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## Advanced Ion Microscopy Focus Topic Room 203B - Session HI-ThA

### Emerging Ion Sources, Optics, and Applications

**Moderators:** John A. Notte, Carl Zeiss Microscopy, LLC, Shinichi Ogawa, National Institute of Advanced Industrial Science and Technology (AIST)

**2:20pm HI-ThA-1 Development of Gas Field Ionization Source using Gas with Low Ionization Energy that Enables Sample Processing and Observation, Shinichi Matsubara, H Shichi, T Hashizume, Hitachi, Japan**

INVITED

Practical use of a gas field ionization ion source (GFIS) has been tried for decades, but it was known difficult to realize. The GFIS has an extremely high brightness and a small source size, so that the convergence performance is excellent. As the atomic structure formation technology of the emitter tip has matured, the He - GFIS has been used in the real world scanning ion microscopes (SIMs). At first, their application was focused on the observation of the surface of samples because of its surface sensitivity, long focal depth, and a high resolution. Recently, however, Ne - GFIS has also been used for applications on fine direct fabrication which was difficult with a gallium liquid metal ion source.

We are developing GFISs which emit various kinds of ions and their own characteristics has been investigated. We are also developing ion-switching techniques which enable quick switching between fabrications and observations. With these techniques, we expect to create innovative applications difficult to realize with other technologies. In the previous studies we showed that an  $H_3^+$  ion is superior for observation with a low damage. Its energy dispersion is comparable with a  $He^+$  ion (0.5 eV) but the sputtering rate is expected to be smaller. We also showed that the  $H_3^+$  and  $Ne^+$  ions can be switched within 1 s by using a mixed gas of  $H_2$  and  $Ne$ , and by changing the emitter voltage. With this technique, we can instantly switch between fabrication and observation. Regarding the Ar-GFIS which is promising for fine fabrication, we showed that the effective fabrication rate given by the product of the current and the sputtering rate is highest among the  $Ne$ ,  $Ar$  and  $Kr$ . Furthermore, we have put into practical use of a photomask repairing technology using  $N_2$  - GFIS.

**3:00pm HI-ThA-3 Development of Scanning Helium Microscopy (SHeM), Susanne Schulze, D Ward, M Bergin, S Lambrick, B Allison, J Ellis, A Jardine, University of Cambridge, UK**

Some of the major insights in the development of modern materials have come from scanning probe, electron, and ion microscopy, with advances in resolution and sensitivity enabling new material science. While charged particle beam techniques are widely used they have the serious drawback of causing damage to sample surfaces, while scanning probe techniques are limited to relatively flat surfaces and suffer from limited scan speeds. Instead, we are pursuing a different approach, using neutral atom beams. Here we will report on recent advances and development of the scanning helium microscopy (SHeM) technique.

Since SHeM uses a neutral beam of helium atoms at very low energy (<100 meV), the technique is suitable for measuring a variety of samples including insulators, semiconductors, organic and biological species. It is particularly attractive as the approach does not require any complicated post processing techniques. We will report on recent studies on range of materials and potential new applications, including measurements performed in collaboration with colleagues at the University of Newcastle (Australia) [1,2]. A particular focus will be on describing the underlying mechanisms of contrast formation.

Many of the technological challenges associated with SHeM have now been addressed, including helium focusing, sample preparation and nanoscale manipulation, thus enabling preliminary instruments to be developed [1,2,3]. One of the remaining challenges is adequate detection of neutral atom beams, which is a particular problem due to helium's high ionization energy [3]. Applications that require time-sensitive measurements require a small ionization volume; however, when very high temporal resolution is not required, as with SHeM, very large ionizers with high detection efficiencies can be used. We will also report a recently developed detector, based on the approach recently applied to surface spin-echo experiments [4,5,6], and having the highest yet reported sensitivity for helium atoms.

[1] Nucl. Instr. Meth. Phys. Res B 340 76-80, 2014.

[2] Nature Communications, 7, 10189, 2016.

