

Wednesday Afternoon, October 31, 2012

Helium Ion Microscopy Focus Topic

Room: 19 - Session HI+AS+NS-WeA

Basics of Helium Ion Microscopy

Moderator: A. Gölzhäuser, University of Bielefeld, Germany

2:00pm **HI+AS+NS-WeA1 Basics of Imaging with Ions, D. Joy,** University of Tennessee **INVITED**

Imaging with a helium ion microscope (HIM) offers numerous advantages, both fundamental and practical, as compared to a conventional scanning electron microscope (SEM). At the same time, however, many aspects of ion microscopy seem very different to those found in the SEM. In this presentation therefore we will examine ;

Why ions are a better choice for imaging than electrons and which ion (or ions) might be the best

In which areas of performance and operation the ion beam image is 'better'

The optimum beam energy for ion imaging for different materials; how typical imaging conditions compare to those for the SEM; and why they are different

The types of signals that are available for imaging in the ion microscope and how they compare with their electron beam counterparts

The problems of specimen charging and beam damage with the ion beams

The options available for microanalysis with the HIM

2:40pm **HI+AS+NS-WeA3 Surface Analysis using Channeling Contrast in NUHV Helium Ion Microscopy, B. Poelsema,** University of Twente, Netherlands **INVITED**

Helium Ion Microscopy, HIM, is a novel high-performance technique to image surfaces and with its high resolution, great surface sensitivity, enhanced material contrast, ability to investigate insulating material and large depth of field, it provides a viable alternative to classical scanning electron microscopy. A number of applications require improved vacuum conditions to achieve ultimate performance. The sharply focused He ion beam is, compared to an electron beam in SEM set-ups, very efficient in decomposing, e.g., hydrocarbons present in the chamber and thus on the sample surface, which may obscure a clear view of the sample. Where this phenomenon is beneficial for high resolution structuring, it may well negate the benefits of small spot size and reduce the acquisition time available for spectroscopy in material analysis. To substantially reduce this problem a Near UHV version of the HIM has been developed in close collaboration between the manufacturer, Carl Zeiss NTS, LLC and our group at the University of Twente [1].

We will report on a number of recent observations with special attention for a new contrast mechanism, i.e. dechanneling of ions that extends the high surface sensitivity – usually achieved in secondary electron images – to backscattered ions. We demonstrate [2-4] how monolayer “thick” organic and inorganic films, as well as self assembled monolayers can be visualized, even when adsorbed on heavier substrates, by changes in the backscatter yield. Normally thin layers of a light element on a heavy substrate are “invisible” in backscattered ion yields. The results can be explained semi-quantitatively in terms of changes of the channelling probability. These results highlight the relevance of proper vacuum conditions for achieving monolayer sensitivity.

[1] R. van Gastel, L. Barriss, C. Sanford, G. Hlawacek, L. Scipioni, A.P. Merkle, D. Voci, C. Fenner, H.J.W. Zandvliet and B. Poelsema: *Microscopy and Microanalysis* **17(S2)**, 928-929 (2011)

[2] A. George, M. Knez, G. Hlawacek, D. Hagedoorn, H.H.J. Verputten, R. van Gastel and J.E. ten Elshof: *Langmuir* **28(5)**, 3045-3052 (2012)

[3] G. Hlawacek, V. Veligura, S. Lorbeck, T.F. Mocking, A. George, R. van Gastel, H.J.W. Zandvliet, B. Poelsema: submitted

[4] V. Veligura, G. Hlawacek, R. van Gastel, H.J.W. Zandvliet, B. Poelsema: submitted

Acknowledgments: Gregor Hlawacek, Vasilisa Veligura, Raoul van Gastel, Harold J.W. Zandvliet.

4:00pm **HI+AS+NS-WeA7 Evaluation of W(111) Gas Field Ion Sources Based on Single Atom Tips, R. Urban,** University of Alberta and The National Institute for Nanotechnology, Canada, *J.L. Pitters,* National Institute for Nanotechnology, NRC Canada, *R.A. Wolkow,* University of Alberta and The National Institute for Nanotechnology, Canada

Atomically defined tips gained significant attention over the past decade because they serve as high brightness electron and ion sources. The success of the Scanning Helium Ion Microscope is dependent on the development of an appropriate Gas Field Ion Source (GFIS) to generate the helium ion beam. Single atom tips (SATs) represent a unique subgroup of atomically defined tips where emission only occurs from a single atom at the tip apex. Small virtual source size makes these tips attractive candidates for advanced scanning imaging applications such as SEM, TEM, and scanning ion microscopy (SIM) as well as for non-staining ion beam writing applications.

