Thursday Morning, November 9, 2023

Advanced Focused Ion Beams Focus Topic
Room A107-109 - Session IB-ThM

Advances in FIB Instrumentation, Source, Optics, and Surface Analysis

Moderators: Alex Belianinov, Sandia National Laboratory, **Armin Goelzhaeuser**, Uni Bielefeld

8:00am **IB-ThM-1 TIBUSSII - the First Triple Beam Single Ion Implantation Setup for Quantum Applications***, **Nico Klingner**, G. Hlawacek, S. Facsko,* Institute of Ion Beam Physics and Materials Research, Helmholtz-Zentrum Dresden - Rossendorf (HZDR), Germany*; J. Silvent, A. Delobbe,* Orsay Physics, France **INVITED**

The ongoing miniaturization has reached a point where dopants, impurities or active impurities reach the quantum limit, making deterministic single ion implantation (SII) indispensable. Moreover, applications in quantum computing, spintronics, and magnonics require at the same time, a very precise spatial placement of these implants. Other requirements for such an implantation system would be a wide range of available ion species, the ability to implant at extremely low fluence as well as low voltage operation.

Our new system, named TIBUSSII, is expected to address all of these requirements. It will be the first UHV system to include a liquid metal alloy ion source (LMAIS) focused ion beam (FIB) column, a plasma FIB, and a scanning electron microscope (SEM). The 4-nm SEM will be used for damage-free navigation, orientation and inspection. Both FIB columns are mass-separated columns with three Einzel lenses, a chicane for neutral particles, and additional blankers and features optimized for single ion implantation.

We will show the current status of the system, which is currently being installed and further developed by HZDR and Orsay Physics. To verify the implantation of single ions, we are currently developing a secondary electron (SE) detection system with a sensitivity close to unity. It will be based on a semiconductor detector and is expected to surpass the detection efficiency of existing systems based on electron multiplication, such as channeltrons or microchannel plates.

8:40am **IB-ThM-3 A New Tool for Single Ion Implantation and Nanoscale Materials Engineering: System Design and Source Development***, **Gianfranco Aresta**, K. Stockbridge, K. McHardy, P. Blenkinsopp,* Ionoptika Ltd., UK

Quantum computing is the next great frontier of science. It has the potential to revolutionize many aspects of modern technology, including digital communications, “quantum-safe” cryptography, and incredibly accurate time measurements.

Single impurity atoms in semiconductors are receiving attention as potential quantum technologies, and proof-of-concept devices have shown promise. However, such devices are incredibly challenging to manufacture, as single atoms must be placed within ~ 20 nm of each other within a pure ^{28}Si matrix.

All working devices thus far have been fabricated using hydrogen lithography with an STM followed by atomic layer deposition. This is labour-intensive and requires several days of meticulous preparation to create just a single quantum bit (qubit). Real-world devices will require arrays of hundreds or thousands of impurity atoms, highlighting the requirement for a scalable method of positioning single atoms with nanometer precision.

We report on a new commercial instrument for the fabrication of quantum materials and devices via single ion implantation.

The instrument features a high-resolution mass-filtered focused ion beam (FIB), a high-sensitivity deterministic implantation system, 6-inch wafer handling, and a high-precision stage. The deterministic implantation system allows single ion implantation with confidence levels as high as 98%.

The ion dose delivered to the sample can be adjusted across a wide range, providing many nanoscale materials engineering capabilities in a single tool, from single ion implantation to direct-write capabilities such as isotopic enrichment and targeted ion-implantation of nanomaterials.

The liquid metal alloy ion sources, coupled with a mass filtered column will enable the implantation of many different elements with isotopic resolution. Available sources include silicon, erbium, gold, and bismuth, while many others of technological interest are in development. We will report on the LMIG source development carried out at Ionoptika in collaboration with our partners.

9:00am **IB-ThM-4 ToF-SIMS on a Plasma FIB: Dos and Dont’s***, **Jamie Ford**,* University of Pennsylvania

While time-of-flight secondary ion mass spectrometry (ToF-SIMS) is a well-established analytical technique on dedicated instrumentation, it is uncommon to perform it on a Xe plasma focused ion beam microscope. Introducing a new technique to a central user facility with a novice user base has provided valuable lessons along the way. In this talk I will share some of those lessons, best practices for introducing novel techniques to users along with setting reasonable expectations, how ToF-SIMS can complement more traditional FIB-SEM analytical techniques, and results from the wide variety of polymers, metals, and ceramics investigated in a central academic facility.

