

Thursday Morning, October 31, 2013

Helium Ion Microscopy Focus Topic

Room: 203 A - Session HI-ThM

Basics of Helium Ion Microscopy

Moderator: A. Gölzhäuser, Bielefeld University, Germany

8:40am **HI-ThM3 Imaging with Helium Ions - A New Detector Regime with New Challenges and New Opportunities**, J.A. Notte, Carl Zeiss Microscopy **INVITED**

The helium ion microscope (HIM) is now accepted as a valuable instrument on par with the SEM, the TEM, and Gallium FIB within the family of charged particle microscopes. The introduction of the HIM was sparked by the successful commercialization of the gas field ion source (GFIS), and several scientific papers have already addressed its principles of operation. However, at the opposite end of this instrument, secondary electron (SE) detector has received relatively little attention despite several interesting characteristics. This presentation will give an overview of the HIM's SE detector and the unique circumstances in which it is used.

The SE detector on the HIM commonly operates under conditions and regimes that are distinctly different from the SEM. First, the overall efficiency is of prime importance since the excessive beam currents or excessive averaging often induce undesired sputtering or implantation in some samples. Generally, the areal dosages (measured ions / cm²) that are required must be kept to a minimum, sufficient to achieve the minimum required signal to noise ratio (SNR) and the necessary field of view (FOV). Second, the probe current that is used in the HIM is commonly as small as 0.5 pA or less. While such small currents are adequate for high magnification imaging, the ions arrive infrequently with long intervals wherein no meaningful information is acquired. In contrast, for most charged particle microscopes the incident particles arrive so frequently that their resulting signals overlap. Third, when the incident ions do arrive, each one produces an abundance of secondary electrons - usually three or more - and substantially more for glancing angles. Thus, the amplitude of the detected signal conveys more information than the frequency of the pulses.

While some of these three conditions present challenges for the instrument and for the operator, they also represent a new regime for signal acquisition. Towards that end, a variety of new techniques have been tested with computer simulations and with real experiments. For example, pulse counting has been implemented with somewhat surprising results. Signal integration (as opposed to simple averaging) has also been thoroughly investigated on the instrument with very favorable results. Finally, a new imaging technique called 'quotient mode' has been investigated and seems to offer a unique advantage of a significantly improved SNR available to the HIM.

9:20am **HI-ThM5 Interaction of Energetic Ions and Electrons with Two-Dimensional Materials**, A.V. Krasheninnikov, Aalto University and University of Helsinki, Finland **INVITED**

The experiments (see [1,2] for an overview) on the bombardment of 2D materials with energetic particles indicate that irradiation can have beneficial effects on such targets and that electron or ion beams can serve as tools to change the morphology and tailor the properties of such materials. It is also evident from the experimental and theoretical data obtained so far that the conventional theory of defect production in bulk materials not always works at the nanoscale or it requires considerable modifications. In this talk, our latest theoretical results on the response of graphene [3-6] and inorganic 2D materials like BN [7] and dichalcogenides (MoS₂, etc) [8] to electron and ion irradiation will be presented, combined with the experimental results obtained in collaboration with several groups [5,6,8-11]. I will also touch upon applications of time-dependent density-functional theory (TD-DFT) to ion electronic stopping calculations. I will show that combining TD-DFT with Ehrenfest dynamics and PAW approach, one can calculate electronic stopping power from first principles for a specific trajectory and different charge states of the projectile, and the results of calculations are in an excellent agreement with the experimental data.

1. A. V. Krasheninnikov, K. Nordlund, JAP 107 (2010) 071301.
2. A.V. Krasheninnikov and F. Banhart, Nature Materials, 6 (2007) 723.
3. O. Lehtinen et al., PRB 81 (2010) 153401.
4. E. H. Åhlgren, et al., APL 100 (2012) 23310.
5. J. Kotakoski, et al., PRL 106 (2011) 105505.
6. J.C. Meyer et al., PRL 108 (2012) 196102.
7. N. Berseneva, et al., PRL 107 (2011) 035501.

8. H.-P. Komsa, et al., PRL 109 (2012) 035503.

9. R. Nair, et al., Nat. Phys. 8 (2012) 199.

10. M. Kalbac et al., Advanced Materials 25 (2013) 1004.

11. S. Standop et al., Nano Letters (2013) in press.

10:40am **HI-ThM9 Imaging of Graphene Films by Helium Ion Microscope**, S. Ogawa, T. Iijima, S. Nakaharai, M. Hayashida, S. Sato, National Institute of Advanced Industrial Science and Technology (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 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 nano beam using the helium ion microscope (HIM) [3] and by controlling electrical properties of the graphene films themselves by the helium ion dose [4]. On the other hand it is difficult to characterize whether the graphene films on the silicon oxide layer were single layers or not.

