

Tuesday Afternoon, November 1, 2011

Helium Ion Microscopy Focus Topic

Room: 106 - Session HI+AS-TuA

Basics of Helium Ion Microscopy

Moderator: A. Gölzhäuser, University of Bielefeld, Germany, V.S. Smentkowski, GE-GRC

2:00pm **HI+AS-TuA1 Principles of Helium Ion Microscopy, J.A. Notte, L. Scipioni, L.A. Stern, Carl Zeiss NTS** **INVITED**

The Helium Ion Microscope (HIM) consists of an interesting blend of long established technologies and recent state of the art engineering designs that enable superior charged particle scanning microscopy capabilities. They are capable of providing sub-nanometer spatial resolution with remarkable surface information and a unique ability to image insulating samples. HIMs share many similarities with Scanning Electron Microscopes (SEMs), but also embody new principles that uniquely differentiate HIM hardware and applications from traditional SEMs.

The most significant hardware difference of the HIM compared to SEM is the ion source. On the macroscopic scale the source appears very similar to a standard electron field emission source. However, the detailed tip geometry allows for much higher electric fields to be produced in the vicinity of the tip than what is found in traditional SEM field emission sources. The higher field enables ionization of the neutral helium gas which surrounds the tip, producing the needed helium ion beam. In addition to the high electric field requirement, it is necessary to keep the tip and surrounding imaging gas at cryogenic temperatures. The implementation of source cryogenics while operating the tip at ~ 35 keV, and also maintaining mechanical motion for both source translation and tilting, introduces significant engineering challenges in the design of a HIM.

Due to fundamental differences between helium ion and electron interactions with the sample under observation, the HIM is capable of producing images that are significantly different from those produced by traditional SEM. Since the entire electron population created due to an incident helium ion is of very low energy, only those electrons near the point of helium incidence are capable of escaping from the sample, resulting in images that are rich with surface information and possess superior spatial resolution. Due to the much higher secondary electron yield associated with helium bombardment of a sample relative to electron bombardment, and the fact that the incoming particles are positively charged, the net charge state of the sample is always positive, unlike the SEM case. Furthermore, due to the strong affinity of a helium ion to capture an electron, the net charge on the sample always exists as a surface charge. The positive surface charge can be easily neutralized with an electron flood gun, thus enabling charge free imaging on highly insulating samples. These various unique imaging principles make the HIM a versatile and unique imaging instrument.

2:40pm **HI+AS-TuA3 Design and Performance of a Near Ultra High Vacuum Helium Ion Microscope, R. van Gastel, University of Twente, The Netherlands, L. Barriss, J.A. Notte, Carl Zeiss NTS, G. Hlawacek, University of Twente, The Netherlands, L. Scipioni, A.P. Merkle, D. Voci, Carl Zeiss NTS, C. Fenner, L'Vestus Energy Inc., H. Zandvliet, B. Poelsema, University of Twente, The Netherlands**

The advent of He Ion Microscopy (HIM) as a new technique to image materials and microstructures has enabled a new look at materials that is based on the interaction of swift light ions with matter, as opposed to that of more commonly used high (and low) energy electrons [1]. Initial Carl Zeiss Orion® He Ion Microscope instruments have demonstrated high-resolution imaging, combined with great surface sensitivity, the ability to neutralize charge very efficiently, and with enhanced materials contrast when ion induced secondary electrons are used for imaging. The use of Rutherford backscattered ions to form images has provided a new imaging modality that emphasizes differences in elemental composition and it can also be used to probe samples in-depth.

The HIM provides obvious benefits in terms of novel modes of contrast, surface sensitivity, lateral resolution, depth of field and charge compensation. To achieve ultimate performance, the chamber vacuum of the existing platform may be improved. For instance, carbon deposits due to beam interaction are readily seen due to the surface sensitivity of the technique. At sufficiently high current densities the sharply focused He ion beam may very efficiently decompose or cross-link residual hydrocarbons that are present in the instruments vacuum, more so than an electron beam in a SEM setup. Not only can this obscure a clear view of the sample,

thereby negating the benefits of the small spot size, it also limits the available acquisition time for spectroscopic measurements. In addition to this, some materials (Au in particular) have yielded unexpectedly high sputtering rates. On the one hand, this has proven extremely useful in the field of nanopatterning for sensors, plasmonics or other device fabrication applications at the sub-10nm level when operating at high doses. On the other hand, it is undesirable when the instrument is used for materials characterization.

