabriel Lippmann, Luxembourg, *S. Sijbrandij, J.A. Notte*, Carl Zeiss Microscopy

The ORION Helium Ion Microscope (HIM) has become a well-established tool for high resolution microscopy [1] and nanofabrication [2]. The source can operate with both helium and neon [3]. While secondary electrons are used for high-resolution high-contrast imaging, some compositional information can be obtained from backscattered He/Ne ions.

In order to get chemical information with much higher sensitivity, we have investigated the feasibility of performing Secondary Ion Mass Spectrometry on the HIM [4]. In order to reach these objectives, the secondary ion formation process under He^+ and Ne^+ bombardment has been investigated and optimized along with the experimental beam parameters such as spot size and dwell time [5]. We have determined experimentally secondary ion yields under helium and neon bombardment for a range of semiconductor and metal samples. While basic yields are low due to the use of noble gas primary ions, they may be enhanced by several orders of magnitude for both negative and positive secondary ions by caesium and oxygen flooding respectively [6]. Measurement of yields has allowed us to determine detection limits for these samples under typical ORION imaging conditions.

More recently an extraction and detection system for secondary ions has been developed for the Helium Ion Microscope by the CRP - Gabriel Lippmann. We have investigated secondary ion emission for semiconductor (Si, InP and GaAs) and metal (Cu, Ni) samples on the ORION. Both total secondary ion depth profiles and secondary ion images have been obtained under helium and neon bombardment.

The obtained results are very encouraging and the prospects of performing SIMS on the ORION are very interesting. In this paper we will present an overview of our results to date and first experimental results of secondary ion detection on the Helium Ion Microscope.

References

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[2] D. Winston et al, Nano Letters 11 4343 (2011)

[3] F. Rahman et al., Scanning 33 (2011) 1

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

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5:40pm **HI-ThA12 Blunt Tungsten Tip Cleaning with Nitrogen Gas Reaction in Ultra-high Vacuum**, ***I.-Y. Park***, *B. Cho, C. Han, J. Kim, S.J. Ahn*, KRISS, Republic of Korea

The ultra-sharp tips are an essential part for probing and charged particle beam generation in current high resolution microscope. There are a lot of required conditions of tip fabrication and preparation for the high performance of microscopes. Among them, tip cleanliness is very important for the stable and high charged particle current. Here, we describe a simple and efficient method to clean the tungsten tip under UHV(ultra-high vacuum).

Tungsten is preferably adopted for tip material because extremely sharp tip can be easily obtained through electrochemical etching and has higher evaporation field value than ionization field value of rare gases. However, the drawback is poor resistance to surface oxidation; also the surface is contaminated during etching and exposure to atmosphere. In order to eliminate the contaminants, a proper annealing treatment in UHV can remove the contaminant from the tip surface and field evaporation (desorption) can eliminate intensively in the vicinity of the tip apex. In case of annealing, the tip is generally cleaned at approximately 1000 K for several seconds or minutes. However, high temperature could induce the surface diffusion which causes atoms to migrate from the tip apex to tip shank, thereby increasing the radius of tip[1]. Field evaporation cleaning method needs the ultra-sharp tip to produce the field enhancement at the end of tip with a few kV, otherwise it is difficult due to breakdown of high voltage. The nitrogen gas reaction with tungsten surface can sharpen the tip until atomically defined level[2], so we adopted this phenomenon to clean the tip which rarely occur field evaporation with less than 10 kV due to large radius of tip. Firstly, we annealed the tip about 700 K for a few seconds. After that, we inject the nitrogen gas around 10^{-8} mbar and helium gas up to 10^{-5} mbar to observe directly the cleaning process through an atomic-scale FIM(field ion microscope) in real time. We can monitor the cleaning and sharpening process simultaneously with FIM. This whole process starts from a base pressure in the low 10^{-10} mbar range; during the cleaning, the chamber is back filled with 10^{-5} mbar. On this, vacuum pressure returns to the 10^{-10} mbar with pumping system. The technique considered here can find applications in blunt tip cleaning and making from blunt tip to the few atom tip sequentially in UHV condition.

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