**3:20pm HI-ThA-4 Fabrication of Trimer/Single Atom Tip for GFIS by Field Evaporation without Tip Heating, Kwang-Il Kim, University of Science and Technology, Republic of Korea; Y Kim, T Ogawa, Korea Research Institute of Standards and Science (KRISS), Republic of Korea; S Choi, Kyungpook National University, Republic of Korea; B Cho, S Ahn, I Park, Korea Research Institute of Standards and Science (KRISS), Republic of Korea**

The application of the helium ion microscope (HIM) has expanded in various fields, such as nano-patterning, material science, and biology, due to its high spatial resolution for imaging and high-precision machining [1-3]. HIM realized sub-nm resolution with gas field ion sources (GFIS) which generate s ion beams from one or three topmost atoms of tips to obtain high beam current density. However, it is difficult to fabricate atomically sharp tips, such as trimer/single atom tip (TSAT), in an ultra-high vacuum (UHV) condition. TSAT can be typically fabricated by either a build-up method or field-assisted reactive gas etching method with oxygen and nitrogen [4-7]. However, these methods usually adopt resistive tip heating at about 1000 K as pre-cleaning of tip surface before the tip sharpening process. This heating leads to complex system because of a power supply circuit to provide or flow a current through a heating loop, where the tip was welded. In our study, we show that TSAT can be fabricated by field evaporation effect with an oxide layer which remains on the tip surface owing to the absence of tip heating.

As the result of this study, we could get a single crystalline field ion microscopy (FIM) image of W(111) with fabricating a TSAT by field evaporation phenomenon without tip surface cleaning by high temperature heating process. The oxide layer which remained after electrochemical etching process induces etch-like phenomenon in UHV condition without any additional gas injection. In order to analyze verify the proposed etching process, the analytical techniques of transmission electron microscope (TEM), energy filtered transmission electron microscope (EFTEM), and electron energy loss spectroscopy (EELS) were used. To compare the etching results whether the insulating layer present or not, we did additional experiment for tip heating. It was found that tungsten oxides contained in the insulating layer of the tip surface causes the etching. This method is much simpler than conventional methods because it uses only field evaporation phenomenon for fabricating TSAT. Therefore, we can simplify the equipment configuration since there is no need to heat the tip.

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[2] M.Postek et al., Scanning Microscopy 2009 (Vol. 7378, p. 737808)

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[4] M. Rezeq et al., The Journal of chemical physics, 124(20), 204716, 2006

[5] F.Rahman et al., Surface Science, 602(12), 2128-2134, 2008

[6] VT Binh et al., Surface Science, 202(1-2), L539-L549, 1988

[7] TY Fu et al., Physical Review B, 64(11), 113401, 2001

**4:00pm HI-ThA-6 Nano Aperture Ion Source, Greg Schwind, A Botman, S Kellogg, Thermal Fisher Scientific; L van Kouwen, P Kruit, Delft University of Technology, Netherlands**

INVITED

Among focused ion beam (FIB) users there is a constant demand for improved performance. Improved performance is defined as more beam current in the same spot size or same beam current in a smaller spot size. Another common driver is the desire for different ion species; specifically a non-reactive or inert species such as a noble gas. The ideal FIB system would be able to alternate between a heavy mass ion for efficient milling and a light mass ion for minimal sample damage imaging. Furthermore, it is desired to have a wide range of currents available for the different milling applications; high currents (100 nA) for high volume milling and low currents (1 pA) for nano-milling and currents in-between for precise small volume milling applications. The most versatile FIB systems are currently based on the Ga liquid metal ion source (LMIS) and hence are the most used. Specialized high volume plasma ion source based FIBs and gas field ion source (GFIS) FIBs for imaging are fulfilling specialized applications. To date, there has not been a good alternative specie ion source replacement for the Ga LMI.