9:20am **IB-ThM-5 Multimodal Characterization of Biological Samples on FIB Instruments Combining Nano-Scale SIMS, SE and STIM Imaging Under Ambient or Cryogenic Conditions***, **Antje Biesemeier**, T. Taubitz, O. De Castro, J. Audinot, H. Hoang, P. Philipp,* Luxembourg Institute of Science and Technology (LIST), Luxembourg **INVITED**

Focused Ion Beam (FIB) based Secondary Ion Mass Spectrometry (SIMS) is a potent technique for imaging and chemical analysis at the ultrastructural level. In a plethora of life and materials science domains, it already proved excellent sensitivity, dynamic range and mass resolution. In this regard and together with leading manufacturers of FIB instrumentation, LIST is developing tailored and compact double focusing magnetic sector mass spectrometers for multimodal analysis at high spatial resolution (< 20 nm, [1]). The advantages over time-of-flight (TOF) systems include the ability of working in the DC mode (providing significantly higher SI counts for a given analysis duration) and higher overall transmission. Moreover, our magnetic sector system is equipped with a continuous focal plane detector, allowing parallel detection of all masses for each scanned sample pixel over the selected mass range. Depending on the FIB platform the SIMS is installed on, the users can correlate SIMS with secondary electron imaging (SE, [1-4]), back-scattered electron imaging (BSE, [2]), and/or scanning transmission ion microscopy-based analysis (STIM, [3, 4]) within the same instrument. The range of primary ions that have been used for magnetic sector FIB-SIMS include He^+, Li^+, Ne^+, Ga^+, Bi^+ and Cs^+.

As a close to native state sample preparation is mandatory for many biomedical research questions, we recently designed and built a new cryo FIB-SIMS platform that is based on an ultra-high brightness Gas Field Ion Source and that is equipped with a custom-made piezo-driven 5-axis cryo-stage along with sub-systems for cryo transfer and sample preparation (such as a specialised low humidity nitrogen atmosphere glovebox). The platform includes detection systems for SIMS, SE and STIM imaging by He^+/Ne^+, which combined with the cryo-capabilities result in an ideal tool for in-situ correlative studies on cryo lamella or other beam sensitive materials like battery materials [5].

Here, we will present an overview of our most recent developments and exemplary data sets from the field of nanotoxicology, demonstrating sub-cellular distribution and chemical identification of individual metal nanoparticles embedded in biological matrices after exposure.

[1] Rep. Prog. Phys. 84 (2021) p. 105901.

[2] Anal. Chem. 94, 30 (2022), p. 10754.

[3] Anal. Chem. 93, 43 (2021), p. 14417.

[4] J. Nanotechnol. 11 (2020), p. 1854.

[5] This work has received funding from the EU’s Horizon 2020 Research and Innovation Programme (grant agreement no. 720964) and was supported by the Luxembourg National Research Fund via the projects INTER/DFG/19/13992454 and FNR CORE C21/BM/15754743.

11:00am **IB-ThM-10 Mobile and Non-Mobile Hydrogen in Hydrogen-Charged Zirconium Alloy***, **Edward Gillman**,* Naval Nuclear Lab

Zirconium alloys are widely used for nuclear fuel cladding due to their mechanical behavior, resistance to corrosion, and low thermal neutron absorption cross-section. Over time, hydrogen is absorbed into zirconium alloys and exists as a solid solution so long as the concentration remains within the solubility limit for the alloy. Hydrogen migrates in the alloy and the migration depends on energy-dependent diffusional processes. When the concentration of hydrogen exceeds its solubility in zirconium alloy a non-mobile, brittle hydride phase can form. Embrittlement of zirconium alloy can result in reduced fracture toughness and in a phenomena known as Delayed Hydride Cracking (DHC). Hydrogen in solid solution is quite mobile within zirconium alloys at reactor operating temperatures requiring methods to predict hydrogen migration through the life of the zirconium alloy component. The dissolution and precipitation solvi for zirconium hydrides from zirconium in solid solution is an important aspect in hydrogen migration models. Here we use Time-of-Flight Secondary Ion Mass Spectroscopy (TOF-SIMS) to determine mobile and non-mobile hydrogen in hydrogen-charged zirconium alloys based on the hydrogen concentration in an uncharged commercial standard and LECO analysis for total hydrogen in hydrogen-charged zirconium alloy.

11:20am **IB-ThM-11 Visualization of the Pore Formation in Carbon Microspheres by Charge-compensated Helium Ion Microscopy***, **Natalie Frese**, M. Wortmann, M. Westphal,* Bielefeld University, Germany*; E. Diestelhorst, B. Brockhagen,* University of Applied Sciences and Arts, Germany*; K. Sattler,* University of Hawaii*; A. Gölzhäuser,* Bielefeld University, Germany

Hydrothermal carbonization of aqueous saccharide solutions yields carbonaceous microspheres, which can be post-carbonized by high-temperature pyrolysis to enhance their electrochemical properties for applications in energy storage devices. The pyrolysis leads to the formation of hierarchical porosity, i.e. micro-, meso- and macropores. The underlying mechanism of the pore formation was revealed for the first time using charge-compensated helium ion microscopy. It was shown that oxygen-rich nanoclusters gradually aggregate at the sphere surface, which then disintegrate in a narrow temperature range, leaving behind equally sized mesopores. The observed mechanism sheds light on the formation of hierarchically porous hard carbon materials more broadly.

