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fit4nano Workshop 2023

17 - 19 July, Lisbon
Faculty of Pharmacy, UL

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Thematic issue of the Beilstein Journal of Nanotechnology: Focused ion and electron beams for synthesis and characterization of nanomaterials

A special issue of the Beilstein Journal of Nanotechnology will be accepting submissions related to the workshop topics at https://www.beilstein-journals.org/bjnano/series/103. The Beilstein Journal is a platinum open source journal with no subscription or publishing fee. All submissions will undergo rigorous peer review by independent reviewers.

In particular, the issue is focused on the following aspects:

1. 1D, 2D and 3D additive manufacturing (nanoprinting) of functional nanostructures for superconducting, magnetic and plasmonic applications.
2. Spatially resolved removal or modification of materials using gas-assisted electron/ion beam lithography.
3. Focused ion beam (FIB) milling and development of new ion beam sources and gas precursors.
4. Characterization methods using focused charged particle beams with techniques such as cryo-electron microscopy, helium ion microscopy, and secondary ion mass spectroscopy.
5. Development of novel instrumentation for current and future research on applications of multiple ion species plasma FIB, effective structural modification of nanomaterials using laser FIB, and FIB–SEM tomography for advanced microstructural analysis.
6. Theoretical aspects of electron and ion interactions and surface kinetics processes.

Submission deadline: August 31, 2023

Workshop Program

Monday, 17 July

9:00-10:00	G. Hlawacek	FIT4NANO Core group meeting (on invitation only)
10:00-12:00	G. Hlawacek	FIT4NANO MC meeting (on invitation only)
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13:00-13:20		Welcome
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14:00-14:20	Djouher Bedrane	Coaxial Ion Source: pressure dependence of gas flow and field ion emission Page 15
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14:40-15:00	Yang Li	Study of Focused Rb Ion Beam Applications Page 18
15:00-15:20	Kaih Mitchell	Focused Ion Beam Milling with Cold Rubidium Page 20
15:20-16:00		Coffee Break
16:00-16:40	Karen Kavanagh	Negative Ion HIM Page 21
16:40-17:00	Paul Räcke	Focused Beams of Highly Charged Ions for Quantum Technologies Page 23
17:00-17:20	Katarzyna Berent	Comparison of Ga+ FIB and Plasma FIB systems used for microstructural characterization Page 24
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12:40-14:00		Break
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Legend

Legend
Invited Speaker
Industry talk

Poster Contributions

Oral Presentations

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OC1: Ionic liquids: from space thrusters to focused ion beams

C. S. Perez-Martinez

London Centre for Nanotechnology, University College London, WC1H 0AH, London, United Kingdom
E-Mail: carla.perezmartinez@ucl.ac.uk

Ionic Liquid Ion Sources (ILIS) are devices with applications ranging from space propulsion to nanomanufacturing. Ionic liquids are room temperature molten salts, or mixtures of cations or anions that are liquid at room temperature without need for a solvent. The cations are usually large organic molecules, while the anions may be complex organic or simple inorganic ions. An example, 1-ethyl-3-methylimid azolium tetrafluoroborate, EMI-BF4, is shown in Figure 1(a).
In ILIS, a micro-tip emitter is covered with ionic liquid and biased to a high voltage with respect to a downstream metallic extractor, see Figure 1(b). The electric field causes the liquid to deform into a sharp meniscus. At the apex of the meniscus, the electric field is high enough to trigger evaporation of ions from the liquid. The resulting beam can be used to propel spacecraft, from nanosatellites on near Earth orbits to deep space missions, or to treat materials.
ILIS could be advantageous in materials processing: the large variety of ionic liquids available gives ample choice of chemistries tailored to different purposes. For example, irradiation of silicon using ILIS with EMI-BF4 results in enhanced etching, thanks to the capability of the fluorine in the ion beam to create volatile species that prevent the redeposition of dislocated atoms [1]. Furthermore, ILIS have the crucial advantage of providing negative ions by simply reversing the polarity of the power supply. Irradiation with negative ions can mitigate charging during treatment of dielectric substrates [2]. Ion species available with ILIS include monoatomic species such as I- or Cl-, or kilodalton organic molecules. ILIS are also bright point sources with properties that could make them amenable to operation in a focused ion beam (FIB) column [3].
This talk will review the emission properties of ILIS4, the advancements in improving the reliability and lifetime of the ion sources, the achievements in materials processing, and discuss the opportunities and challenges in the implementation of ILIS in wafer-scale etchers and in FIB applications.