In this presentation we will discuss the basic considerations that went into the design of a Near-UHV (NUHV) Orion Plus® He Ion Microscope. We will detail how the improved vacuum level is anticipated to alter those processes that are directly relevant to the imaging performance of the instrument such as beam interaction in the surface region and the emission of secondary electrons. First applications that the instrument was used for will be highlighted and its impact in the areas of surface physics, notably catalysis, corrosion, and other research areas that require increased imaging sensitivity, both laterally and in depth, will be discussed.

References

[1] B. Ward, J. Notte, and N. Economou, *J. Vac. Sci. Technol.* 24, 2871 (2006).

3:00pm **HI+AS-TuA4 Sub-10 nm Scanning Helium Ion Beam Lithography, K. van Langen, E.W.J.M. van der Drift, Delft University of Technology, Netherlands, E. van Veldhoven, D.J. Maas, TNO, Netherlands, P.F.A. Alkemade, Delft University of Technology, Netherlands**

Since the launch of the novel sub-nanometer helium ion microscope by Zeiss / Alis in 2006 nanofabrication with this tool has gained a lot of interest [1]. Key characteristic in this matter is the directional interaction of the helium ion with matter with negligible backscattering. In ion milling it enables very steep structuring when compared to the Ga⁺ ion equivalent [2]. In a similar comparison helium ion beam-induced deposition in a precursor gas ambient yields tall and smooth nanostructures [3], partially also because the sputtering by helium ions is at least an order of magnitude lower than by gallium ions.

The present contribution deals with scanning helium ion beam lithography (SHIBL). Thusfar two initial SHIBL studies on hydrogensylsesquioxane (HSQ) resist were reported [4,5]. In the present work performance of SHIBL is compared with state-of-the-art electron beam lithography (EBL). As resist materials we explored HSQ, polymethylmethacrylate (PMMA), and the inorganic resist of aluminumoxide. The latter material choice is motivated by the need for enhanced mask selectivity in pattern transfer in the sub-10-nm area.

The results for HSQ and PMMA can be summarized as:

- smallest feature size of 5 nm, equivalent to the best EBL performance [6]
- clear pattern densities up to 10 nm full-pitch, which is better than in EBL
- sensitivity 1-2 orders of magnitude better than in EBL.

As for the inorganic resist, 5-nm features have been realized.

In a semi-quantitative and comparative approach the results will be explained and future prospects will be outlined.

1 R. Hill, F.H.M. Faridur Rahman, *Nucl. Instr. and Meth. A* (2010), doi:10.1016/j.nima.2010.12.123, in press

2 L. Scipioni, D. C. Ferranti, V. S. Smentkowski, R.A. Potyrailo, *J. Vac. Sci. Technol. B* (2010) 28: C6P18

3 P. Chen, E. van Veldhoven, C.A. Sanford, H.W.M. Salemink, D.J. Maas, D.A. Smith, P.D. Rack, and P.F.A. Alkemade, *Nanotechnol.* (2010) 21: 455302

4 V. Sidorkin, E. van Veldhoven, E. van der Drift, P. Alkemade, H. Salemink, D. Maas, *J. Vac. Sci. Technol. B* (2009) 27: L18

5 D. Winston, B.M. Cord, B. Ming, D.C. Bell, W.F. DiNatale, L.A. Stern, A.E. Vladar, M.T. Postek, M.K. Mondol, J.K.W. Yang, K.K. Berggren, *J. Vac. Sci. Technol. B* (2009) 27: 2702

6 H. Duan, D. Winston, J.K.W. Yang, B.M. Cord, V.R. Manfrinato, and K.K. Berggren, *J. Vac. Sci. Technol. B.* (2010) 28: C6C58

4:00pm **HI+AS-TuA7 Contrast Performance in Helium Ion Microscopy, D.C. Bell, Harvard University** **INVITED**

In order to achieve ultra high resolution imaging with secondary electron imaging, it is critical that the electric potential of the specimen surface is

well controlled. For electrically conductive samples this can be achieved by simply grounding the specimen. However, imaging of electrically insulating specimens can provide challenging or impossible to image due to uncompensated charge resulting from the electron or ion beam interaction with the specimen surface. The main reason for the uncompensated charge is that the insulating specimen has insufficient conductivity through mobility of either electrons or holes to quickly restore the neutrality of the scanned area. The buildup charge causes significant deflection and distortion of the ion or electron beam. Which is more appropriate, to use charge compensation with high kV helium ions or employ a low kV SEM image to obtain the required surface information? This paper will present a systematic examination of the surface information provided by both techniques, including SEM charge compensation mechanisms.