11:40am **IB-ThM-12 3D Volume and Surface Imaging Applications using Focused Ion Beams from LMAIS***, **Alexander Ost**, A. Nadzeyka, L. Bruchhaus, T. Richter,* Raith GmbH, Germany

State-of-the-art Focused Ion Beam (FIB) technologies are in high demand nowadays as they allow not only to perform nanoscale patterning, but also ion imaging at high spatial resolution and analytical surface measurements with Secondary Ion Mass Spectrometry (SIMS). The liquid metal alloy ion source (LMAIS) technology, including the GaBiLi and AuGeSi sources, has been established for nanofabrication in the recent years [1]. Its excellent beam current stability, patterning and also imaging resolution [2], as well as fast adjusting of the sputtering yield with switching from one ion to the other within a few seconds allow a versatile use of this source technology.

Visualization of nanoscopic samples in 3D is of high interest in various domains, including nanotechnology, life and materials sciences, since it allows to study the surface and internal structure of the material compared to a simple 2D image. While a common method for 3D volume reconstruction consists of slicewise imaging and milling of the sample involving stage tilt, the new GaBiLi source paves the way for a new approach to obtain 3D volume information. The GaBiLi source has the advantage to alternately analyze the sample by imaging with Li^+ primary ions at high spatial resolution (down to 1.5 nm) in secondary electron (SE) mode and switching quickly to milling mode with Bi^+ primary ions at a high sputtering rate. Using this Mill&Image workflow the ion beam is always perpendicular to the sample surface and no sample tilt is needed. The set of SE images can be compiled into a 3D stack and cross-sectional views allow to visualize interior structures of the sample (Figure 1 a).

An alternative approach for 3D reconstruction, limiting surface sputtering and fully taking into account the surface topography, has been developed recently. Therefore, series of electron or ion microscopy images [3,4] are acquired around a region of interest (ROI). The images are implemented into a photogrammetry software used to obtain a 3D surface model (Figure 1 b) allowing detailed observation at all possible angles and magnifications, and even further numerical analysis [4].

In this contribution, we will demonstrate the capabilities of the Raith VELION FIB-SEM system equipped with GaBiLi/AuGeSi sources for 2D and 3D imaging workflows and give an outlook for combining in-situ 3D topographical information with analytical surface information from SIMS using these sources.

[1] L. Bischoff et al. Appl.Phys.Rev. 2016, 3(2),021101.

[2] N. Klingner et al. Beilstein J.Nanotechnol. 2020,11,1742–1749.

[3] F. Vollnhals, T. Wirtz. Anal.Chem. 2018, 90(20),11989–11995.

[4] A. D. Ost et al. ES&T 2021, 55(13),9384-9393.

12:00pm **IB-ThM-13 Application of Helium Ion Microscope in Site Specific Material Radiation Studies***, **Vaithiyalingam Shutthanandan**, S. Lambeets, A. Devaraj,* Pacific Northwest National Laboratory

Helium ion microscopy (HIM) enables not only the imaging of materials with Helium ions but also the irradiation of materials with a focused Helium beam (0.25 nm diameter beam spot) to achieve controlled displacement damage and Helium dosing. In the past, several different ODS steels, nanostructured ceramic materials, and nanolayered thin films have been investigated to understand the fundamental mechanism of radiation damage. In many of these investigations, high-energy He ion irradiations were carried out in a large area over the entire specimen, followed by the characterization of radiation damage. The spot size of ion irradiation beams from conventional sources was in the order of 100s of microns or larger, preventing site-specific irradiation damage investigation of individual microstructural features. In such cases, often the overall irradiation damage evolution in the material would be a cumulative response of the entire material microstructure (grain boundaries, interphase interfaces, second phase precipitates, nano-crystalline regions, and other preexisting defects) to the ion beam irradiation. A nanoscale site-specific He ion irradiation method can aid in decoupling and individually analyzing the He ion irradiation response of different microstructural features in a mutually exclusive manner. We have developed methods to use the helium ion microscope (HIM) to irradiate specific sites (i.e., near grain interiors vs. grain boundaries or near and on precipitates) of metallic materials using helium ions in a controlled manner and to characterize these materials in combination with focused ion beam scanning electron microscopy (FIB/SEM), TEM, XPS, and APT. In this talk, recent studies utilizing HIM as a radiation tool will be discussed in detail.

Advanced Focused Ion Beams Focus Topic
Room A107-109 - Session IB-ThA

In Situ FIB Applications

Moderators: Valerie Brogden, University of Oregon, **Shida Tan**, Intel Corporation

2:20pm **IB-ThA-1 Surface Near Helium Damage in Materials Studied with a High Throughput Implantation Method***, **Peter Hosemann,*** University of California at Berkeley, Lawrence Berkeley National Laboratory*; M. Baloocha, S. Stevenson, Y. Xie,* University of California at Berkeley **INVITED**

Helium damage in materials is of interest to the nuclear fusion, fission and spallation community. Helium generation in bulk material can cause embrittlements and swelling while Helium implantation in surface near areas can lead to blistering, fuzz formation and spalling. All phenomena listed are based on the accumulation of Helium into nanosized bubbles as a function of temperature and external stress states. Studying these phenomena traditionally requires ion beam accelerators and large samples. In this work we introduced nanobeam ion implantation methods which enable rapid multi dose ion beam implantation in surface near regions to enable basic scientific studies in single crystal and polycrystal materials such as Cu, Si, W,. The combination of Helium ion beam implantation using the Helium Ion Beam Microscope, Atomic Force Microscopy, Nanoindentation and Transmission Electron Microscope allows to bring insight into the formation of blisters, the linking up of Helium bubbles and the associated deformation and cracking mechanism. We were able to confirm previously posed hypothesis in tungsten blistering as well as show the dose threshold for silicon amorphization.