Graphene flakes were mechanically exfoliated from a crystal of HOPG using adhesive tape, and then deposited on a silicon wafer with a 300-nm-thick surface thermal oxide layer. The number of graphene layers was identified by sight with an optical microscope based on interference color and then characterized by HIM using brightness ratio of the graphene films and the silicon oxide surface. Brightness of the surface of the silicon oxide showed linear dependency to beam currents but with some offset for different HIM contrast conditions, and to normalize the brightness ratio at several imaging conditions, the brightness was compensated by the offset. HIM images show higher brightness ratio for single layer graphene films with darker brightness than multi-layer graphene films and much higher spatial resolution than the optical microscope, while it is not sufficient to determine layer numbers of the films so far. Helium ions dose higher than 1E16/cm² decreased the brightness ratio. Detail of the brightness ratio and its dependency on the layers of the graphene films will be discussed.

This work was partly supported by JSPS through the "FIRST Program," initiated by CSTP, Japan.

[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)

11:00am **HI-ThM10 Secondary Electron Contrast for Few Layer Graphene in Helium Ion Microscope**, Y. Zhou, H. Zhang, Trinity College Dublin, Ireland

The one layer, atomic thin graphene has attracted numerous interests since its discovery, and reveals great potential application in the fields of nano-devices. However, the electrical structure for few layer graphene will be influenced by the layer thickness, thus the device performance will also be affected. As a result, the determination of graphene layer thickness becomes important. Recently, graphene secondary electron (SE) contrast in scanning electron microscope (SEM) provides a new method to determine graphene layer thickness. However, the mechanism of graphene SE contrast is still unclear, which limits the application and needs further exploration.

The recent developed Helium Ion Microscope offers a new and effective tool to investigate the mechanism of graphene SE contrast. The ultimate small source size, small energy dispersion and high gun brightness of HIM brings out a sub-nanometer resolution for graphene metrology. Meanwhile, HIM also has a lower SE energy distribution than SEM. thus SEs in HIM will be more surface sensitive. All the advantages of HIM reveal that it is an effective tools to study the SE emission in graphene from a new aspect, and may help us to clarify some uncertainty of the contrast mechanism.

Here, we used a Carl Zeiss Orion Helium Ion Microscope to investigate graphene SE contrast at the typical acceleration voltage of 30KV. Exfoliated few layer graphene flakes on silicon oxide substrates exhibited higher SE yield (brighter contrast) than substrates. Graphene layers could also be clearly distinguished for more than five layers with almost linear SE contrast dependence. An ultra large SE yields more than 200% was measured from the free-standing graphene. Thus we attributed the SE emissions in HIM and low voltage SEM to the SE emission from graphene itself, with very little contribution from substrate SE attenuations. Similar SE contrast variation and high SE yields for few layer graphene flakes could be observed in SEM at very low acceleration voltages below 0.2KV. We also observed the influence of graphene work function to the SE contrast for graphene flakes less than four layers.

The results could help us to understand the graphene SE contrast mechanism more clearly. The linear layer dependence SE contrast also offered an effective method to determine the graphene layer thickness.

[3] M. Rudneva, E van Veldhoven, S.K. Malladi, D.Maas, H.W. Zandbergen. *J. Mat. Sci.*, 28, 8, (2013), 1013-1020

11:20am **HI-ThM11 Monte Carlo Simulations of Helium and Neon Ions Beam Induced Deposition and Etching.** *R.T. Timilsina*, The University of Tennessee Knoxville, *D.A. Smith, P.D. Rack*, The University of Tennessee Knoxville and Oak Ridge National Laboratory