One key advantage of the Helium Ion Microscope technology in the case of imaging highly charging specimens is the electron flood gun can be utilized to neutralize the positive charge buildup and facilitate high-resolution imaging. A flood gun is used to charge the surface to a negative potential (using electrons as the neutralizing particles). When utilizing the electron flood gun, the electron beam and He ion beam are synchronized and adjusted with respect to one another, so that the low energy electrons are not interfering with the secondary electron imaging.

Some of our research from the past year has been surprising and may provide a foundation for a change in analysis techniques of different materials. The nature of the Helium ion beam interactions with the sample shows enhanced edge contrast which is especially useful for critical dimension measurements; one particularly interesting development is the imaging of non-conducting materials showing a contrast due to three apparent mechanisms simultaneously - atomic number, channeling contrast and a possible enhanced edge contrast. The advantages of Helium ion microscopy is still being investigated and still are proving some exciting results.

5:00pm **HI+AS-TuA10 Helium Ion Beam Induced Deposition Examined using a 3D Monte Carlo Simulation**, *D.A. Smith, P.D. Rack*, University of Tennessee Knoxville, *P.F.A. Alkemade, H. Miro*, Delft University of Technology, The Netherlands

The growth of nanostructures has traditionally been dominated by electron beam induced deposition (EBID) or gallium ion beam induced deposition (Ga-IBID). While EBID provides smooth sidewalls and good resolution for nanopillar growth, the cross-section for dissociation is low and etching is difficult as sputtering is negligible. Ga-IBID is a relatively faster method of producing nanostructures, however it suffers from lower resolution, alters deposited materials, and leaves an etching residue. A new tool in this field has been recently explored: the helium ion beam microscope. This tool has been modified to perform IBID using high energy helium ions. It has been found that He-IBID combines the high resolution of EBID with the speed of Ga-IBID. Moreover, there is less implantation damage and minimal sputtering compared to Ga beams.

To examine this process, a 3-dimensional Monte Carlo simulator has been designed based on ion-solid-precursor interactions. This simulation system, named EnvisION, can provide useful knowledge of how user-controlled parameters can be optimized for highly efficient growth of nanostructures using this tool. In this work, an in-depth explanation of the simulation will be presented, including an example of its use examining the growth efficiencies of nanopillars grown on a silicon substrate using the (CH₃)₃Pt(CpCH₃) precursor via He-IBID. Furthermore we compare how the morphology changes with dwell times, refresh time, precursor coverage and surface diffusion in order to span the range of growth regimes from mass-transport limited to reaction-rate limited deposition. The simulated morphologies predicted using the EnvisION simulator are compared to experimentally grown pillars to validate the simulation.

5:20pm **HI+AS-TuA11 TEM Specimen Preparation with Light Ions**, *L. Giannuzzi*, L.A. Giannuzzi & Associates LLC

Much research with light energetic ions such as He⁺ and Ne⁺ from gas field ionization sources has focused on imaging and nano-machining. It is a natural progression to question the viability of TEM specimen preparation using these light ions. Of vital importance for TEM specimen preparation quality is the understanding of surface ion implantation and amorphization damage. Theoretical calculations using SRIM indicate that there may be a damage trade off between vacancy formation, ion range, and dose. That is, the range of light ions is much greater than conventional heavy ions (e.g., Ga⁺), and can indeed penetrate directly through a TEM specimen. While this may indicate the possibility of light ions damaging the entire TEM specimen thickness, light ions produce far less vacancies per ion compared to heavy ions for the same dose. However, since the sputter yield of light ions is smaller than heavy ions, a larger dose of light ions may be necessary to achieve sufficient material sputtering. This theory will be supplemented with experimental results.

5:40pm **HI+AS-TuA12 The Possibilities of the HIM for Imaging and Nanopatterning of EUVL Masks**, *D.J. Maas, E. van Veldhoven, N.B. Koster*, TNO, Netherlands, *P.F.A. Alkemade, E.W.J.M. van der Drift*, Delft University of Technology, Netherlands

Although Helium Ion Microscopy (HIM) was introduced only a few years ago [1], many new application fields are emerging. Key issue is the directional interaction of the primary helium ion beam with the sample at and just below its surface with negligible backscattering. The sub-nanometer sized probe of the 10-35 keV ion beam generates Secondary Electrons (SEs) that have a typical energy between 0 and 20 eV. Taking all together the SE signal stems from an area that is very well localized around the point of incidence of the primary beam. This makes the HIM well-suited for both high-resolution imaging as well as high resolution nanofabrication [2]. We explore the possibilities to use the HIM simultaneously for imaging and nano patterning of EUVL masks.