3:00pm **IB-ThA-3 Modes of Strain Accommodation in Cu-Nb Multilayered Thin Film on Indentation and Cyclic Shear***, **Mayur Pole**, A. Devaraj, T. Ajantiwalay, S. Tripathi, M. Olszta, T. Wang,* PNNL*; B. Gwalani,* North Carolina State University*; Z. Lu, H. Mehta,* PNNL

Two-phase layered thin films with a high density of semi-coherent interfaces exhibit excellent mechanical properties and thermal stability. In this study, a magnetron-sputtered Cu-Nb dual-layered thin film (~500nm for Cu and ~150nm for Nb) having an amorphous interface between Cu and Nb with a high density of aligned growth twins in Cu is subjected to severe surface deformation. The material is loaded using indentation and cyclic shear under tribological testing. The strain accommodation in the subsurface microstructure after deformation varies based on the local structure and deformation mode. Grain refinement and crack formations in the stressed region of the Nb layer and localized crystallization of the amorphous interface are observed after indentation and scratch testing. Pronounced detwinning of growth twins in the Cu layer under the cyclic shear strain leaves large dislocations sites and loops which are observed both by high-resolution transmission electron microscopy and experiment-guided molecular dynamic (MD) simulations. Our simulations provided insights into understanding the pathway for the detwinning process under cyclic shear loading.

3:20pm **IB-ThA-4 Investigating the Site-Specific Mechanical Properties of Advanced Aluminum Alloys via in-Situ Micromechanical Testing Inside the Plasma FIB***, **Tanvi Ajantiwalay**, A. Devaraj,* Pacific Northwest National Laboratory

Plasma focused ion beam (PFIB) has the potential to fabricate large damage-free specimens for various analytical applications. The use of heavier xenon (Xe) ions instead of conventional gallium (Ga) ions provide faster-milling rates and no ion-implantation. In this work, we demonstrate the utilization of PFIB to fabricate site-specific specimens for micromechanical testing of various aluminum (Al) alloys processed via advanced techniques such as friction stir, and additive manufacturing. Both these techniques modify the local microstructure of the base material to achieve grain refinement and hence optimum mechanical properties. A correlation between the microstructure and mechanical properties is thus established through in situ micromechanical testing inside the PFIB/SEM, which, is eventually beneficial to improve the process parameters and the overall performance of these alloys.

3:40pm **IB-ThA-5 Evolution of Stress Fields During Crack Growth and Arrest in Micro-Cantilevers During *in Situ* Bending***, **Michael Meindlhumer,** M. Alfreider,* Montanuniversität Leoben, Austria*; M. Burghammer, M. Rosenthal,* ESRF, The European Synchrotron, France*; R. Daniel, A. Hohenwarter, C. Mitterer, J. Todt, D. Kiener, J. Keckes,* Montanuniversität Leoben, Austria **INVITED**

In order to improve our understanding of the fracture behaviour in micro-cantilevers it is necessary to elucidate the multiaxial stress and strain fields throughout their irreversible deformation, especially in the regime where simplified homogeneous linear elastic assumptions are not valid anymore In this contribution, cross-sectional X-ray nanodiffraction (CSnanoXRD) with a resolution of 200nm was used for the detection of the multi-axial strain fields associated with crack growth during *in situ*stepwise deformation of (i) a notched clamped cantilever prepared from a multi-layered thin film composed of four alternating brittle CrN and semi-ductile Cr layers on high-speed steel and (ii) a freestanding cantilever fabricated from a nanocrystalline FeCrMnNiCo alloy. Both cantilevers were manufactured by focused ion beam milling. The Cr/CrN clamped cantilever was loaded stepwise to 150 and 460 mN and multi-axial stress distributions were retrieved in a region of interest of 40×30µm^2.

An effective negative stress intensity of −5.9±0.4MPa·m^{½} accompanied by a plastic zone around the notch tip arose in the notched Cr sublayer as a consequence of residual stress in the thin film. The *in situ* experiment indicated a strong influence of the residual stresses on the cross-sectional stress fields evolution and crack arrest capability at the CrN-Cr interface. In detail, crack growth in the notched Cr layer to the adjacent CrN-Cr interface occurred at a critical stress intensity of 2.8±0.5MPa·m^{½}.

