

Advanced Ion Microscopy and Ion Beam Nano-engineering Focus Topic

Room 301 - Session HI+AP-WeA

Emerging Ion Sources, Optics, & Applications

Moderators: Rosa Cordoba, University of Valencia, Spain, Florian Vollnhals, INAM, Germany

2:20pm HI+AP-WeA-1 Rationalizing and Controlling the Composition and Properties of Materials Deposited Using Charged Particles, **Howard Fairbrother**, Johns Hopkins University

Focused ion beam-induced deposition (FIBID) and focused electron beam-induced deposition (FEBID) are charged particle nanofabrication techniques able to directly fabricate 2D and 3D metal-containing nanostructures from organometallic precursors. These techniques provide for precise control over beam position for nanoscale pattern writing without the use of a mask or organic solvents and as such are more environmentally benign and less wasteful than traditional lithography techniques. However, one of the biggest issues with charged particle deposition techniques is the difficulty in controlling the composition of the deposits. Indeed, the creation of pure metal nanostructures in FEBID or FIBID is often a goal for achieving optimal materials properties, but is rarely realized in practice without the need for post deposition processing. In this presentation, and using Pt as an example, I will describe how UHV surface science studies can be used to aid in the design of precursors and selection of deposition conditions that favor the creation of pure metal deposits. Examples will include, (i) the electron beam induced deposition of Pt nanostructures from $\text{Pt}(\text{CO})_2\text{Cl}_2$, where surface science studies have identified the two sequential steps which can lead to Pt formation, notably the initial deposition event ($\text{Pt}(\text{CO})_2\text{Cl}_{2(\text{ads})} + e^- \rightarrow \text{PtCl}_{2(\text{ads})} + 2\text{CO}_{(\text{g})}$) as well as the subsequent removal of Cl via an ESD type process ($\text{PtCl}_{2(\text{ads})} + e^- \rightarrow \text{Pt}_{(\text{ads})} + \text{Cl}_{(\text{g})}^-$) and (ii) ion-beam induced deposition of Pt from MeCpPtMe_3 and $\text{Pt}(\text{CO})_2\text{X}_2$ precursors, where surface science studies have revealed the greater utility of effecting deposition with lighter ions (e.g. H_2^+ , He^+), due to their ability to access deposition conditions over which nearly pure Pt can be deposited as compared to heavier ions (e.g. Ar^+) where Pt sputtering rates preclude the formation of Pt. The microstructure and properties of these Pt deposits as well as the possibilities to use analogous approaches for the charged particle deposition of other metals (e.g. Au) will also be discussed.

3:00pm HI+AP-WeA-3 Ion Beam Induced Reactions and Deposition of $\text{Pt}(\text{CO})_2\text{Br}_2$ and $\text{Pt}(\text{CO})_2\text{Cl}_2$, **Mohammed Abdel-Rahman**, P. Eckhart, Johns Hopkins University; J. Yu, A. Chaudhary, L. McElwee-White, University of Florida; H. Fairbrother, Johns Hopkins University

Direct-write lithography techniques, such as focused ion beam induced deposition (FIBID), are an attractive alternative to traditional lithographic techniques. However, traditional precursors (Me_3PtCpMe , $\text{Pt}(\text{acac})_2$, for chemical vapor deposition perform poorly as FIBID precursors and result in carbon-contaminated metal deposits. To overcome this limitation, $\text{Pt}(\text{CO})_2\text{X}_2$ (X = Br, Cl) were chosen as alternative precursors because of its low carbon content and desirable volatility for FIBID. FIBID deposits were created from commercially available $\text{Pt}(\text{CO})_2\text{Cl}_2$ and lab synthesized $\text{Pt}(\text{CO})_2\text{Br}_2$, complemented by *in situ* UHV studies using XPS and MS to determine the sequence of ion-induced processes that underly the deposition mechanism. Deposition and UHV studies were conducted with Ar^+ , He^+ , and H_2^+ to determine the effect of ion properties (mass and reactivity) on the deposition process. Results obtained on thin films of precursors molecules deposited on inert substrates at temperatures < 200K under UHV conditions indicate that, regardless of the ion or precursor, the deposition mechanism proceeded via rapid and complete CO loss forming PtX_2 . Subsequent reactions are dominated by sputtering of the PtX_2 species with halogen loss proceeding quicker than Pt loss. Consequently, pure Pt deposits are formed beyond a critical ion dose. Although varying the ion does not alter the deposition mechanism, the relative reaction rates for the deposition and sputtering processes increase with ion size following the pattern $\text{Ar}^+ > \text{He}^+ > \text{H}_2^+$. This information can be used to select the steady-state conditions best suited to form pure Pt deposits in the absence of post-deposition purification steps.