The HIM is a high-resolution surface imaging tool. In practice, the optimum dose for imaging is a balance between maximizing S/N ratio, while minimizing sample damage. Imaging work at TNO van Leeuwenhoek Laboratory (VLL) [3] focuses at sensitive materials such as e.g. DUV and EUV resists and EUV masks, which are difficult to image in a SEM due to their charging behavior. An electron flood gun in the HIM offers effective charge cancellation, which enables high-resolution imaging of insulation structures and for pin pointing defects on a EUV reticle. In this presentation we will show images of particles down to 5 nm on reticles.

Furthermore, to explore the possibilities of the helium ion microscope as a nanofabrication tool, the HIM at the TNO VLL is equipped with a pattern generator and a gas injection system (GIS). This presentation will show our latest nano structuring results made with Helium Ion Beam Induced Processes: deposition and etching. It is expected that the unique capabilities of the HIM in combination with the GIS are suited for EUV mask repair. These capabilities offer the possibility of circuit repair in the latest and smallest semiconductor technology nodes (beyond 22 nm). In both cases sub-surface damage due to scattered He ions is a matter of concern and topic of investigation. At this moment we are capable of etching 13 nm lines with 25 nm spacing on a EUV dummy mask with approximately 80 nm of TaN absorber. Furthermore we demonstrate Pt deposited lines of 13 nm width at a 16 nm spacing.

References

- [1] J. Morgan, J. Notte, R. Hill, and B. Ward, *Microscopy Today* 14, (2006) 24
- [2] D.J. Maas et al., *Proc. SPIE Vol. 7638, 763614* (2010) 1-8
- [3] <http://www.vanleeuwenhoeklab.com/>

Helium Ion Microscopy Focus Topic

Room: East Exhibit Hall - Session HI-TuP

Aspects of Helium Ion Microscopy Poster Session

HI-TuP1 From HIM to NIM: The Prospects of a Neon Ion Microscope, *F.H.M. Rahman, L.A. Stern, J.A. Notte*, Carl Zeiss NTS

From the time of its conception, the gas field ion source (GFIS) was operated with a variety of gas species - each considered for some particular virtue that depended on the particular application. However, practical issues such as vibration, cost, and stability prevented the commercial introduction of the GFIS for 50 years. The one gas species that was deemed to be most suitable was helium, and this was recently offered as a commercial product in the form of the ORION helium ion microscope in 2006. Now with several years of continued learning, the neon GFIS is being reconsidered in order to determine its suitability for the GFIS and the applications that it might enable.

The virtues of neon arise from its intermediate mass, one third the mass of gallium, and five times the mass of helium. While the helium probe offers minimal damage under normal imaging dosages (10^{15} ions/cm²), the neon beam can sputter at much higher yield (typically 10 times the rate of helium - nearly half the yield of gallium). Compared to helium, the neon ions also penetrate less deeply, and produce many fewer sub-surface dislocations per surface sputtering event. For example, with a helium beam normally incident upon aluminum at 30 keV, there are about 1200 vacancies per sputtered atom according to SRIM. Under these same conditions, the neon beam produces just about 212 vacancies per sputtered atom, and these are located much closer to the surface. Also, the distribution of sputtering atoms is more localized to the incident beam location when neon is used. Compared to gallium, neon is expected to offer a much smaller probe size, and permit nanofabrication with much higher fidelity.

Experimental results will be presented to characterize the basic properties of the focused ion beam from our prototype neon GFIS system. Images will be provided to demonstrate our first cross-section milling and imaging characteristics.

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(See supplementary PDF online)

HI-TuP2 Helium Ion Microscope (HIM) Milling of Solid-State Nanopores for Single-Molecule Detection Devices, *A.R. Hall*, University of North Carolina Greensboro, *J. Yang, D. Ferranti, L.A. Stern, J. Huang, J.A. Notte*, Carl Zeiss NTS

We report the formation of solid-state nanopores using the highly focused ion beam and lithographic capabilities of a scanning Helium Ion Microscope (HIM). We will discuss several aspects of the fabrication process, offering the advantage of high sample throughput along with fine control over nanopore dimensions. We will compare characteristics of the resultant devices with those made by the established technique of transmission electron microscope milling and demonstrate the utility of our nanopores for biomolecular analysis.