The freestanding FeCrMnNiCo cantilever was loaded to 22, 45 and 34mN loads, which corresponds to conditions where elastic loading, crack tip blunting and void formation and coalescence with the crack front are the governing mechanisms, respectively. In that case, CSnanoXRD data were evaluated in a region of 30×35µm^2 centered around the crack tip. At a load of 22mN, a bending stress up to ±1 GPa was evaluated, while directly in front of the notch the crack opening stress raised to 4GPa. In a 200nm circular zone around the notch the measured stress distributions deviated evidently from the linear-elastic fracture mechanics assumptions. At 45mN, crack opening stresses increased to 4.5GPa and a 1µm wide distinct plastic zone formed. Further loading lead to a breakdown of the commonly assumed crack tip singularity and a significant decrease of the evaluated stress magnitude.

The quantitative experimental stress results provide unprecedented insights into the gradual stress evolution at the crack tip and across the cantilevers as well as associated fracture processes in nanocrystalline materials.

Advanced Focused Ion Beams Focus Topic
Room Oregon Ballroom 203-204 - Session IB-ThP
Advanced Focused Ion Beams Poster Session

IB-ThP-1 Emission of Multiple Ion Species from a Single Ion Source: Top-Down FIB with LMAIS on a Lithography Platform*, **Torsten Richter**, A. Ost, A. Nadzeyka, P. Mazarov, L. Bruchhaus, F. Meyer,* Raith GmbH, Germany

Focused Ion Beams (FIB) technologies are broadly used in nanoscale science related applications, and they are inherently applied for direct nano-patterning, resist based processes [1] as well as ion microscopy [2]. FIB patterning has become a direct, versatile, and precise fabrication method of smallest features at high reproducibility. Therefore, high demands are made on the FIB in terms of beam stability, but also the sample stage requires a high degree of stability, accuracy and automation for nanoscale patterning and imaging.

The Liquid Metal Alloy Ion Source (LMAIS) is a versatile FIB source technology able to emit various ion species [3] at high stability. Light and heavy ions such as Silicon and Gold or Lithium and Bismuth are emitted simultaneously from a single ion source (AuGeSi or GaBiLi) [4] and separated using a downstream Wien filter. This source technology allows the optimization of lateral resolution as well as depth resolution, sputter yield or avoiding sample contamination by selecting the most suitable ion species. Combining the LMAIS with a high-precision laser interferometer stage, the Raith VELION FIB-SEM offers new process pathways reaching from nm-sized feature to wafer-scale patterning.

Besides nanofabrication, novel 3D ion microscopy imaging workflows have become possible thanks to the top-down FIB geometry on the VELION, becoming thus a powerful ion microscope for sample 3D reconstruction. Milling with bismuth allows a fast and homogeneous surface sputtering at highest depth resolution, while switching to Lithium ions enables 2D imaging at high lateral resolution (down to 1.5 nm).

In this contribution, we present the latest advances in LMAIS source technology along with related applications such as resist based ion beam lithography and introduce 3D ion microscopy using both light and heavy ions from LMAIS.

[1] Lei Zhang et al., Nanotechnology 31 325301 (2020)

[2] N. Klingner et al., Beilstein J. Nanotechnology. 11, 1742 (2020)

[3] L. Bischoff et al., Appl. Phys. Rev. 3, 021101 (2016)

[4] W. Pilz et al., JVSTB 37, 021802 (2019)

IB-ThP-2 Roadmap for Focused Ion Beam Technologies*, K. Höflich,* Ferdinand Braun Institut, Germany*; G. Hobler,* TU Wien, Austria*; F. Allen,* University of California at Berkeley*; T. Wirtz,* LIST, Luxembourg*; G. Rius,* Institut de Microelectrònica de Barcelona, Spain*; **Gregor Hlawacek**,* Helmholtz Zentrum Dresden-Rossendorf, Germany

This roadmap document comprises a review of the current state-of-the-art of advanced focused ion beam (FIB) processing and technology followed by an outlook on required future developments curated by a diverse group of stakeholders.

FIBs play an important role in scientific research in fields ranging from health and biology to quantum technology and nuclear fusion research. However, usually FIBs are perceived as tools for the preparation of samples for other methods such as transmission electron microscopy or atom probe tomography. The intention of this document is to show that this is a clear underestimation of the method by showcasing current and past applications as well as providing a guideline for academia, industry and funding agencies on necessary future developments.

The roadmap starts with presenting the state-of-the-art of the FIB technology and instrumentation. The working principle of the FIB is described and an overview of additional instrumentation and detectors who widen the applicability of the method is given. In the second section the available instruments for the simulation and prediction of the focused ion implantation and milling process is given. This includes advanced simulation techniques such as DFT and MD but also computational efficient methods like BCA which can be used in the every day lab work by FIB users. The core part of the review describes the various applications which go beyond the preparation of TEM samples and include in addition to the above mentioned applications also the fields of spintronic and magnonics, super conductivity, photonics, micromechanics, MEMS/NEMS and many more.

In the last part the authors comprising the relevant stake holders give an overview of the required future development which will enable FIB technology to stay at the forefront of research in the discussed fields. This outlook part is partly based on a survey conducted within the European COST Action CA19140 FIT4NANO which unites more than 200 users, developers and manufacturers of FIB technology. The intention of this part is to act as a guideline for academic and commercial developers as well as funding agencies to steer the future developments in a direction agreed upon by the community.