3:20pm HI+AP-WeA-4 Next Generation Ion Beam Resists: Sub-10 nm Helium Ion Beam Lithography, **Scott Lewis**, G. Derose, California Institute of Technology

A new class of metal organic resist materials that is based on a heterometallic ring (Figure 1) has been demonstrated with helium ion
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beam lithography while demonstrating extraordinarily high etch selectivity for silicon of >5:1 (at 8 nm half pitch (HP)) when subjected to a pseudo-Bosch inductively coupled plasma-reactive-ion etch (ICP-RIE). The resist was designed using our latest Monte Carlo simulator (Figure 2a), which we developed because there are no simulators for ion beam lithography. Ion simulation packages such as SRIM/TRIM provide accurate data for ion penetration and propagation into a material for applications such as ion implantation, it fails to provide data on the creating a secondary electron (SE) and creating SE's cascade with further orders of SE's which are essential as they will have large effect on the sensitivity of the resist and will have a large contribution on the proximity effect. Exposing the resist to 35 keV helium ions, produced a nano pattern with a resolution of 8.5 nm HP (Figure 2b and c for a plan and tilted view), and obtained a low exposure dose of 22 pC/cm. This dose is 3 orders of magnitude lower than what is required with EBL when comparing to 100 and 30 keV electrons. To account for the low doses, this talk describes how the helium ion interacts with the resist and shows that the exposure mechanism occurs in two parts. Firstly, the helium ions are confined to the incident beam because of the resist properties of high molecular weight and low density limits the number of scattering sites that the ion encounters. Secondly, the helium ion interaction yields significantly more SE's per incident ions than electrons which had a significant contribution to the exposure efficiency. This is why that a dose lower by three orders of magnitude when compared to electron beam lithography. Through Monte Carlo simulation and X-ray photoelectron spectroscopy we will explain how the resist achieves the extraordinary high dry etch selectivity seen here (Figure 2d) where the 8.5 nm HP pattern was successfully transferred via the dry etching process into the silicon substrate. We will show through the use of Monte Carlo simulations how we were able to dramatically improve the resist by increasing its sensitivity and improving its LER while maintaining the same resolution and its high dry etch selectivity. The resist has demonstrated the flexibility to be exposed by ions, electrons and EUV, which makes it a versatile lithographic material with the potential for further customization to address a wide range of nanofabrication challenges.

4:20pm HI+AP-WeA-7 Novel Source Development for Focused Ion Beam Implantation and Irradiation, **Edward S. Bielejec**, M. Titze, A. Katzenmeyer, A. Belianinov, Sandia National Laboratories; Y. Wang, Los Alamos National Laboratory; B. Doyle, Sandia National Laboratories

INVITED
We will present on Sandia National Laboratory's Ion Beam Laboratory (IBL) development of novel sources for focused ion beam implantation and irradiation capabilities. The IBL operates seven focused ion beam (FIB) systems that range in ion energy from less than 1 keV to greater than 70 MeV, including a wide range of ion species from protons (H) to lead (Pb) over a range of spot sizes from nm to mm. In particular, we will concentrate on the development of liquid metal alloy ion sources (LMAIS) for our two mass filtered FIB systems, the A&D nanoplant and the Raith Velion, both of which include high spatial resolution with CAD based patterning to enable the formation of arbitrary patterned implantation.

