Nanoscale Science and Technology
Room 114 - Session NS2-MoM

Imaging at the Nanoscale including Focused Ion Beam and Electron Microscopy

Moderator: Alec Talin, Sandia National Lab

10:30am **NS2-MoM-10 3D Reconstructions of Dislocation Networks via Focused Ion Beam Electron Channeling Contrast Imaging**, ***Julia Deitz***, *A. Polonsky, T. Ruggles, L. Jauregui, A. Allerman,* Sandia National Laboratories **INVITED**

Electron Channeling Contrast Imaging (ECCI) performed in a scanning electron microscope (SEM) serves as a rapid method for structural defect characterization in a wide array of crystalline materials such as metals and semiconductor materials/devices. Largely due to ease associated with sample preparation, much of this characterization has been performed in plan-view. The ability to instead perform ECCI cross-sectionally is advantageous for characterization of defects below 150 nm of the surface, sometimes at buried interfaces. Additionally, cross-sectional ECCI would demonstrate the potential to achieve three-dimensional (3D) analysis of dislocation networks, giving more detail on dislocation-dislocation interactions. With increases in serial sectioning implementations in the focused ion beam (FIB) SEM, automation routines are well positioned to obtain 3D dislocation network reconstructions. In this contribution, we demonstrate a 3D reconstruction of a dislocation network in an AlGaN via ECCI performed in the FIB-SEM and discuss practical challenges to collecting this data.

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11:00am **NS2-MoM-12 Transmission Electron Microscopy Investigation of Carbon Nanotube Growth on Stainless Steel Substrates**, ***Joshua Hancock***, *R. Vanfleet,* Brigham Young University

Carbon nanotubes (CNT) are a unique nanomaterial with a wide variety of research applications. One of the most common chemical vapor deposition (CVD) methods to grow CNTs is via the thermal decomposition of a hydrocarbon precursor gas by a metallic catalyst. This is usually done using an iron catalyst deposited on a "noble" or nonreactive substrate, such as alumina. In this setup, the foreign iron is treated to form nanoparticles on the substrate that act as the active sites for CNT growth. Stainless steel is a novel substrate because it does not require the addition of a foreign catalyst to grow carbon nanotubes. Instead, the substrate itself can be treated to become catalytic for CNT growth.

We investigate the effects of CNT growth on stainless steel and form a model for how nanotube growth occurs on the substrate. 316 stainless steel chips are first exposed to a flow of air at high temperature to oxidize the surface. The gas flow is then switched to ethylene to reduce the oxide layer and initialize CNT growth. The active temperature and exposure time of these steps are varied between samples to understand how each affects the growth and how the growth process evolves over time.

To better understand the growth mechanisms, we investigate the CNT-substrate interface at the base of the nanotubes. Focused ion beam (FIB) processing was used to create cross-sections of the CNTs and substrate. To protect the original material from ion beam damage and to contrast redeposited material, samples were coated in a thin layer of alumina using atomic layer deposition (ALD) prior to FIB processing. The final cross-sections were analyzed in a scanning transmission electron microscope (STEM) to allow for high resolution imaging and energy dispersive x-ray spectroscopy (EDX).

Results from STEM and EDX analysis have allowed us to form a basic model for CNT growth on stainless steel. CNTs were seen to grow out of iron-rich metallic nanoparticles embedded in the oxide layer. The oxide layer was also seen to lose iron over time, suggesting that these iron nanoparticles were reduced out of the oxide by the hydrocarbon precursor gas. The base particles were seen to sink into the oxide, leaving a hole when CNTs were removed. The diameters of the nanotubes were also seen to grow over time, suggesting that carbon infiltration is actively coating our CNTs. The effects of the sinking particle and infiltration quickly isolate the catalyst, leading to very short CNTs (1-10 microns).

11:15am **NS2-MoM-13 Focused Ion Beam Species Affect Beam Chemistry Applications**, ***Gavin Mitchson***, Thermo Fisher Scientific

Some modern plasma focused ion beam (PFIB) systems offer the capability to switch between multiple ion species. Each ion species (typically xenon, argon, oxygen, or nitrogen) can exhibit unique strengths and weaknesses for specific materials or applications. For example, oxygen often performs exceptionally well for cross sectioning resin-embedded samples and polymeric materials [1-3]. Low energy argon polishing can provide enhanced TEM image quality [4, 5]. However, signification portions of the parameter space remain unexplored and fundamental questions around limits of performance remain unanswered.