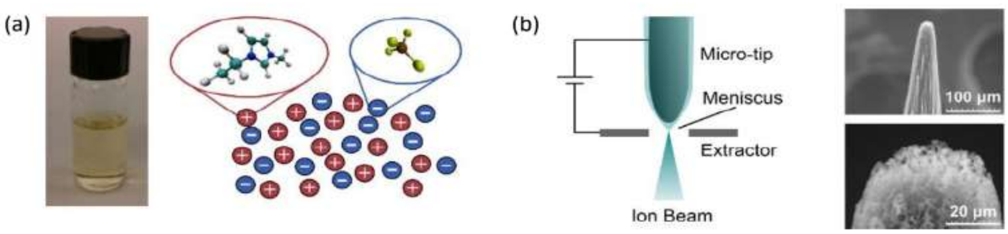

Figure 1. (a) The Ionic liquid 1-ethyl-3-methylimidazolium tetrafluoroborate, EMI-BF_4 (b) ILIS Schematic and scanning electron micrographs of a porous ILIS emitter tip.

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OC2: Coaxial Ion Source: pressure dependence of gas flow and field ion emission

D. Bedrane[1], M. Lagaize[1], A. Houël[2], A. Delobbe[2], Ph. Dumas[1], S. Veesler[1], and E. Salançon[1]

[1] Centre Interdisciplinaire de Nanosciences de Marseille, CNRS, Aix-Marseille Université, CINaM-UMR7325, Campus de Luminy, Case 913, 13288 Marseille Cedex 09, France
[2] OrsayPhysics, Cht De L'arc, 95 AV des Monts Aurélien, 13710 Fuveau
E-Mail: djouher.bedrane@cnrs.fr

Several ion sources are being developed for focused ion beams. In his paper "Quest for high brightness, monochromatic noble gas ion sources," published in 2005 [1], Tondare made a comparison between different sources. He concludes about encouraging results for a « Needle-in-capillary type GFIS (gasfield ion source) » [2,3]. We developed a coaxial ion source (CIS) based on needle-in-capillary type GFIS. The gas is injected locally near the apex of the tungsten tip, so that ionization is produced at the apex at room temperature, large ionization currents can be obtained without degrading the propagation conditions of the beam.

Different experiments have been performed with the coaxial ion source. In this presentation, we show that compared with field ionization in a partial pressure chamber, at room temperature, using the coaxial ion source increases the ion current a hundredfold for the same residual low pressure (see figure 1). The different gases flow regimes are studied here, and has no impact on the ionization efficiency. A fuller characterization remains to be performed, but we estimate that the brightness reaches 3 × 1011 A/m2/sr at 12kV extracting voltage, it exceeds the LMIS (Liquid metal ion source) by an order of magnitude. These results are promising for a further development of the CIS for focused ion beams.