It is this aim supported by the divers group of contributors to the review which makes this roadmap relevant and timely for many fields of research.

IB-ThP-3 A Multi-Scale Understanding of the Three-Dimensional Microstructure of the Cornea Using Oxygen Plasma Focused Ion Beam, Scanning Transmission Electron Microscopy and Micro-CT Techniques*, **Valerie Brogden**, M. Scanagatta-Long, H. Uehara, A. Lin,* University of Oregon **INVITED**

The cornea is a transparent tissue of the eye which is used to focus light and consists of multiple layers including the epithelium, stroma, and endothelium.In certain genetic conditions, the endothelium deteriorates, leading to loss of pumping function and the formation of excrescences of collagen known as guttata. This condition describes Fuchs' endothelial corneal dystrophy (FECD), an inheritable disease which causes corneal fluid accumulation and eventual clouded vision. Since human corneal endothelial cells do not proliferate in vivo, the only treatment for advanced FECD is corneal transplantation. A shortage of donor corneas necessitates a new therapy for FECD.

Currently, the structural nature of guttata formation is not well understood. With a better understanding of guttata structure, new therapeutic methods may be developed. In order to investigate the three-dimensional structure of the endothelial layer on multiple length scales, we utilized Plasma Focused Ion Beam Scanning Electron Microscopy (PFIB-SEM) tomography in conjunction with Scanning Transmission Electron Microscopy (STEM) and Microcomputed Tomography (μ-CT).

While xenon is the most commonly used PFIB species in materials sciences, oxygen is proving to be particularly useful for creating artifact-free cuts into biological tissue. Researchers have found that oxygen PFIB can be used to remove curtaining artifacts in organic samples significantly faster than xenon PFIB.

The above reasons make oxygen PFIB the ideal technique for understanding 3D volumes of biological structures where sub-micron resolution is necessary. However, due to the novelty of this technique, it has yet to be popularized. To demonstrate oxygen PFIB application to biological samples, a normal mouse cornea (C57BL6J) and an FECD mouse cornea (Col8a2^{Q455K}) were examined as test samples.

Upon investigation of the 3D renderings of PFIB SnV data, we noted that the diseased cornea appears more topographical than the healthy cornea and the nanostructure of the guttata can be observed. This suggests that oxygen PFIB SnV with 3D rendering is a powerful technique for understanding the microstructure of the corneal endothelium. Paired with μ-CT and STEM imaging, a correlative, three-dimensional, multiscale understanding of the cornea is possible.

IB-ThP-5 Focused Ion Beam Implantation by Deceleration*, M. Titze,* Sandia National Laboratory*; J. Poplawsky,* Oak Ridge Natinal Laboratory*; E. Bielejec,* Sandia National Laboratory*; **Alex Belianinov**,* Sandia National Laboratories

Ion implantation is a key capability for a growing number of scientific and industrial areas, including quantum information sciences, and the semiconductor industry. As devices become smaller, new materials and processes are introduced and quantum technologies transition to being mainstream, traditional implantation methods may fall short in terms of energy, species, and positional precision. In this talk we will show data demonstrating Au implants into Si at energies 10 eV– 450eV in a Raith Velion focused ion beam system by decelerating ions using bias and keeping the beam focused. The implants were validated using atom probe tomography. Our data reveal that standard implant modeling approaches fail to agree with experimentally measured depths, potentially due to surface sputtering and lattice enrichment. Finally, we discuss how our results pave a way to much lower implantation energies, while maintaining high spatial resolution.

Advanced Focused Ion Beams Focus Topic
Room B110-112 - Session IB1-FrM

Advances in FIB Specimen Preparation

Moderators: Tanvi Ajantiwalay, Pacific Northwest National Laboratory, **Gregor Hlawacek**, Helmholtz-Zentrum Dresden - Rossendorf

8:20am **IB1-FrM-1 An Air-Free Transfer Mechanism For FIB SEM**, ***Valerie Brogden**, J. Garman, S. Wiemholt, K. Langworthy,* University of Oregon **INVITED**

Lithium-ion batteries are increasingly prolific in the landscape of modern technology. Analysis of lithium metals with techniques such as Scanning Electron Microscopy (SEM) and Focused Ion Beam (FIB) can reveal critical information about the microstructure and composition of these materials. However, a problem presents itself when loading lithium metals into SEM/FIB systems. Lithium and other battery materials react with oxygen and can be quickly contaminated by exposure to atmosphere. Therefore, samples must be manipulated in an oxygen-free glove box. There is not currently a solution for mounting lithium samples onto a SEM/FIB stage for analysis without exposing the samples to atmosphere.

In this paper, we present a prototype for a glove box load lock that addresses this problem directly. This device bolts directly onto a ThermoFisher Plasma FIB and allows researchers to remove samples from positive-pressure packaging and mount the samples directly on the stage in an oxygen-free environment, and then pump the tool down to vacuum without contamination by atmosphere.

