Thursday Morning, October 22, 2015

Helium Ion Microscopy Focus Topic Room: 211B - Session HI+AS+SS+NS-ThM

Focused Ion Beam Technology (08:00-

10:00)/Fundamentals of Helium Ion Microscopy (11:00-12:20)

Moderator: Gregor Hlawacek, Helmholtz-Zentrum Dresden - Rossendorf, Leonidas Ocola, Argonne National Laboratory

8:00am HI+AS+SS+NS-ThM1 Ga+ Ion Beam Nanofabrication Techniques of 3D Micro- and Nano- Fluidic Devices, *Leonidas Ocola*, Argonne National Laboratory

Three-dimensional (3D) fluidic geometries have been fabricated in the past by using several layers of Polydimethylsiloxane (PDMS) molds or doublesided Si etch steps [1], which require highly accurate chip bonding to complete the fluid path and multiple process steps. An alternative to this method is the use of direct write ion beam micromachining as a means to fabricate key components of a microfluidic device that require variations in depth as well as variations in width. 3-D microfabrication currently is mainly constrained to excimer lasers [2-3] and therefore is inherently diffraction limited. Grey scale lithography is also used for 3D structures but has limited capability. On the other hand, ion beam micromachining can scale down below the diffraction limit with no change in the technique and almost unlimited depth bandwidth. The focused ion beam / scanning electron microscope (FIB/SEM) is a powerful tool used for sample analysis and characterization. When equipped with a sophisticated pattern generator and lithography technology it can expand its use to new applications in nano- and micro-fabrication. Ion beam micromachining is akin to electron beam lithography, where a beam of charged particles are steered to draw structures contained in a computer aid design (CAD) file. Unlike electron beam lithography, one can program arbitrary depths by manipulating the dwell time, or dose, of a particular structure. In this paper the work reported previously [4-5] has been expanded to large and complex geometries to place emphasis on the applicability of ion beam micromachining to practical microfluidic applications, such as straight 3D mixers and serpentine 3D mixers with sections as deep as 70 microns and channel widths as large as 30 microns. We have found that these devices can achieve full mixing of aqueous solutions in about an order of magnitude faster than traditional devices. The challenges encountered and overcome to fabricate these mixers will be described and the scalability of different fabrication techniques to nano-fluidics will be revisited.

References

- 1. R. H. Liu et al., J. MEMS 9 (2000) 190
- 2. Y. Liao et al., Lab Chip, 12 (2012) 746
- 3. A. Ródenas et al., Proc. SPIE 8542 (2012) 854217
- 4. A. Imre et al., J. Vac. Sci. & Technol. B 28 (2010) 304
- 5. E. Palacios et al., J. Vac. Sci. Technol. B 28 (2010) C6I1

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8:20am HI+AS+SS+NS-ThM2 Adding 3D to Conventional SEM or FIB Surface Imaging Information - *In situ* Surface Sensing and Nanoprofilometry for Focused Electron and Ion Beam Induced Processes Verification, *Andre Linden*, Raith America, Inc., *A. Rudzinski*, *M. Levermann*, *T. Michael*, Raith GmbH, *E. Maynicke*, RWTH Aachen

Nanopatterning processes and corresponding parameters are typically well understood for standard nanofabrication applications using resist based electron beam lithography (EBL) or FIB milling processes (e.g. for TEM lamella preparation).

Recently however, the bandwidth of nanofabrication applications for dedicated nanopatterning tools has significantly broadened and is no more limited to resist based EBL and mere, standard FIB milling tasks. Some latest generation multi-technique electron and ion beam nanolithography tools even facilitate additional *in situ* processes such as resistless focused electron or ion beam induced processes - e.g. material deposition or gas enhanced etching. The number of variable parameters for such complex processes involving e.g. new gas chemistry or ion species is nearly "infinite". Moreover, smart and flexible patterning strategies, e.g. by using loops in conjunction with various multi-directional patterning modes, have significant impact on the final nanostructure's definition and performance, so that a straight *in situ* characterization of e.g. material deposition, milling

or etching rates becomes crucial for most efficient understanding and subsequent optimization of such processes.