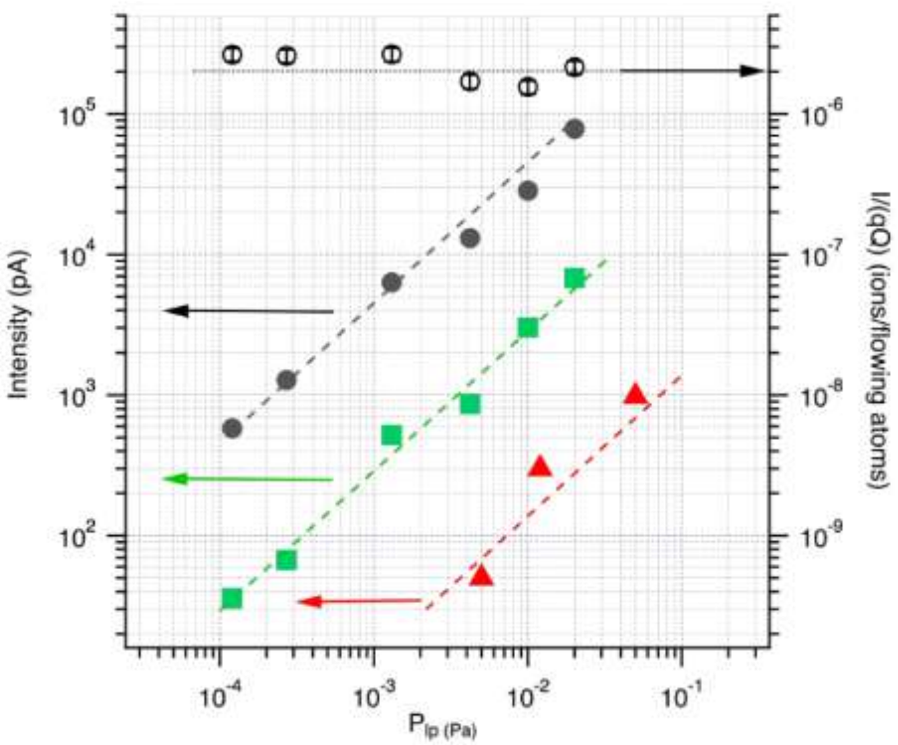

Figure 1. Intensity I (vertical left axis) vs. PlP for three different experiments: for a given CIS geometry, at V = 12.0 kV (solid black circles) and at V = 7.0 kV (solid green squares) and, finally, for a partial pressure experiment at V = 12.2 kV (solid red triangles). The vertical right axis shows the ionisation yield I/(qQ) for the CIS geometry at V = 12.0 kV only (open black circles). Unity slope

dashed lines are a guide for the eyes. Horizontal dotted line shows that I/(qQ) is almost constant. Gas used: argon.

References

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OC3: Crossbeam fs Laser: Integrated Automated Clean Workflow to Access Multiple Deeply Buried Regions of Interest

K. Atlasov

ZEISS Research Microscopy Solutions, Carl Zeiss Microscopy GmbH, ZEISS Group, Carl-Zeiss-Promenade 10, 07745 Jena, Germany
E-Mail: kirill.atlasov@zeiss.com

OC4: Study of Focused Rb^+ Ion Beam Applications

Y. Li[1], S. Xu[1], and E. J. D. Vredenbregt[1]

[1] Department of Applied Physics, Eindhoven University of Technology, P.O. Box 513, 5600 MB Eindhoven, the Netherlands
E-Mail: e.j.d.vredenbregt@tue.nl

Focused ion beams (FIBs) are important tools for materials science and the semiconductor industry. With their applications in milling and material deposition, FIBs enable circuit editing and mask repair during device development and failure analysis in wafer processing. Here a prototype Rb FIB system is presented. The essential innovation is the use of a cold-atom ion source [1] based on photoionization of a laser-intensified and cooled atomic Rb beam. The whole source is mounted onto a commercial FIB column for milling and deposition experiments [2-3].
Stable performance of the Rb^+ beam enabled beam profiling using a knife-edge method. Working at 8.5 keV and 6 pA, the beam was determined to have a d_{50} of 160 nm. After optimization, a d_{50} of a few nm and a predicted reduced brightness of near 1×10^6 A/(m^2·sr·eV) should be achievable, which is comparable with that of the commercial Ga^+ LMIS. Different energies and currents are also available with the beam.
Milling performance is firstly studied using several standard substrates. The experimental results show that Rb^+ has good sputter yield ability in most substrates used, such as B, Diamond, Cu, Au, GaAs, and InP as shown in Fig.1, even compared to 30 keV Ga^+. It should be noted that the Rb FIB system uses 8.5 keV beam energy, which can have the advantage of a short ion projection range due to the low beam energy. A further study has shown that the irradiation damage in the Si substrate causes around 22 nm amorphous thickness and the Rb^+ ion staining is limited to 5.1% in atomic concentration.
Focused-ion-beam-induced deposition (FIBID) was conducted on the Rb FIB using $(MeCp)Pt(Me)_3$ and $W(CO)_6$ as precursors for depositing Pt and W. Parallel deposition experiments were also made on a commercial Ga FIB. Table 1 contains a summary of some key FIBID performance parameters of Rb^+ and Ga^+ under similar beam conditions. The chemical composition of the deposits was achieved from lamella samples (shown in Fig.2) via TEM-EDX. These performance parameters reveal that Rb^+ can create Pt and W deposits at comparable rates and metal contents as Ga^+ while leaving a smaller primary ion content.

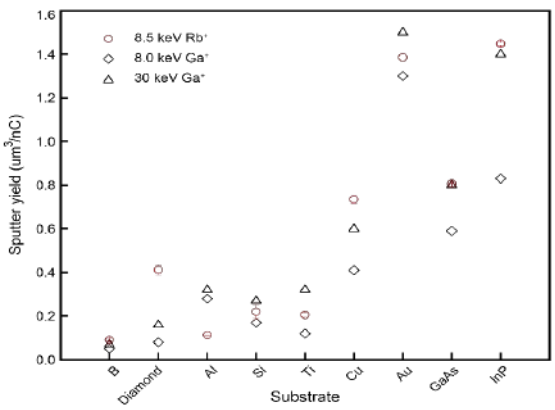

Figure 1. Sputter yield summary of Rb^+ on standard substrates. The diamond markers represent 8.0 keV Ga^+, the triangle markers are for 30 keV Ga^+, and the circle markers represent 8.5 keV Rb^+.