HI-TuP3 Imaging and Identification of Self Assembled Monolayers using HIM, *G. Hlawacek, A. George, J.E. ten Elshof, R. van Gastel, H. Zandvliet, B. Poelsema*, University of Twente, The Netherlands

Helium Ion Microscopy (HIM) is a new and versatile tool for imaging and characterizing surfaces, buried interfaces, thin films and tackling many other problems in modern material science. HIM utilizes ionized Helium to scan the specimen surface. Secondary electrons created by the impinging ions allow to record morphology images with an unmatched lateral resolution of less than 0.35 nm. In addition, back-scattered ions carry the elemental information of the scattering partner - allowing for an elemental identification of the surface composition.

Here, we report on the visualization of thin self assembled monolayers (SAM) deposited on (001) silicon wafers, covered by a thin native oxide. In particular, SAMs formed by (3-Mercaptopropyl)trimethoxysilane (MPS) and Triethoxy-1H,1H,2H,2H-tridecafluoro-n-octylsilane (TDFOS) have been patterned into a rectangular stripe pattern using a two step gas-phase silanization process. The clever use of channeling into the underlying bulk (001) silicon, together with a work-function based evaluation of the secondary electron data allows a clear assignment of different sample areas to the different chemical species. This is possible for both the electron and the ion generated image. The importance of channeling to distinctly and visibly tag the different SAMs will be demonstrated.

HI-TuP4 Analysis of Metal Nanoparticles in Biological Tissues Specimens Using the Helium Ion Microscope, *V.S. Smentkowski, L. Denault, D. Wark*, GE-GRC, *L. Scipioni, D. Ferranti*, Carl Zeiss SMT

The Helium Ion Microscope (HIM) is a newly introduced instrument that has a number of beneficial characteristics that are of importance for the analysis of biological/tissue samples, including: (1) the ability to perform high lateral resolution imaging, (2) high depth of field, (3) and the ability to analyze charging samples. In this poster, we summarize the first HIM analysis of spleen tissue samples that have been treated with a metal contrast agent. We show the advantages of HIM over techniques such as Scanning Electron Microscopy (SEM). The HIM analysis are complimented by surface analysis using Time of Flight Secondary Ion Mass Spectrometry (ToF-SIMS) in order to demonstrate that the contrast observed by HIM is indeed associated with the contrast agent.

HI-TuP5 Fabrication of Carbon Nanomembranes by Helium Ion Beam Lithography, *X. Zhang, H. Vieker, A. Beyer, A. Götzhäuser*, Bielefeld University, Germany

A helium-ion microscope can be used as beam writing tool on electron beam photoresists, such as hydrogen silsesquioxane (HSQ). It has been demonstrated to have a high resolution, a high sensitivity and a low proximity effect.

Here we report the fabrication of carbon nanomembranes from aromatic self-assembled monolayers (SAMs) with a helium ion beam as direct writing tool. Cross-linking of SAMs is achieved by exposure with helium ions which results in the formation of mechanically stable carbon nanomembranes. The required doses for cross-linking with helium ions are approximately one order of magnitude lower than with electrons. The cross-linked SAMs were transferred to either silicon substrates with an oxide layer for optical characterization or transmission electron microscopy (TEM) grids for preparing free-standing carbon nanomembranes.

With helium ion based cross-linking we fabricated patterned nanomembranes as well. Furthermore, the proximity effect and the sample damage on the nano-scale pattern is investigated and discussed.

HI-TuP6 Layer Thickness Homogeneity Determination via Rutherford Backscattering in Helium-Ion Microscopy, *H. Vieker, K. Rott, A. Beyer, G. Reiss, A. Götzhäuser*, University of Bielefeld, 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 (RBS) ion contrast depends mainly on the elemental composition of the investigated sample surface. The escape depth of RBS ions is much larger than for secondary electrons. Thus whole layers with a wide range of thicknesses will contribute to a RBS ion image, whereas the SE image is far more surface sensitive, i.e. insensitive to buried parts under the sample surface.

In this contribution we examine RBS ion imaging as tool to characterize thickness variations of layered samples with well defined compositions. In a model example the homogeneity of a gold layer on a silicon substrate is investigated. The achievable spatial resolution for detecting buried inhomogeneities is analyzed. Furthermore we present examples with multiple layers.