In contrast to elaborately using additional analytical equipment outside the vacuum and subsequently re-introducing the sample for further processing and optimization, we have implemented a distance sensitive nanomanipulator with nanoprofilometric capabilities into our professional multi-technique nanofabrication tools, which allows *in situ* characterization of nanostructures in 3D with ~10nm resolution by collecting topographic sample surface information.

First results of direct *in situ* growth rate determination of focused electron beam induced material deposition (FEBID) for process calibration as well as 3D surface topographic information of challenging milling applications will be presented.

8:40am HI+AS+SS+NS-ThM3 Nanofabrication Using Gas-Assisted Focused Ion Beams, *Chad Rue*, FEI Company INVITED

A brief introduction to gas-assisted etching for Focused Ion Beams (FIBs) is given, including typical chemical precursors for various applications, and appropriate beam control parameters such as pixel overlap, dwell time, and refresh time. These factors are reasonably well-understood for pattern dimensions that are large compared to the size of the ion beam. However, for applications such as nanofabrication, which require high milling precision over small areas, the limiting size of the ion beam and its associated activated volume begin to influence the milling performance. The remainder of the discussion will focus on the relatively-unexplored regime in which the size of the pattern dimension is comparable to the size of the ion beam itself. The influence of various beam control parameters, particularly refresh time, becomes critically important to the milled profile of the desired structure. Redeposition effects, peripheral erosion, and mill rate trends are discussed. Operating tips and tricks are described, including the use of drift compensation strategies. The minimum physically achievable via size is examined. For a 10 pA Ga⁺ beam at 30 keV, used to mill a via in a SiO2 substrate with XeF2-assist, the minimum achievable via size (FWHM) is found to be 50 ± 10 nm, and is relatively independent of depth or aspect ratio. Implications for nanofabrication are discussed and examples are shown.

9:20am **HI+AS+SS+NS-ThM5** The Psychology and Applications of a **Bipolar Plasma Focused Ion Beam**, *Rod Boswell*, ANU, Australia, *N. Smith, P. Tesch, N. Martin*, Oregon Physics

A new high brightness ion source has been developed using bi-polar power supplies that can be used with either positive or negative ions. This has involved a redesign of the plasma source and the acceleration optics to allow high currents to be focused with an energy of up to 30kV. We expect to make significant advances in Ultra High Resolution SIMS with a negative oxygen beam; a second application is the milling of structures in glass with a O- beam, such as a microfluidic set of channels. At the higher voltages mentioned above, it should be possible to cut cross sections of Through Silicon Vias in glass substrates. The challenges encountered in creating and extracting the negative ions will be discussed along with some performance and application data.

9:40am HI+AS+SS+NS-ThM6 Advanced FIB Applications with New Ion Species and Large Area Capabilities, *Sven Bauerdick, L. Bruchhaus,* Raith GmbH, Germany, *J. Fridmann,* Raith America, Inc., *P. Mazarov, A. Nadzeyka, R. Jede,* Raith GmbH, Germany

Focused ion beam (FIB) systems are applied to a wide range of applications in R&D nanofabrication, both for creating functional devices as well as for preparing sample imaging and analysis. With different ion species on one hand and very sophisticated patterning approaches on the other hand it is possible to improve results and provide solutions for more advanced applications. Here we show and discuss the capabilities of Ga and new ion species like Au or Si with high resolution, long-term stability and easy handling, which is combined with an instrument design enabling large area or elongated patterns by write field stitching or truly continuous writing, respectively.

The type of ion defines the nature of the interaction mechanism with the sample and has significant consequences on the resulting nanostructures or samples. Therefore, we have extended the FIB technology towards the delivery of multiple ion species selectable into a nanometer-scale focused ion beam by employing a liquid metal alloy ion source (LMAIS). A mass separation filter is incorporated into the column to allow for fast and easy switching between different ions. The respective capabilities of mainly Ga, Au and Si have been investigated (resolution, milling rate, imaging, implantation) and according results and applications will be presented.