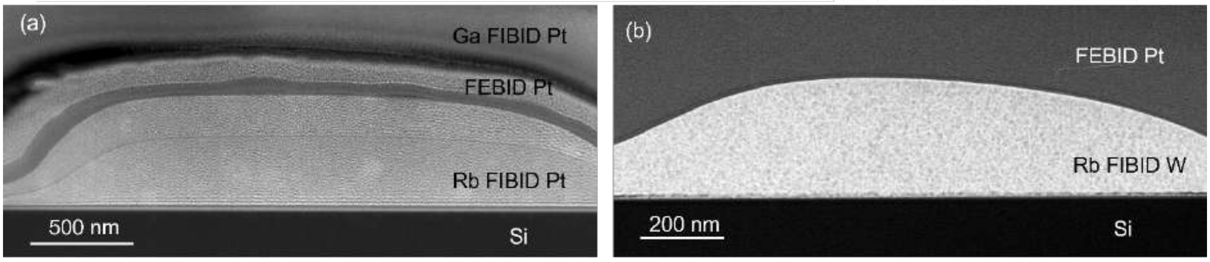

Figure 2. TEM images of (a) Rb^+ induced Pt deposition on Si and (b) Rb^+ induced W deposition on Si.

	Ion	Beam energy (keV)	Beam current (pA)	Deposition yield (μm^3/nC)	Composition (Atomic %)	Resistivity ($\mu\Omega\cdot$cm)	Grain size (nm)
Pt dep	Rb^+	8.5	6.5	0.90	C:O:Pt:Rb 37:33:26:3	$(1.2_{\pm0.4})\times10^4$	$13_{\pm3}$
	Ga^+	8.0	8.5	0.73	C:O:Pt:Ga 22:14:37:27	$(6.4_{\pm0.7})\times10^3$	$14_{\pm2}$
W dep	Rb^+	8.5	10	0.092	C:O:W:Rb 8:14:70:8	340-413	
	Ga^+	8.0	12	0.11	C:O:W:Ga 10:5:70:10[4]	150-225 [4]	

Table.1 FIBID characterization results. The resistivity was measured on deposition over glass with Cr electrodes for contact. The Pt deposits had a granular microstructure while the W deposits had a dense and uniform inner structure.

References

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[4] Stewart, D.K., *et al*. *Proc SPIE*. 1089, 18. (1989).

OC5: Focused Ion Beam Milling with Cold Rubidium

K.T. Mitchell[1], R.W. Speirs[1], C.J. Billington[1], A.J. McCulloch[1], and R.E. Scholten[1]

[1] School of Physics, The University of Melbourne, Parkville, Victoria 3010, Australia
E-Mail: kaihm@student.unimelb.edu.au

Focused ion beam (FIB) sources based on ionisation of laser-cooled atoms promise to expand the capability of FIB techniques for nanotechnology. Enhanced beam brightness, due to the low temperature of the generated ions, enables higher resolution imaging and milling. Advanced ionisation schemes could allow for deterministic single ion implantation and ion trajectory correction. More than 30 elements can be laser-cooled, allowing for high brightness sources for many new ion species.

A FIB apparatus using laser-cooled rubidium atoms has been developed for use in nanofabrication and imaging. Our rubidium FIB aims to achieve a higher beam brightness and a smaller focus spot size than existing state of the art gallium FIB systems. The ion source is designed to achieve brightness in excess of 10^7 A $m^{-2}sr^{-1}eV^{-1}$, with sufficient beam current for both milling and microscopy.

We present the design and preliminary results from our cold atom ion source. We generate a neutral cold atomic beam from a vapour loaded 2D magneto-optical trap (MOT) [1]. The transversely cooled atoms are pushed out of the 2D MOT along the longitudinal dimension, are further cooled in a polarisation gradient cooling stage, and are then photoionised. We use a two-step photoionisation process, allowing control over the ionisation volume and beam energy [2], maximising beam brightness. This ionisation scheme can be adapted to allow for unique capabilities including: coincident electron/ion detection and feedback for high-fidelity heralding of ions [3]; Rydberg exceptional state field ionisation for reducing beam energy spread and chromatic aberration; and Rydberg blockade for isolating single ions.