Beam chemistry is a critical component for many applications and to-date the effect of ion species in this space remains unexplored. For example, high quality protective caps are critical for most cross-sectioning and TEM lamella preparation processes. These protective caps are typically formed using at least one of a metallic precursor, a non-metallic carbonaceous precursor, or an insulating silicate-based precursor. In other situations, etchant precursors provide either selective material removal, enhanced material removal rates, or improved surface finish quality [6]. Since the initial PFIB system development and release, most workflows have relied on the Xe^+ ion species for beam chemistry processes. Critical knowledge around how other ion species perform with different beam chemistry precursors is missing.

We report the results of an extensive survey of different ion species and beam chemistry precursor combinations. All the ion species are capable of depositing various materials, although there are differences in the density of the deposited material and typical growth rates. Materials deposited using lighter ion species (argon, oxygen, and nitrogen) typically exhibit significant porosity and bubble formation when the ion beam energy exceeds some threshold, typically between 2-5 keV, which can obfuscate the relative yield comparisons (see Figure 1). For xenon, higher beam energies (below about 12 keV) are typically adequate for dense non-porous depositions. We also observed some interesting differences in etch rate as a function of halogenated etchant gas flow rate for argon and xenon ion species when milling silicon and aluminum substrates. For aluminum, the etch rate suppression or enhancement depends on the ion species, beam energy, and etchant precursor (Figure 2). We speculate that the different interaction volume sizes affect the relative kinetics of the possible gas-sample interaction pathways.

11:30am **NS2-MoM-14 Detection Efficiency Enhancement for Deterministic Single Ion Implantation**, ***Kristian Stockbridge***, Ionoptika, Ltd., UK; *D. Cox,* University of Surrey, UK; *G. Aresta,* Ionoptika, Ltd., UK; *R. Webb, S. Clowes, B. Murdin,* University of Surrey, UK

Techniques for deterministic implantation of single ions are currently of high interest for quantum tech-nology applications such as single photon emitters[1,2] and solid-state qubits[3]. Here we present our capabilities for single ion implantation over a range of ion species into different target materials at different implant energies (<100keV) using a liquid metal alloy ion source (LMAIS) Ionoptika Q-ONE single ion implanter.

For some systems their low secondary electron (SE) yield can limit our ability to efficiently detect single ion implantation, and this therefore limits the number of error-free deterministic implants we can ex-pect to achieve. We present on-chip ion beam induced charge (IBIC) detection for 25keV Bi+ and 50keV Bi2+ implantation into a Si device. The detection efficiency using IBIC is increased close to 100%. Alt-hough coincident SE detection was performed, the active substrate suppressed the emission of SEs such that the on-chip detection dominated.

SiO2 appears to be the target which gives consistently the best secondary electron detection efficiency. We therefore also investigate implantation through thin films of atomic layer deposited (ALD) SiO2 to enhance the detection efficiency of targets where on-chip detection may be incompatible.

[1] T. Herzig et al., Diamond for Quantum Applications Part 2, Semiconductors and Semimetals, Elsevir, 2021, Vol. 104, Chapter 1, pp 1-30

[2] K. Groot-Berning et al., 'Deterministic Single-Ion Implantation of Rare-Earth Ions for Nanometer-Resolution Color-Center Generation', Phys. Rev. Lett., vol. 123, p. 106802 (2019).

[3] M.T. Mądzik et al. 'Conditional quantum operation of two exchange-coupled single-donor spin qubits in a MOS-compatible silicon device', Nat Commun, vol. 12, p. 181 (2021).

11:45am **NS2-MoM-15 Large Area Grating Fabrication for Neutron Imaging**, ***Sarah Robinson**, R. Murphy,* National Institute of Standards and Technology (NIST)*; Y. Kim,* National Institute of Standards and Technology (NIST)/ University of Maryland, College Park*; J. LaManna, C. Wolf, K. Weigandt, D. Hussey, N. Klimov,* National Institute of Standards and Technology (NIST)

At NIST, we are developing a far-field neutron interferometer (INFER) to sample structural information from nanometer to micrometer length scales. INFER incorporates both absorbing source gratings and silicon phase gratings to improve interferometer visibility. This presentation will discuss the process development and fabrication to create high quality gratings with periods ranging from 700 nm to 3 mm to facilitate these measurements. We will discuss design considerations including optimizing stepper photolithography, minimizing field stitching errors and leveling issues during photoresist exposure, and characterizing highly uniform gratings over a full 100 mm diameter silicon wafer. Additionally, we plan to discuss how the quality of the gratings impacts the visibility of a moiré pattern at the detector and thus the resolution of this neutron imaging technique.

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