Moreover we investigated, optimized and tested milling approaches for pattern (write field) stitching and for truly continuous patterning based on precise stage movement while milling/ cutting with the ion beam. An improved beam pattern needs to mimic the looping strategy of conventional milling, so that grooves with defined depth, steep sidewalls and minimum re-deposition can be achieved. This combination of functionality enables applications like nanofabrication of micro-fluidic mixers, zone plates, large area gratings, or wafer-level nanopore devices as well as sample investigation e.g. imaging, X-sectioning and preparation in automated way. Examples for new nanofabrication techniques like large area hard masking by implantation, both for reducing and increasing the rate in standard etching processes, or seamless direct milling of nano-fluidic channels over cm's will be discussed.

11:00am HI+AS+SS+NS-ThM10 SIMS on the Helium Ion Microscope : a Powerful Tool for High-resolution High-sensitivity Nano-Analytics, Tom Wirtz, D. Dowsett, Luxembourg Institute of Science and Technology (LIST), Luxembourg, S. Sijbrandij, Carl Zeiss Microscopy INVITED While the ORION Helium Ion Microscope NanoFab has become an ideal high resolution imaging and nanofabrication tool, its analysis capability is currently limited. By contrast, Secondary Ion Mass Spectrometry (SIMS) is an extremely powerful technique for analysing surfaces owing in particular to its excellent sensitivity, high dynamic range, very high mass resolution and ability to differentiate between isotopes. The combination of He/Ne microscopy and SIMS would not just offer the prospect of obtaining SIMS information limited only by the size of the probe-sample interaction (~10 nm) but also of directly correlating such SIMS images with high resolution (0.5 nm) secondary electron images of the same zone taken at the same time. We have therefore investigated the feasibility of combining SIMS with Helium Ion Microscopy from a fundamental and instrumental point of view

In order to reach good detection limits when probing very small voxels in imaging applications, the ionization probability of the sputtered atoms and molecules needs to be maximized. When using He⁺ and Ne⁺ bombardment, the intrinsic yields are low compared to the ones found in conventional SIMS. However, the yields may be drastically increased by using reactive gas flooding during analysis, namely O₂ flooding for positive secondary ions and Cs flooding for negative secondary ions. Our results show that both negative and positive ion yields obtained with He⁺ and Ne⁺ bombardment may be increased by up to 4 orders of magnitude when using such reactive gas flooding. This optimization of secondary ion yields leads to detection limits varying from 10^{-3} to 10^{-6} for a lateral resolution between 10 nm and 100 nm.

The prototype instrument we developed during this feasibility study contains extraction optics allowing the emitted secondary ions to be extracted with a maximized efficiency and without negatively impacting the focusing of the incoming He⁺ or Ne⁺ ion beam (broadening or distortion of the ion beam due to the electric fields). These extraction optics are coupled to a specially designed compact high-performance magnetic sector double focusing mass spectrometer that we developed for the purpose of HIM-SIMS. The specifications of this mass spectrometer include high mass resolution with optimized transmission (M/ Δ M > 1000 at 100% transmission or M/ Δ M > 3000 at 50% transmission), full mass range (H-U) and parallel detection of several masses.

The results are very encouraging and the prospects of performing SIMS on the Helium Ion Microscope are very interesting. In this paper we will present the main findings of our feasibility study, including fundamental, instrumental and application aspects.

11:40am HI+AS+SS+NS-ThM12 Nanometer TOF-RBS and TOF-SIMS in a Helium/Neon Ion Microscope, *Nico Klingner, R. Heller, G. Hlawacek, S. Facsko, J. von Borany*, Helmholtz-Zentrum Dresden - Rossendorf, Germany

Helium ion microscopes (HIM) have become powerful imaging devices within the last decade. Their excellent lateral resolution down to 0.3 nm and their high field of depth make them a unique tool in surface imaging [1]. So far the the analytical capabilities of a HIM are rather limited or need complex detection setups. In addition we will discuss major challenges and physical limitations of ion beam analysis in the HIM.