References

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OC6: Negative Ion HIM

P. Jackle[1], A. Bunevich[1], D. L. Silva[1], M. Dehnel[2], and K. L. Kavanagh[1]

[1]Department of Physics, Simon Fraser University, Burnaby, BC, V5A 1S6, Canada
[2]D-Pace Inc., Nelson, BC, V1L 4B6, Canada
E-Mail: kavanagh@sfu.ca

High-current negative-ion beams have always been more difficult to obtain than positive ion beams [1]. Negative helium (He^-) ion beam in an accelerator is often produced via double charge exchange between a positive helium ion (He^+) source and a low-pressure alkali metal vapour [2]. While the efficiency of this reaction is adequate (1% – 5%), the alkali metal vapour introduces unwanted side effects including contamination of the accelerator and target.
We are using our He Ion Microscope (HIM) as an intensive source of He+ ions to measure charge up take after transmission through suitable thin membranes. We question whether there are materials that might offer at least a similar charge uptake efficiency as do the alkali metal vapours. We started with carbon foils which have been carefully studied down to low energy ions (10 keV) [3, 4]. The He+ beam (15 keV to 30 keV, 50 fA to 10 pA) collides with a carbon foil laid over a beam-limiting aperture. The transmitted particles (He^+, He^o, and He^-) are separated electrostatically by the octopoles normally used for beam scanning into beam spots focused onto a digital camera below the sample stage.
An example of one HIM result is shown in the figure below [5]. With a 20 nm graphite membrane, most of the transmitted beam neutralized with perhaps 10% He^+ions remaining, with a small fraction (0.05%) of He^-ions also detected. The figure shows a negative He ion spot deflected in the opposite direction to the larger transmitted He^+ion spot, with the majority of transmitted He found in a diffuse neutral beam limited in its detected scattered diameter by a column aperture. The negative He ion fraction obtained is consistent with previous reports [3, 4]. The fraction of electron uptake varies with the energy loss and hence velocity of the exiting ion. Thicker amorphous carbon films, eg. 50 nm, transmitted only neutral ions. We are currently investigating thinner amorphous or crystalline films of various compositions including 2D materials and will discuss the latest results at the meeting.

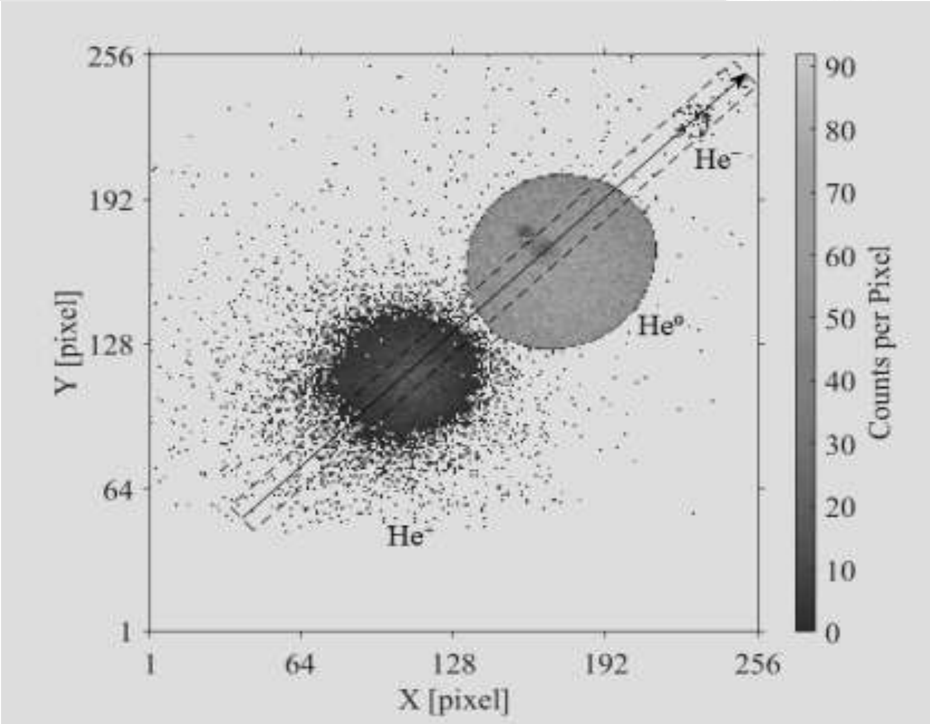

Figure 1. Transmission helium ion microscopy image of a He$^+$ion beam transmitted through graphite (20 nm) covering a limiting aperture (25 keV, 4 pA, total dose 2.5 × 10^6ions). The intensity per pixel is indicated by the grey scale at the right. Submitted to J. Inst. (2022) proc. 8th Intl. Symp. on Negative Ions, Beams, and Sources, Padova, Italy, 2022.

