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.

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