HI-TuP7 Multi-Technique Approach to Study the Degradation Mechanism of used JLab Photocathode Samples, *V. Shutthanandan, Z. Zhu, M.I. Nandasiri, S.V.N.T. Kuchibhatla, S. Thevuthasan, W.P. Hess*, Pacific Northwest National Laboratory, *C. Hernandez-Garcia*, Jefferson Lab

Degradation of the photocathode materials in accelerator-based photoinjectors represents a challenge for sustained beam delivery in proposed fourth generation light sources. The quantum yield in most existing photocathodes degrades over time leading to machine downtime for quantum yield replenishing and in some instances to photocathode replacement. Several photocathode degradation processes have been proposed including ion back bombardment, photochemistry of surface adsorbed species and irradiation-induced surface and bulk defect formation. At present, no consensus exists within the user community as to the mechanisms of photocathode damage. Better understanding of degradation mechanisms of existing photocathode materials could lead to improved emission properties and longer operating lifetime. Existing photocathode materials range from metallic (e.g. copper) to semiconducting (e.g. GaAs) with various structures, dopants, and surface preparations. Photocathode emission requirements include high electron yield and low thermal emittance at high repetition rate. The goal of this work is to thoroughly

characterize the used photocathode samples obtained from Jefferson lab using helium ion microscope (HIM), Rutherford backscattering spectrometry (RBS) in channeling and random directions, secondary ion mass spectrometry (SIMS), atom probe tomography (APT) and atomic force microscopy (AFM) to understand the degradation mechanism. Four different GaAs samples (two control including one as prepared and the other as annealed but not used, and two used to delivered 1000 and 7000 Coulombs) were analyzed using these techniques. HIM images obtained at the damaged spot from the 7000 C sample clearly show that the surface at this spot is severely damaged. In addition, some cracks are clearly visible on the surface. HIM images collected at the tilt angle of 20° clearly show that these damage features are protruding above the surface of the photocathode samples at the center region of the spot. Stylus profilometer measurement on this spot reveals that the spot has peaks and valleys; the height of the main peak is around 7000 nm while the depth of the valleys ranges from 1000 to 3000 nm. It appears that the material in this area is melted. HIM images collected from all four samples clearly show that there is a systematic variation in the topography of the samples as a function of prolonged use of the photocathodes. The larger the usage time the smaller the structures are. Detailed analysis of these samples using RBS, SIMS together with HIM will be discussed.

Wednesday Morning, November 2, 2011

Helium Ion Microscopy Focus Topic

Room: 106 - Session HI+AS+BI+NS-WeM

Nano- and Bio- Imaging with Helium Ion Microscopy

Moderator: A. Götzhäuser, University of Bielefeld, Germany, V.S. Smentkowski, GE-GRC

8:40am **HI+AS+BI+NS-WeM3 Helium Ion Microscopy Techniques for Imaging and Characterization of nano-Device Materials and Structures**, S. Ogawa, T. Iijima, National Institute of Advanced Industrial Science and Technology (AIST), Japan **INVITED**

This paper presents several imaging modes for nano-devices fabrication that may make HIM a tool of particular value to soft materials such as low-k dielectrics (low-k) with less transformation and more materials contrast which reflects damaged areas, and copper interconnect buried in dielectrics, and shows luminescence induced by the focused helium ion beam using the HIM for the first time.

Imaging of ~100 nm pitch patterned low-k is important for LSI Cu/low-k interconnect processes, while SEM imaging results in changes to the low-k line edge roughness and shape by damage during an electron beam irradiation. The HIM could provide low-k dielectric secondary electron (SE) image with nm order resolution, deeper focus depth, less transformation because of three order magnitude lower thermal energy transfer into a unit volume of the low-k than the SEM under an appropriate operation condition¹⁾.

During the imaging, even at very low helium ion current, surfaces of samples were atomically etched off, as in a graphene patterning, and then blistering or physical etch occurred with the increase of the helium ion current. This makes the interpretation of the HIM SE imaging difficult but helpful. Damaged areas at side walls of the low-k regions in a 140 nm pitch interconnect were successfully seen with a different contrast from non-damaged low-k regions at an "optimized" helium ion beam condition²⁾, which was similar to a TEM/Valence EELS result. On the other hand, using the SEM, the damaged areas contrast in the low-k regions could not be imaged.