Acknowledgments
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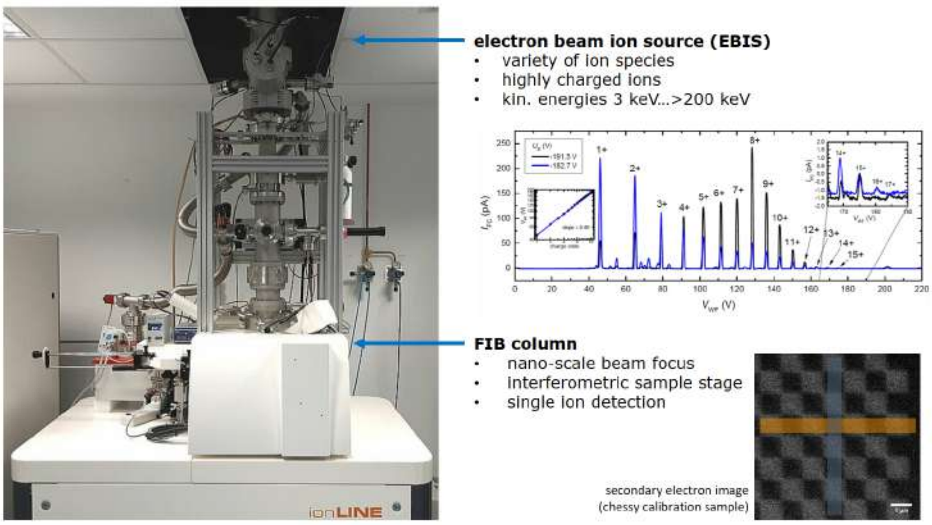
OC7: Focused Beams of Highly Charged Ions for Quantum Technologies

P. Räcke[1,2,3], D. Reinhardt[2,3], J. Meijer[2,3], and D. Spemann[1,3]

[1] Tools, Leibniz Institute of Surface Engineering (IOM), 04318 Leipzig, Germany
[2] Applied Quantum Systems, Felix Bloch Inst. for Solid State Physics, Universität Leipzig, 04103 Leipzig, Germany
[3] Leibniz Joint Lab "Single Ion Implantation", 04318 Leipzig, Germany
E-Mail: paul.raecke@physik.uni-leipzig.de

Solid-state systems such as diamond, silicon or silicon carbide are promising hosts for single photon sources, quantum sensors or qubits in the form of point defects, single dopants or colour centres within their crystalline structure. While extensive research has accumulated detailed knowledge regarding the control and behaviour of single centres or ensembles, their spatially precise, deterministic and reproducible fabrication is still a major challenge on the way to scalable quantum technologies. Ion implantation is a widely and very successfully used tool for semiconductor doping and material modification since the beginnings of semiconductor research and industry. The utilisation of focused ion beam (FIB) techniques is particularly useful for novel quantum technologies requiring nano-scale precision of single ion placements.

In the Leibniz Joint Lab "Single Ion Implantation", we have built a unique ion implanter, which consists of a commercially available FIB machine, equipped with an electron beam ion source (EBIS). It is capable of producing highly charged ions of a variety of ion species from the gaseous state and is designed to incorporate single ion detection methods for deterministic ion implantation. So far, focused beams of hydrogen, helium and argon ions were used to optimise the ion optical system, characterise single ion detection at the sample stage and for defect engineering studies. Furthermore, we are in the process of extending the capabilities towards nitrogen, phosphorous and other ion species. On the one hand, this ion implanter enables precise low-keV ion implantation for quantum applications; on the other hand, the possibility to choose from multiple up to very high charge states enables convenient ion kinetic energy selection from less than 10 up to several hundred keV, which is remarkable considering the compactness of the system. This presentation gives an overview of the functionality of our focused ion beam implanter and highlights some recent results, including single ion detection and local colour-centre creation in diamond.