The new Gas Field Ion Microscope is able to deposit and etch material at the nanoscale in a highly controlled manner, but in order to exploit this capability it is necessary to have a detailed quantitative model of the process. A Monte Carlo simulation for He⁺ and Ne⁺ ion beam induced deposition (and etching) has been developed which provides data in excellent agreement with the observed experimental results over a wide range of experimental conditions. The ion beam induced nanoscale synthesis of PtC_x (where x~5) using the trimethyl (methylcyclopentadienyl)platinum(IV) (MeCpPt^{IV}Me₃) precursor is investigated by performing Monte Carlo simulations of helium and neon ions integrated with a gas handling routine to mimic the precursor adsorption and decomposition. The simulation results show that the helium beam leads to more lateral growth relative to the neon beam because of its larger interaction volume. The lateral growth of the nanopillars is dominated by molecules deposited via secondary electrons in the both simulations. Using a low precursor residence time of 70μs resulting in an equilibrium coverage of ~4%, the neon simulation has a lower deposition efficiency (3.5%) compared to that of the helium simulation (6.5%). At larger residence time (10ms) and consequently larger equilibrium coverage (85%) the deposition efficiencies of helium and neon increased to 49% and 21%, respectively; which is dominated by increased lateral growth rates leading to broader pillars. The nanoscale growth is further studied by varying the ion beam diameter at 10 ms precursor residence time. The study shows that total SE yield decreases with increasing beam diameters for the both ion types. Finally, experimentally we have shown that He ion deposited material has a larger room temperature resistivity (~3.5x10⁴ - 2.2x10⁵ μΩ-cm) and temperature dependent transport behavior consistent with a granular material in the weak intergranular tunnel coupling regime. Conversely Ne ion deposited material has a much lower room temperature resistivity (~600 - 3.0x10³ μΩ-cm) and temperature dependent electrical behavior representative of strong intergranular coupling. The Ne ion deposited nanostructure has larger platinum nanoclusters, which is rationalized via Monte-Carlo ion-solid simulations that show the neon energy density deposited during growth is much larger due to the smaller ion range as shown in The observed platinum grain coarsening and subsequently lower resistivity for the Ne ions beam induced deposits is correlated to the enhanced platinum mobility via the enhanced nuclear stopping of the Neon ions.

11:40am **HI-ThM12 Helium Ion Microscope; a Single Beam for Imaging and Fabrication.** *E. van Veldhoven, N.B. Koster, F.T. Molkenboer, D.J. Maas*, TNO Technical Sciences, Netherlands, *H.W. Zandbergen, P.F.A. Alkemade*, TU Delft, Netherlands

At TNO, we focus on imaging novel materials and developing new nanofabrication applications for mainly the semiconductor and solar industry. The helium ion microscope (Orion plus Zeiss) creates new opportunities for exploration [1]. The microscope provides a sub nanometer spot size with ions that hardly scatter back. For the secondary electron image, it produces only low energy SE. The obtained image has an unique contrast, which contains information about the morphology and often grain and material contrast are clearly present. The SE's appear only from a very local interaction volume which gives a high surface sensitivity. Single layers, small particles and thin layers of contamination can be made relative easily visible even on charging surfaces which are of great interest in the semiconductor and solar industry.

The small interaction volume created by charged species is unique and opens new ways for nanofabrication. Novel recipes are being developed to obtain high, small and dense deposition yields for Pt-precursor and small and dense high etching yields with the XeF₂-precursor. With the Oxford OmnigisTM and the Raith Elphy MultibeamTM. A wide set of parameters like beam current, acceleration voltage, refreshment rates, gas flows, writing patterns are being included in our research for true 3D-nanofabrication. Direct sputtering of materials for thin films are highly promising since no helium can stay trapped in the bulk material [2]. Recently we showed that it is possible to perform incisions into bulk material without any helium trapping yielding in high quality TEM samples [3]. The HIM enables a novel way for dense and high resolution nanofabrication and imaging.

[1] D Maas, E van Veldhoven, P Chen, V Sidorkin, H Salemink, E van der Drift, P Alkemade; *Proceedings. of SPIE 7638* (2010)

[2] M. M. Marshall, J. Yang, A.R. Hall, *Scanning*, 34, 2 (2012), 101-106

Thursday Afternoon, October 31, 2013

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. Dorfmueller*, *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] 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]

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. Götzhäuser*, 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. Shuthanandan, 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

[1] L. Scipioni, C.A. Sanford, J. Notte, B. Thompson, and S. McVey, *J. Vac. Sci. Technol. B* 27, 3250 (2009)

[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. Sijbrandij and J. Notte, *Appl. Phys. Lett.* 101 041601 (2012)

[5] D. Dowsett, T. Wirtz, N. Vanhove, L. Pillatsch, S. Sijbrandij and J. Notte, *J. Vac. Sci. Technol. B* 30 06F602 (2012)

[6] P. Philipp et al., *Int. J. Mass Spectrom.* 253 (2006) 71

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.