In this study SATs were fabricated from single crystal W(111) wire using a gas and field assisted etching process. By carefully controlling etching parameters SATs with extraction voltages between 5 and 17 kV were formed for various tips. During tip formation, we also used neon as an imaging gas to evaluate a W(111) tip shape during nitrogen-assisted etching. The neon image allows for the observation of atomic structure not available while imaging with helium and helps to elucidate the atomic structure of the tip during and after the etching to a single atom. The field ion microscopy (FIM) patterns (intensity maps) from SAT were fitted with 2D Gaussian curve to evaluate ion beam divergence and amplitude. The divergence of helium beam with respect to helium pressure and applied voltage will be discussed for various SATs.

Angular current density of various SATs was evaluated from their FIM patterns recorded by a microchannel plate and ion current measurements using a Faraday cup. The volume under the 2D Gaussian surface was found to be directly proportional to total ion current carried by an ion beam. The ion current was found to be linearly proportional to He pressure. However, comparing various tips it was found that the ion current increased faster than the extraction voltage. This suggests improved He capture by a broader tip base. The effect of the shape of the base of the tip was also evaluated and it was found that the beam opening angle varied with the size of the tip base leading to a forward focussing effect. The relative angular current densities from SATs supported on different tip curvatures were also evaluated and found to increase at a faster rate than current, also indicating that a forward focusing effect was in effect. This indicates that SATs on large bases would prove optimal for ion current generation in a scanning ion microscope.

4:20pm **HI+AS+NS-WeA8 Single-atom Tip as an Emitter of Gas Field Ion Sources, I.-S. Hwang, H.-S. Kuo,** Academia Sinica, Taiwan, Republic of China, *T.-Y. Fu,* National Taiwan Normal University, Taiwan, Republic of China, *J.-L. Hou, C.-Y. Lin, Y.-H. Lu, W.-T. Chang, T.T. Tsong,* Academia Sinica, Taiwan, Republic of China

Thermally and chemically stable single-atom tips (SAT) or nanotips are highly desirable for emission of high-brightness gas field ion beams. In 2001, Fu et al. demonstrated a Pd-covered W(111) SAT through vacuum deposition of an ultra-thin Pd film on a clean W tip surface followed by thermal annealing [1]. Later, Kuo et al. further simplified the preparation process by replacing the tip cleaning and the vacuum deposition with electrochemical processes and successfully prepared several different types of noble metal-covered W(111) SATs [2]. This type of SATs is thermally stable and chemically inert, and thus can be regenerated through a gentle annealing if the apex is contaminated.

We have successfully generated hydrogen, helium, argon, and oxygen ion beams using a Ir/W SAT and characterized these ion sources [3]. The first two lightest ions provide the lowest sputtering rates, which is beneficial for scanning ion microscopy. The argon ion has a large mass and can provide a high sputtering rate, suitable for ion milling. Due to the high secondary ion yields, an oxygen ion beam may be applied to secondary ion mass spectrometry. The ion beam profiles indicate that the half opening angle is ~0.5°. This single spot indicates that emission occurs only from the topmost atom. This small source size and the small opening angle are particularly favorable for achieving high angular intensity, high brightness, and low spherical aberration, which are important characteristics for a focused ion beam system. The ion current of these gas ion beams are very stable and the tip does not show any degradation under fields above 5 V/Å after a total operation time of 80 hours. Since the SAT can be regenerated for more than 50 times, therefore its lifetime is long enough for most practical applications.

In addition, we have also prepared a pure iridium SAT based on oxygen-induced crystal faceting of the Ir(210) [4]. We have shown that the Ir-SAT

can be a good field ion emitter, capable of emitting a variety of gas ion beams, including He^+ , H_2^+ , N_2^+ , and O_2^+ , with high brightness and stability. In particular, nitrogen is a very corrosive gas for metal tips under strong positive electric fields. If we can achieve a stable emission of nitrogen ion, it also means many other gas field ion beams may also be emitted from this Ir-SAT. This may greatly broaden the application of focused ion beam technology.