OC8: Comparison of Ga+ FIB and Plasma FIB systems used for microstructural characterization of a solid oxide fuel cell (SOFC) electrode

K. Berent[1], V. Bertschová[2], K. Novotný[2], S. Kakizawa[3], T. A. Prokop[4], A. Takasaki[3], and G. Brus[4]

[1] AGH University of Krakow, Academic Centre for Materials and Nanotechnology, 30 Mickiewicza Ave., 30059 Krakow, Poland
[2] TESCAN ORSAY HOLDING, a.s., 21 Libušina třída, 62300 Brno, Czech Republic
[3] Shibaura Institute of Technology, College of Engineering, Department of Engineering Science and Mechanics, 3-7-5 Toyosu, Koto-ku, Tokyo 135-8548, Japan
[4] AGH University of Krakow, Faculty of Energy and Fuels, 30 Mickiewicza Ave., 30059 Krakow, Poland
E-Mail: kberent@agh.edu.pl

Solid Oxide Fuel Cells (SOFCs) are energy conversion devices that can convert the chemical energy of a fuel directly into electrical energy [1]. This technology is very promising because provide clean and low carbon emission energy. One of the advantages of SOFCs is their high efficiency, which can be up to 60%, it is higher than traditional combustion-based power generation systems. The conventional SOFC single cell structure is a three-layer one consisting of two porous electrodes (anode and cathode) separated by a dense ion conducting electrolyte. Each component of the microstructure has a specific transport function [2].

Our research focuses on the microstructural analysis of electrodes, which plays an important role in optimizing the conductivity and reaction rate of SOFCs. In the first stage, we used gallium (Ga^+) Focused Ion Beam (FIB) coupled with a Scanning Electron Microscope (SEM) for 3D reconstruction of the electrode microstructure, but its removal rate of material is limited. This limitation was overcome by using xenon (Xe^+) Plasma FIB (PFIB) technique, which can mill structures of up to several hundred microns [3]. Electron tomography was carried out on an FEI Versa 3D SEM and TESCAN AMBER X PFIB SEM. Avizo software ver. 2022.1 was used to stack and segment the SEM images yielding a three dimensional reconstruction and to quantify the microstructure. 3D reconstruction of SOFC anode obtained using both techniques is shown in Fig. 1.

The use of Xe^+ ion beam allowed us to analyze a larger volume of material. The microstructural properties affecting electrochemical performance, such as TPB (*Triple Phase Boundary*) length, tortuosity, and phase volume fraction was calculated. PFIB compared to Ga^+ FIB result in more data statistics, accurate and representative microstructural data, particularly when studying complex and intricate microstructures. However, special attention should be paid to the step size. The difference in imaging resolution between PFIB (15 nm) and Ga-FIB (2.5 nm) is significant. Our measurements show that if we calculate the average grain size of different phases, we get a larger size for the PFIB because we lose information from the smallest particles, which were a few nanometers in size.

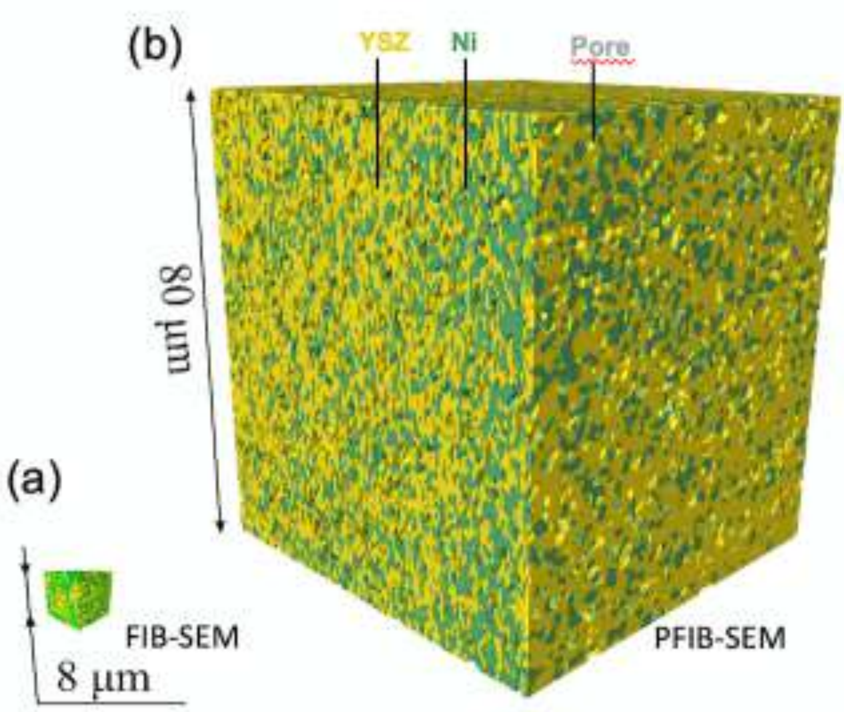


Figure 1. 3D reconstruction of SOFC anode using (a) Ga^+ FIB-SEM and (b) PFIB-SEM.