Thursday Afternoon Poster Sessions

Helium Ion Microscopy Focus Topic

Room: Hall B - Session HI-ThP

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Aspects of Helium Ion Microscopy Poster Session

HI-ThP2 Imaging Nascent Soot Particles: Tiniest Soot Particles are Not Structurally Homogeneous. *M. Schenk*, Bielefeld University, Germany, *S. Lieb*, University of Southern California, *H. Vieker*, *A. Beyer*, *A. Götzhäuser*, Bielefeld University, Germany, *H. Wang*, University of Southern California, *K. Kohse-Höinghaus*, Bielefeld University, Germany

Structural and morphological probing of nascent soot has been a challenging problem historically. Transmission electron microscopy (TEM) shows that mature soot is usually composed of stacks of polycyclic aromatic hydrocarbons arranged in a turbostratic fashion with a certain degree of microscopic crystallinity. Whether this observation can be extrapolated to nascent soot undergoing rapid mass and size growth in a flame remains an open question. In particular, recent studies show converging evidence that nascent soot may have an aromatic core-aliphatic shell structure not seen from previous TEM studies. The aliphatic component in the shell appears to be weakly bound among itself and with the aromatic core. In TEM probing, the possibility of high-energy electron beam damage or structural modification particles also remains an open question. Evidence of this possibility emerged as early as the mid 1980s when Iijima (S. Iijima, *J. Electron Microsc.* 34 (1985) 249-265) demonstrated the structural instability of gold nanoparticles ~ 3 nm in diameter under electron beam irradiation in a TEM. Sample damage can arise from electron beam induced chemical bond breaking and/or evaporation of the aliphatic component along with structural change and crystallization of the remaining particle material.

To explore the aforementioned problems and to find more suitable techniques, we report here results of two "softer" microscopic techniques: Helium Ion Microscopy (HIM) and phase imaging Atomic Force Microscope (AFM). In comparison to TEM, both techniques present far less sample damaging during imaging. The present study focuses on the HIM imaging of nanometer-sized soot particles sampled from a stagnation-point ethylene-oxygen-argon flame, under the conditions of Abid et al. (*A. D. Abid, Combust.Flame* 154 (2008) 775-788).

HI-ThP3 Helium Ion Microscopy as a Tool to Investigate Thin Layer Thicknesses. *H. Vieker*, *K. Rott*, *U. Werner*, *A. Beyer*, *G. Reiss*, *A. Götzhäuser*, Bielefeld University, Germany

The recently developed helium-ion microscope allows remarkable surface resolution with the secondary-electron (SE) detector. Simultaneously, backscattered ions can be detected that allow imaging with a substantially higher elemental contrast. This Rutherford backscattered ion (RBI) contrast depends mainly on the elemental composition of the investigated sample surface. The escape depth of backscattered ions is much larger than for secondary electrons. Thus whole layers with a wide range of thicknesses will contribute to a RBI image, whereas the SE image is far more surface sensitive.

In this contribution we examine RBI imaging as a tool to characterize thickness variations of layered samples with well defined compositions. The homogeneity of gold layers on silicon substrates is investigated and compared to simulations. The achievable spatial resolution as well as the use of reference samples to measure layer thicknesses will be addressed.

HI-ThP4 Helium Ion Microscopy and Ionoluminescence of Defects. *G. Hlawacek*, *V. Veligura*, *R. van Gastel*, *H.J.W. Zandvliet*, *B. Poelsema*, University of Twente, Netherlands

Defects are an unavoidable and often unwanted side product of Helium Ion Microscopy (HIM). We will discuss the role of defects and try to show examples of their useful application.

Point defects created using HIM can be analyzed in-situ using ionoluminescence. However, such point defects can also be exploited to create areas with specific optical properties, in particular areas that either absorb light or emit light of a certain wavelength when excited.

Going beyond normally used ion doses allows to investigate defect agglomeration, blister formation and the subsequent surface restructuring. We present examples of materials modification at doses starting from $1 \times 10^{17} \text{ cm}^{-2}$ up to $1 \times 10^{22} \text{ cm}^{-2}$. Examples of surface structures formed under extreme ion fluencies at different temperatures will be presented for a wide range of materials including technological relevant materials for nuclear applications.

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