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 / cm^2) 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.

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) ($MeCpPt^{IV}Me_3$) 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 ($\sim 3.5\times10^4$ - 2.2×10^5 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 ($\sim 600 - 3.0\times10^3$ 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 XeF_2-precursor With the Oxford Omnigis^{TM} and the Raith Elphy Multibeam^{TM}. 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

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

Authors Index

Bold page numbers indicate the presenter