- [1] T.-Y. Fu et al., Phys. Rev.B **64** (2001), 113401.
- [2] H.-S. Kuo et al., Nano Lett. **4** (2004), 2379.
- [3] H.-S. Kuo et al., Appl. Phys. Lett. **92** (2008), 063106.
- [4] H.-S. Kuo et al., Nanotechnology **20** (2009), 335701.

4:40pm **HI+AS+NS-WeA9 Helium Ions for Imaging and Nanofabrication on the nm Scale**, *E. Van Veldhoven, H.H.P.Th. Bekman, F.T. Molkenboer, N.B. Koster, D.J. Maas*, TNO Technical Sciences, The Netherlands

The Helium ion microscope (HIM, Zeiss Orion Plus™) has unique features. This microscope unravels a new application area for imaging sensitive and charging surfaces with (sub) nm resolution [1]. The beam-sample interaction generates secondary electrons with low energy and a low quantity of backscattering ions. These properties are very interesting for using the microscope not just for imaging only, but for nanofabrication too [2]. To explore all the capabilities for nanofabrication, the HIM is equipped with a pattern generator (Raith Elphy Multibeam™) and a gas injection system (Omniprobe Omnis™) to explore direct write, lithography and gas induced applications.

In this contribution we would like to focus on imaging charging materials and our latest results for using the HIM for developing new applications for mainly the Semiconductor Industry. We consider a few HIM-based methods for TEM sample preparation. It is possible to use the HIM for making a thin wedge without significant artifacts like bubble formation and amorphization. With the gas injection system we develop new recipes for very local deposition and etching. These recipes are used for feasibility studies for mask repair and circuit editing. The helium ion microscope offers a novel way for nanofabrication and imaging on the nm scale.

5:00pm **HI+AS+NS-WeA10 Towards Secondary Ion Mass Spectrometry on the Helium Ion Microscope**, *T. Wirtz, N. Vanhove, L. Pillatsch, D. Dowsett*, Centre de Recherche Public – Gabriel Lippmann, Luxembourg, *S. Sijbrandij, J. Notte*, Carl Zeiss

The ORION Helium Ion Microscope (HIM) has become a well-established tool for high-resolution microscopy [1]. The high brightness ALIS gas field ion source can operate with helium and, after special prototype modifications, with neon [2]. However, the detection of backscattered atoms can provide only limited specimen composition information. By contrast, Secondary Ion Mass Spectrometry (SIMS) is an extremely powerful technique for analyzing surfaces due to its excellent sensitivity, high dynamic range, very high mass resolution and ability to differentiate between isotopes. In order to get chemical information with a higher sensitivity and a high lateral resolution, we have investigated the feasibility of performing SIMS on the HIM.

Therefore, the secondary ion formation process under He^+ and Ne^+ bombardment has to be investigated and optimized. To investigate secondary ion formation an experimental study was performed; to investigate sputtering effects on resolution and practical implementation aspects a simulation approach was taken.

First, secondary ion yields for different elements sputtered from different materials exposed to helium and neon ion beams were experimentally determined on a test set-up. The basic yields could be increased by several orders of magnitude by using reactive gas flooding (i.e. O_2 and Cs^0 [3,4]). Afterwards, detection limits have been calculated taking into account the experimentally obtained useful yields. Depending on the dwell time, ppm sensitivity can be obtained for Ne^+ bombardment on silicon with oxygen flooding. Second, a detailed study of the sputtering phenomena using TRIM simulations was carried out in order to determine the effect of the collision cascade on the lateral resolution. The diameter (FW_{50}) of the area from which sputtered atoms originate has been determined for 10 keV He^+ and Ne^+ bombardment on different materials. While the obtained results are very encouraging, the practical instrumentation aspects have to be investigated as well in order to obtain a high secondary ion transmission and maintain the excellent primary beam characteristics. Therefore, the practical limitations imposed by adding an extraction system to the HIM have been studied in detail with respect to the extraction geometry.

In general, the combination of high-resolution microscopy and high-sensitivity chemical mapping on a single instrument will lead to a new level of correlative microscopy.

- [1] L. Scipioni et al., J. Vac. Sci. Technol. B **27**, 3250 (2009)

- [2] F. Rahman et al., Scanning **33**, 1 (2011)
- [3] K. Franzreb et al., Surf. Sci. **573**, 291 (2004)
- [4] P. Philipp et al., Int. J. Mass Spectrom. **253**, 71 (2006)

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