We will present a new and relatively easy to implement method for ion beam analysis in the HIM by means of time of flight spectrometry to obtain elemental information from the sample. We will demonstrate the flexibility and applicability of the method to image samples with target mass contrast, to analyze the target compositions, and to measure depth profiles of films with few tens of nm thickness.

Pulsing the primary helium or neon ion beam and measuring the time of flight of ejected particles allows to obtain the energy of the backscattered particles as well as the mass of the ionized, sputtered target atoms. This has been achieved by chopping the primary ion beam down to pulse widths of 18 ns by use of the built-in beam blanker and a customized plug-on beam blanking electronics. The secondary particles are detected by means of a multi channel plate mounted on a flange of the HIM.

We will show TOF-RBS and TOF-SIMS measurements for different materials, which can give complementary information. Lateral resolved TOF-SIMS allows to quickly obtain qualitative elemental mapping while the TOF-RBS gives the standard-free quantitative sample composition of regions of interest. We will also show, that with TOF-RBS depth profiling of nm-thick layers is possible.

[1] G. Hlawacek, V. Veligura, R. van Gastel, and B. Poelsema, J. Vac. Sci. Technol. B 32(2), 2014

12:00pm HI+AS+SS+NS-ThM13 Improving Pattern Fidelity in Helium Ion Beam Lithography using Pixel Dose Optimization, N. Kalhor, TU Delft, Netherlands, W. Mulckhuyse, TNO Technical Sciences, Netherlands, Paul Alkemade, TU Delft, Netherlands, D. Maas, TNO Technical Sciences, Netherlands

Scanning Helium ion beam lithography (SHIBL) with a sub-nanometer beam probe size at the sample surface is a promising technology for highresolution lithography with high pattern density.¹ The advantages of SHIBL compared to e-beam lithography are higher sensitivity and a lower proximity effect. Remarkably, there are unique similarities in the activation response of resists to He-ions and extreme-ultraviolet (EUV) photons in EUV lithography (EUVL). Both primary beams produce low energy secondary electrons (SEs) and are not hindered by proximity effect. Recently Maas et al. experimentally demonstrated these similarities and suggested SHIBL as a promising method for pre-screening chemically amplified resists (CARs) prior to their final performance evaluation in an EUV scanner.²

However, unlike an EUV photon which only interacts with one resist molecule, an He-ion scatters inelastically in the resist and causes a chain of collisions with resist molecules, producing one or more SEs per collision. Also, a small dose-to-clear of 0.085 ions/nm^2 for SHIBL in a CAR was measured.² Hence, Maas et al. hinted at ion shot noise as a limiting factor in pattern fidelity in SHIBL.²

Here, we present a heuristic resist activation model for single-pixel dose SHIBL. The model employs a point-spread function (PSF) to account for all contributing factors in the resist activation. Ion shot noise impact is modeled with Poisson statistics. We show a good agreement between the model and our experimental single-pixel dose SHIBL results for line-andspace (LS) and contact hole patterns. Our model indicates pattern fidelity in sensitive CAR is not only limited by ion shot noise; instability of the He-ion source emission and post-exposure resist processing can also play important roles. Moreover, we introduce optimized-pixel-dose SHIBL to improve critical dimension uniformity (CDU), line width roughness (LWR), exposure latitude and throughput gain. In this approach, we calculate an optimum ion dose map for a given binary pattern such that the pattern's edges are exposed at the steepest part of the PSF to improve resist-pattern contrast and to minimize ion shot noise effect. Pixel dose optimization is advantageous to single-pixel exposure when the feature size is larger than the FWHM of the PSF. We discuss this by comparing our modeling results for single-pixel and optimized-pixel-dose SHIBL exposure modes for a desired LS pattern. We show that pixel-dose optimization could reduce LWR by ~45% (~1.3 nm) with a concurrent 20% dose reduction.

¹V. Sidorkin et al., J. Vac. Sci. Technol. B **27**, L18 (2009)

²Maas et al., SPIE Proc. **9048**, 90482Z (2014)

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