A new imaging mode, through the inter-level dielectric, of the underlying copper, was explored. Cu interconnect was seen through a 130 nm thick low-k dielectrics. The incident helium ions might generate secondary electrons (SEs) at the buried Cu surface and the SEs of 1-2 eV energy passed through the dielectric of a few eV band gap without any energy transfer, and then the image was obtained. Helium ion channeling at the Cu surface area varied the secondary electron quantity, and it might generate a crystal orientation contrast of the buried Cu metal.

Luminescence induced by the focused helium ion beam was studied using the HIM³⁾. Helium ion beam of a few pA current was irradiated to a SiO₂ film, and peaks in a spectrum were observed at around 281, 447, and 672 nm; these positions were different from those by a conventional SEM cathode luminescence. The further study will be presented.

L.Stern, W.Thompson and J.Notte of Carl Zeiss are acknowledged for their discussions in the Cu / low-k works.

1) S. Ogawa, et al, Jpn. J. Appl. Phys., 49 (2010) 04DB12, 2) S. Ogawa, et al, Proc. of 2011 IEEE IITC (2011)

9:20am **HI+AS+BI+NS-WeM5 He Ions Image the Au (111) Herringbone Reconstruction**, V. Veligura, G. Hlawacek, R. van Gastel, H. Zandvliet, B. Poelsema, MESA+ Institute for Nanotechnology, University of Twente, Enschede, The Netherlands

The herringbone reconstruction of the Au(111) surface was directly visualized using an Ultra High Vacuum Helium Ion Microscope. Ion channeling phenomena arise from the different atomic ordering in the outermost layer of the crystal. First, we investigated the channeling contrast from the bulk Au fcc structure by imaging polycrystalline Au on glass films. The contrast that was observed as a function of crystal orientation was found to conform to what is calculated from a simple hard sphere model. Consequently, the herringbone reconstruction was investigated. It is a periodic zigzag structure of the three different types of crystal stacking (fcc, hcp and bridge sites connecting these regions) and, ideally, has a period of 6.3 nm. The different stacking of the atoms that constitute the surface reconstruction leads to lateral variations of the secondary electron yield that can be resolved in HIM imagery. The existence of the herringbone reconstruction on the sample was independently confirmed through STM measurements and the quantitative details from both techniques are similar, but seem to be affected by the differences in vacuum

conditions. An influence of both the ion beam and vacuum environment on the visibility of the herringbone reconstruction is observed in our UHV-HIM system.

9:40am **HI+AS+BI+NS-WeM6 Imaging of Graphenoid Nanomembranes with Helium-Ion Microscopy**, A. Beyer, A. Turchanin, A. Götzhäuser, University of Bielefeld, Germany

Helium-ion microscopy is known for its high surface sensitivity. Here we present a study about imaging extremely thin nano-scale objects: graphenoid nanomembranes which consist exclusively of atoms near the surface. Such freestanding nanomembranes with a thickness of 1 nm are made from self-assembled monolayers (SAMs) by cross-linking and subsequent transfer to transmission electron microscopy (TEM) grids or other suitable substrates. We show that these nanomembranes exhibit a substantially higher contrast in helium-ion microscopes as compared to electron microscopes.

Cross-linking of SAMs is performed by large area exposures with electrons or photons which yield extended nanomembranes. On the other hand, patterned exposures allow the fabrication of nanosieves, i.e. perforated nanomembranes. Advantages in imaging such patterned cross-linked SAMs as well as freestanding nanosieves with the helium-ion microscope will be discussed.

10:40am **HI+AS+BI+NS-WeM9 Nanofabrication and Biological Imaging with the Helium Ion Microscope**, D.S. Pickard, National University of Singapore **INVITED**

The Helium Ion Microscope (HIM) is a new imaging technology based on a high brightness and stable Gas Field Ion Source (GFIS). The GFIS employed exhibits a low energy spread (<1 eV), small virtual source size (< 0.3 nm) and a high brightness > 4 x 10⁹ A/cm².sr [1]. This, in conjunction with the shallow escape depth (<1 nm) of the secondary electrons generated by the incident 30 keV helium ions, contribute to the HIM's primary advantage in the imaging of solid samples: its high spatial resolution (0.25 nm) [2]. We have applied this novel technology across a broad spectrum of multidisciplinary applications (from basic materials science and semiconductor applications to the biological sciences) to assess its utility and possible advantages over alternative techniques.

One area where our investigations have gained significant traction is in the imaging of biological specimens. The utility of this instrument in addressing topics of the biological sciences is due in part to the HIM's high spatial resolution. However, in the context of biological specimens, it is the ability to image non-conductive samples without the application of a metal (or other conductive) overcoat and without the need of a background gas (both of which degrade resolution and surface details), which has proven to be a distinguishing attribute. This opens up a whole new range of biological problems that can be solved rapidly and with less risk of artifacts.