We will present an overview of the available LMAIS for the mass filters systems as well as fabrication details and results using both novel Pb and N sources. The Pb source is based on a SnPb alloy using a custom Kovar wire tip in place of the standard tungsten tip usually used for FIB applications. This source has demonstrated a long lifetime comparable to our other alloy-based sources of greater than 2000 mA*hr and less than 50 nm resolution without optimization of the tip design. The atomic N source is based on an AuSn alloy implanted with nitrogen up to the saturation limit and verified using elastic recoil detection (ERD). This N source has demonstrated a relatively short lifetime of less than 100-200 mA*hr and is limited to a total N ion production rate of approximately 2,000 to 10,000 ions/s corresponding to up to approximately 1 fA of current of singly charged nitrogen ions. The demonstration of these sources works to continue the development of high resolution localized implantation and irradiation capabilities enabling the fabrication of custom implanted samples for cutting edge physics and quantum optics experiments.

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5:00pm **HI+AP-WeA-9 Focused Ion Beams from GaBiLi LMAIS for Nanofabrication and Ion Imaging**, *Torsten Richter, P. Mazarov, A. Nadzeyka, L. Bruchhaus, U. Mantz*, Raith GmbH, Germany

Focused Ion Beams (FIB) are broadly used in nanoscale science related applications, and they are inherently applied for direct nano-patterning, nanofabrication as well as for nano-analytics. FIB has become established as a direct, versatile, and precise fabrication method of smallest features at high fidelity. High demands are made on the ion beam that is used for direct FIB in terms of beam stability, patterning resolution and adjusting of the sputter yield. Liquid Metal Alloy Ion Source (LMAIS) is an emerging FIB source technology that provides a versatile solution to deliver various ion species from a single source for FIB nanofabrication to enhance resulting nanostructures [1]. However, beside nanofabrication FIB is utilized as a primary beam for SIMS analysis [2] and light ions such as Lithium in particular are well suited for sample imaging due to their low sputter yield and surface sensitive properties.

GaBiLi ion sources provide light and heavy ions from a single Liquid Metal Alloy Ion Source (LMAIS) fulfil requirements for both nanofabrication and nano-analytic [3]. Lithium, Gallium and Bismuth ions are emitted simultaneously, and ion species are separated subsequently in an ExB filter. Therefore rapid, easy, and reliable switching between light Lithium ions, and heavy Bismuth or Gallium ions enables not only novel nanofabrication processes but also satisfies analytical demands. GaBiLi allows 3D mill&image by imaging at highest lateral resolution by light Li ions and permits sample delayering at highest depth resolution with Bi ions or even Bi clusters [4]. SIMS takes advantage of optimizing either sputter yield or ionization yield of ejected ions.

In this contribution we present an overview of direct nanopatterning applications and related workflows such as 2-step bowtie nanofabrication with Lithium and Bismuth from GaBiLi (Figure 1) and ion beam imaging results that were obtained with Lithium ions (Figure 2).

[1] J. Gierak, P. Mazarov, L. Bruchhaus, R. Jede, L. Bischoff, Review of electrohydrodynamical ion sources and their applications to focused ion beam technology, *JVSTB* 36, 06J101 (2018).

[2] J. N. Audinot, P. Philipp, O. De Castro, A. Biesemeier, Q.H. Hoang, T. Wirtz, Highest resolution chemical imaging based on secondary ion mass spectrometry performed on the helium ion microscope. *Rep Prog Phys*. 2021 Sep 15;84 (10).

[3] N. Klingner, G. Hlawacek, P. Mazarov, W. Pilz, F. Meyer, and L. Bischoff, Imaging and milling resolution of light ion beams from helium ion microscopy and FIBs driven by liquid metal alloy ion sources, *Beilstein J. Nanotechnol.* 11, 1742 (2020).

[4] A. Tolstogousov, P. Mazarov, A.E. Ieshkin, S.F. Belykh, N.G. Korobeishchikov, V.O. Pelenovich, D.J. Fu, Sputtering of silicon by atomic and cluster bismuth ions: An influence of

projectile nuclearity and specific kinetic energy on the sputter yield *Vacuum* 188 110188 (2021).

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