Advanced Focused Ion Beams Focus Topic
Room B110-112 - Session IB2-FrM

Advances in TEM and APT Specimen Preparation

Moderators: Tanvi Ajantiwalay, Pacific Northwest National Laboratory, **Gregor Hlawacek**, Helmholtz-Zentrum Dresden - Rossendorf

9:00am **IB2-FrM-3 Correlative and In Situ TEM/APT Technique Reveals Insights into Early Oxide Film Formation in a High Entropy Alloy**, ***Bharat Gwalani**,* Engineering Bldg I 911 Partners Way **INVITED**

Oxide film formation is a critical process that occurs on the surface of materials when they come into contact with oxygen. In some cases, oxide films can help protect the material from further corrosion, but in other cases, they can lead to degradation of the material over time. Understanding the early stages of oxide film formation is therefore important for designing materials that are resistant to corrosion. This study employed a combination of correlative and in situ techniques, specifically transmission electron microscopy (TEM) and atom probe tomography (APT), to investigate the initial stages of oxide film formation in a high entropy alloy (HEA). The research aimed to gain insight into the mechanisms of oxide film formation and the chemical changes that occur during the process. By analyzing the nano-scale chemical changes of the oxide film and the underlying HEA as a function of time, we observed the phase transformation pathway resulting from a single-layer to a multi-layer oxide film, which provided important insights into the process of oxide film formation. The study demonstrates the potential of these techniques in providing a deeper understanding of the processes involved in oxide film formation, enabling the design of materials with improved corrosion resistance and durability.

9:40am **IB2-FrM-5 Applications of Advanced Focused Ion Beam System to Energy Storage Materials**, ***Yaobin Xu**, X. Cao, W. Xu, J. Zhang, C. Wang,* Pacific Northwest National Laboratory

Battery technology has received considerable attention in recent decades due to the rapid growth of the electric vehicle and consumer electronics markets. An in-depth understanding of the electrode, interfacial structure, and chemical distribution of battery materials is essential to further advance battery technology. Focused ion beam-scanning electron microscope (FIB-SEM) is an analytical method combines ion beam for materials processing and electron beam for imaging. It enables both two-dimensional (2D) and three-dimensional (3D) imaging capability, combined with electron backscatter diffraction (EBSD) and energy dispersive X-ray spectrometry (EDS) detector, could also provide multimodal information collection, which is an effective and powerful analytical approach for battery structural analysis.

Lately, the plasma FIB-SEM (PFIB-SEM) technology has been developed with different ion source and higher ablation efficiency. It promises great potential for battery materials characterization, due to accessing representative 2D area and 3D volume via much faster (compared with traditional Ga^+ system) milling rate as well as enabling Ga^+ free sample preparation on advanced battery system through non-reactive ion source (Xe^+ and Ar^+ ion). Besides, for air and beam sensitive battery materials, like alkali metal electrode, sulfide based solid state electrolyte, combing cryogenic stage and inert gas transfer system, we could image and process them without air contamination during sample transfer and reduce ion/beam damage under cryogenic condition. In this presentation, cryogenic PFIB-SEM has been used to perform 2D microstructure and chemical analysis and 3D imaging/chemical analysis on different battery systems including Li metal anode, lithium nickel manganese cobalt oxide (NMC) cathode and solid-state electrolyte.

The successful demonstration of the cryogenic PFIB-SEM provides exciting possibilities for the investigation of both current and future generations of advanced battery technologies, and will be an important analytical approach for battery research and development.

10:00am **IB2-FrM-6 The Fabrication of Ruthenium single crystal specimen with Focused Ion Beam and Field Ion Microscopy for Atom Probe Tomography**, ***Mark G Wirth**, D. Perea, S. Lambeets,* Pacific Northwest National Laboratory

Metallic surfaces may undergo a series of surface and subsurface structural and chemical transformations while exposed to reactive gases that inevitably change the surface properties. Understanding such surface dynamics from a fundamental science point of view is an important requirement to build rational links between chemical/structural surface properties and design new catalysts with desired performance or new materials with enhanced resistance to corrosion. This is the case for Ruthenium (Ru), a relatively scarce precious material found in various large-scale applications such as chlorine, nitrates, and acetic acid production. Additionally, Ru shows promising properties for low temperature Haber-Bosch process to produce ammonium.

The imaging techniques of Field Ion Microscopy (FIM) and Atom Probe Tomography (APT), when combined, provide unique atomically resolved surface structure and compositional maps used to understand metal oxidation dynamics. FIM enables correlative atomic to nanoscale imaging of the surface of a very sharp metal needle revealing a complex network of crystallographic facets, the nanoscale apex size and hemispherical like shape of which models that of a metal nanoparticle. While APT and Operando Atom Probe (OAP) is used to track the chemical interaction between the complex surface with reactive gases such as O_2 or N_2 through composition mapping.

Compared to other metals, Ru specimen preparation is relatively challenging due to fragility related to its hexagonal close packed (HCP) crystal structure. This makes Ru particularly sensitive to mechanical stresses during FIM and APT experiments. Additionally, Ga implantation from a FIB-based needle specimen preparation approach can lead to the formation surface defects, complicating the indexing of the atomically resolved surface structure. In this work we will focus on the Ru specimen fabrication by FIB and the surface imaging by FIM with the indexing of Miller indices mapped across various surface features.