An equally compelling application is in the field of nano-structuring. The focused helium ions have the ability to directly modify the sample surface under a high ion flux (via surface sputtering). This enables the direct patterning of structures and promises great utility in the fabrication of sub-10 nm devices. It also provides a mechanism for high resolution patterning on nonconventional substrates (such as suspended graphene membranes), where resist-based lithographic techniques are not feasible. Our experiences in sub-10 nm pattern transfer for both graphene and plasmonics applications will be presented.

1. B. Ward, J. Notte, and N. Economou, J. Vac. Sci. Technol. B, Vol. 24, No. 6, Nov/Dec 2006

2. Application Note, Carl Zeiss SMT, "Ultra-High Resolution Imaging in ORION®PLUS", PI No. 0220-2008-ENG, Nov. 21, 2008

11:20am **HI+AS+BI+NS-WeM11 Imaging and Characterizing Cellular Interaction of Nanoparticles using Helium Ion Microscopy**, B.W. Arey, V. Shutthanandan, Y. Xie, A. Tolic, G. Orr, Pacific Northwest National Laboratory

The helium ion microscope (HeIM) probes light elements (e.g. C, N, O, P) with high contrast due to the large variation in secondary electron yield, which minimizes the necessity of specimen staining. A defining characteristic of HIM is its remarkable capability to neutralize charge by the implementation of an electron flood gun, which eliminates the need for coating non-conductive specimens for imaging at high resolution. In addition, the small convergence angle in HeIM offers a large depth of field (~5x FE-SEM), enabling tall structures to be viewed in focus within a single image. Taking advantage of these capabilities, we investigate the interactions of engineered nanoparticles (NPs) at the surface of alveolar type II epithelial cells grown in culture. The increasing use of nanomaterials

in a wide range of commercial applications has the potential to increase human exposure to these materials, but the impact of such exposure on human health is still unclear. One of the main routes of exposure is the respiratory tract, where alveolar epithelial cells present a vulnerable target. Since the cellular interactions of NPs govern the cellular response and ultimately determine the impact on human health, our studies will help delineating relationships between particle properties and cellular interactions and response to better evaluate NP toxicity or biocompatibility.

The Rutherford backscattered ion (RBI) is a helium ions imaging mode, which backscatters helium ions from every element except hydrogen, with a backscatter yield that depends on the atomic number of the target. Energy-sensitive backscatter analysis is being developed, which when combined with RBI image information, support elemental identification at helium ion submicron resolution. This capability will enable distinguishing NPs from cell surface structures with nanometer resolution.

11:40am **HI+AS+BI+NS-WeM12 Application of Helium Ion Microscope on Semiconductor Surface Imaging and Metrology, H.X. Guo,**

National Institute for Materials Science, Japan, *H. Itoh*, National Institute of Advanced Industrial Science and Technology (AIST), Japan, *K. Onishi*, *T. Iwasaki*, *D. Fujita*, National Institute for Materials Science, Japan Scanning electron microscope (SEM) has been used in the semiconductor surface imaging and metrology for more than 50 years. Now, a new tool, Helium ion microscope (HeIM), is developed and applied to this work. SEM and HeIM are the same in some fundamental characteristics. But, the latter has advantages in smaller probe size, higher resolution, and greater depth of field. These abilities enhance the performance of the HeIM in the semiconductor surface imaging and metrology, such as imaging of low- k materials [1] and measurement of critical dimension of the semiconductor devices [2].

A standard sample for scanning probe microscope tip characterization [3, 4] was measured by using HeIM and atomic force microscope (AFM) as shown in Fig. 1 and Fig. 2. Line profile of the HeIM image in Fig. 1 shows high accuracy in edge definition of the sample. The contrast of the image is related to morphology and materials of the sample [5], the probe size of the Helium ion beam, direction of the sample and beam, charge distribution, and so on. All the aspects will be analyzed in our presentation. The AFM image of the sample shown in Fig. 2 is a dilation of the real surface topography of the sample due to the finite-size AFM tip [6]. With an erosion algorithm, the surface of the sample was reconstructed to be compared with HeIM measurement.

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[6] M. Xu, D. Fujita, and K. Onishi, *Rev. Sci. Instrum.*, 80, 043703 (2009)

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