# Thursday Afternoon, October 25, 2018

The Nano Aperture Ion Source (NAIS) is based on electron impact gas ionization inside a sub-micron gas chamber. In a realistic configuration, the simulations predict a source brightness in the range of  $1-3 \times 10^6$  A/m<sup>2</sup>sr V with a corresponding maximum energy spread of 1 eV. Recent efforts have been devoted to measuring the brightness and to understanding how the relevant physical process determine the ion beam performance. Processes investigated include the initial velocity and position distributions of the neutral gas particles, ionization cross sections, electron current density, and ion-neutral scattering<sup>1</sup>. The coulomb interactions and electric fields and their effect on beam performance was determined using analytical models, numerical calculations, and ray tracing. The particle interactions was found to limit the achievable brightness.

The brightness was measured using a small ion focusing column in combination with a knife edge ion transmission detector inside of a conventional SEM. A brightness of  $1 \times 10^5$  A/m<sup>2</sup>sr V was experimentally measured. The experimental value compares favorably to the calculation brightness value for the given experimental conditions, providing optimism that the theoretical B of  $1-3 \times 10^6$  will be achieved if NAIS is properly implemented. Limiting the measured brightness was in the test setup were poor electron beam current density and a weak accelerating field.

1. Leon van Kouwen, *The Nano Aperture Ion Source*, PhD Thesis TU Delft 2017

2. David S. Jun, Development of the Nano-Aperture Ion Source, PhD Tehsis Tu Delft (2014)

4:40pm **HI-ThA-8 Avoiding Amorphization Related Shape Changes of Nano-structures during Medium Fluence Ion Beam Irradiation of Semiconductor Materials**, *Xiaomo Xu*, G Hlawacek, H Engelmann, K Heinig, Helmholtz Zentrum Dresden-Rossendorf, Germany; *W Möller*, Helmholtz-Zentrum Dresden-Rossendorf, Germany; *A Gharbi*, CEA-LETI, France; *R Tiron*, CEA-LETI, MINATEC, France; *L Bischoff*, *T Prüfer*, *R Hübner*, *S Facsko*, *J von Borany*, Helmholtz Zentrum Dresden-Rossendorf, Germany

We present an approach to mitigate the ion beam induced damage inflicted on semiconductor nano-structures during ion beam irradiation.

Nanopillars (with diameter a of 35 nm and height of 70 nm) have been irradiated with either a 50 keV Si<sup>+</sup> broad beam from an implanter or a 25 keV focused Ne<sup>+</sup> beam from a helium ion microscope (HIM). Upon irradiation of the nanopillars at room temperature with a medium fluence ( $2 \times 10^{16}$  ions/cm<sup>2</sup>), strong plastic deformation has been observed which hinders further device integration. This differs from predictions made by the simulations using TRIDYN. However, irradiation at elevated temperatures with the same fluence would preserve the shape of the nanopillars.

It is well known that a critical temperature exists for silicon above which it will recrystallize during ion beam irradiation. This prevents the amorphization of the target material independent of the applied fluence. At high enough temperatures and not for too high flux this prevents the ion beam hammering and viscous flow of the nano-structures. These two effects are responsible for the shape change observed at low temperature. This has been observed previously mainly for swift heavy ions and energies higher than 100 keV. We used HIM and transmission electron microscopy to follow the morphological evolution of the pillars and their crystallinity. While irradiation at room temperature results in amorphization and the related destruction of the nanopillars, irradiation above 650 K preserves the crystalline nature of the pillars and prevents viscous flow. This effect has been observed previously mainly for swift heavy ions and energies higher than 100 keV. Such high-temperature irradiation, when carried out on a nanopillar with Si/SiO<sub>2</sub>/Si layer stack, would induce ion beam mixing without suffering from the plastic deformation of the nanostructure. Due to a limited mixing volume, single Si-NCs would form in a subsequent rapid thermal annealing process via Oswald ripening and serve as a basic structure of a gate-all-around single electron transistor device.

This work is supported by the European Union's H-2020 research project 'IONS4SET' under Grant Agreement No. 688072.

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