Advanced Focused Ion Beams Focus Topic
Room B110-112 - Session IB3-FrM

Beam Induced Defect and Material Engineering

Moderators: Tanvi Ajantiwalay, Pacific Northwest National Laboratory, **Gregor Hlawacek**, Helmholtz-Zentrum Dresden - Rossendorf

10:40am **IB3-FrM-8 Modeling and Experimental Demonstrations of Ion-Solid-Gas and Photon Beam Interactions During Nanoscale Synthesis**, ***Philip Rack**,* University of Tennessee, United States Minor Outlying Islands (the) **INVITED**

We study focused ion beam (FIB) processing using the Monte Carlo based simulation code EnvizION. This code simulates ion-substrate interactions and evolves the substrate to elucidate nanoscale milling and substrate damage. Additionally, we extended the simulation to include ion beam etching with a reactive precursor. The use of precursor gas for etching can greatly enhance material removal, allowing for enhanced etching at a significantly lower dose, and consequently, with much less associated damage to the bulk material. At the same time, the achievable resolution of the etched valley is impacted by a number of factors, such as the competition between chemical etching and physical sputtering, and the impact of the etching shape on gas coverage, and platform level patterning artifacts. EnvizION has been designed as a purpose built FIB simulation code, accounting for changes in the substrate configuration due to physical sputtering, as well as the dynamics of the precursor gas, while maintaining sufficient speed to practically simulate ion doses on the order of millions of ions. We study the etching of SiO2 using a XeF2 precursor gas. Etching simulations are validated against experimental data, and are used to identify how gas flux, beam current distribution, and platform level artifacts combine to determine achievable nanoscale resolution. Etching is studied for both Ne+and Ga+ion beam species, and the effective beam current distribution of each, corresponding to the experimental data, is identified. We additionally study the effect of the competition between etching gas flux and the beam dwell time, and in order to optimize resolution, we identify an intermediate gas flux for which etching resolution is the lowest.

In this presentation we will overview the EvnizION Monte Carlo simulation details including the ion-solid-precursor interactions and various sub-routines including secondary electron generation, surface energy minimization, vacancy-interstitial recombination, subsurface damage accumulation, and various precursor dynamics including adsorption, desorption, surface diffusion, secondary electron induced dissociation and ion-solid energy transfer induced dissociation. For each we will highlight the impact that the physical/chemical property effects the evolving nanoscale resolution of ion beam induced sputtering/chemical etching.

11:20am **IB3-FrM-10 Displacement Damage and Total Ionizing Dose Response of Ga_2O_3 MOSFETs***, **Michael Titze**,* Sandia National Laboratories

Gallium Oxide (here β-Ga_2O_3) is a promising material for compact high-voltage electronics due to its high breakdown voltage compared to Si. Introduction of Ga_2O_{-3} devices in radiation environments however requires prior knowledge of their response to various types of radiation, most notably displacement damage (DD) from neutrons in reactor environments, and total ionizing dose (TID) as experienced in x-ray / gamma environments. Device testing in appropriate environments is typically expensive and requires very long exposures due to the small interaction cross section of neutrons / gammas. Instead, we use a dual-beam focused ion beam and scanning electron microscope system (FIB-SEM) to probe radiation effects through surrogate environments. The FIB is used to generate predominantly DD with a small amount of TID while the SEM generates exclusively TID.

We use SRIM and PENELOPE simulations to determine the amount of DD and TID for each beam and observe the drive current and threshold voltage shift in DD and TID environments. We find that DD degrades the drive current but does not alter the threshold voltage. TID does not alter the drive current but changes the device threshold voltage.

This work was performed, in part, at the Center for Integrated Nanotechnologies, an Office of Science User Facility operated for the U.S. Department of Energy (DOE) Office of Science. Sandia National Laboratories is a multimission laboratory managed and operated by National Technology & Engineering Solutions of Sandia, LLC, a wholly owned subsidiary of Honeywell International, Inc., for the U.S. DOE's National Nuclear Security Administration under contract DE-NA-0003525. The views expressed in the article do not necessarily represent the views of the U.S. DOE or the United States Government.

11:40am **IB3-FrM-11 Helium Ion Microscopy for Morphological Analysis of Thrombi Extracted via Thrombectomy for Acute Stroke***, **Michael Westphal**, N. Frese,* University Bielefeld, Germany*; C. Sommer,* Institut für Neuropathologie, Universitätsklinik Mainz, Germany*; A. Kitsiou, W. Schäbitz,* Universitätsklinik für Neurologie, Evangelisches Klinikum Bethel gGmbH, Universitätsklinikum OWL, Germany*; A. Beyer, A. Gölzhäuser,* University Bielefeld, Germany

Strokes are one of the leading causes of death in the aging Western society. Especially in elderly patients, strokes are frequently recurring events. An essential component of stroke management after acute therapy is to diagnose the cause of the stroke forsecondary prevention. More than 50 thrombi extracted via thrombectomy were examined by chargecompensated helium ion microscopy to investigate possible correlations between their morphology and the origin.

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