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OC9: Automation of FIB-SEM process and open-access control of nanopatterning

M. Hrabovsky[1], J. Dluhos[1], M. Jurasek[2], A. Siudova[2], and T. Boruvka[2]

[1] TESCAN ORSAY HOLDING, Brno, 62300, Czech Republic
[2] TESCAN Brno s.r.o., Brno, 62300, Czech Republic
E-Mail: milos.hrabovsky@tescan.com

One of the main challenges of full utilization of FIBSEM in current Nanoprototyping environment is automation of the process, to reduce downtime. Current portfolio of Tescan Essence software modules offer large variety for lamella prep, 3D tomography, automatic imaging, nanopatterning and depositions.

For highly demanding tasks, where the users require absolute freedom of parameters and steps in his workflow, we offer newly released SharkSEM Automation Toolbox SDK, based on python. Users get the possibility to program and develop their own patterning strategies based on their research in novel materials and patterning techniques. The biggest advantage are complex 3D depositions where precise patterning control is the key to a successful working prototype.

The users can also utilize Tescan Stream Files, for the definition of their own patterning strategy via the scripting interface, making it more compatible with already existing 3rd party tools within the community.

To fully utilize the microscope we allow the users to access the hardware and software controls to for example develop their own detector and test it in real world conditions.

OC10: In-situ Monitoring of Two-Dimensional Transistors under Low Energy Focused Ion Beam Irradiation

M. Titze[1], C. M. Smyth[1], A. K. Behera[1,2], E. S. Bielejec[1], B. L. Doyle[1], and A. Belianinov[1]

[1] Sandia National Laboratories, Albuquerque, NM, 87123, USA
[2] Department of Industrial and Manufacturing Systems Engineering, Kansas State University, Manhattan, KS, 66506, USA
E-Mail: mictitz@sandia.gov

Two-dimensional materials are of interest for a variety of applications, including sensing and photoconversion. Additionally, integration of 2D materials into integrated devices is scheduled by 2030 with research-grade 2D MoS_2-based transistors outperforming current state-of-the-art Si based devices. To dope 2D materials, we anticipate the need for ultra-low energy ion implantation, since 2D materials have inherently single layer atomic thickness. To enable stopping inside the single atomic layer, overlayers could be used, however, the likelihood of an ion stopping within a single buried layer is small. Instead, we reduce the ion landing energy such that the ion energy is sufficiently low for it to stop within the single layer. We hypothesize that by tuning the ion energy we can achieve at least partial incorporation of the dopant ion into the lattice without the need for high-temperature annealing, potentially making this a back-end-of-line compatible ion implantation technique. Here, we present the use of a focused ion beam (FIB) using liquid metal alloy ion sources (LMAIS) that can deterministically implant into 2D materials. The device under test is a lateral geometry, backside gated 2D transistor suspended on 300 nm SiO_2 / Si substrate. The use of LMAIS-based FIB allows implantation of ~1/3rd of all elements, with more sources still being developed. Most importantly, typical dopant species are available via FIB. While volume production by FIB doping is likely not economical, FIB implantation can be used for fast prototyping, and in our case, for in-situ measurement of device characteristics under irradiation to quickly iterate on the most promising implantation parameters.
A picture of an in-situ probes device is shown in Figure 1(a). Only two probes are visible since the 3rd probe is contacting the back gate outside the FOV of the camera. A microscope picture showing the device under test is shown in Figure 1(b). Multiple contact pads are deposited on a 2D flake, with a transistor being formed between any two pads. Notably, the device can be operated in a bipolar configuration and only the gate voltage switches it. The device geometry is schematically shown in Figure 1(c), the SiO_2 acts as the gate dielectric and the Si substrate is used as the metallic gate.

Figure 1. (a) Device as probed in-situ via electrical probes. (b) Microscope image of the 2D flake showing 3 devices contacted by 4 wires. (c) Schematic of a 2D material backgated device. The 300 nm SiO2 acts as the gate dielectric.

While FIB has a spot size as low as <5 nm, here we irradiate the whole device by rastering the beam over it for every iteration. The resulting transfer curves for implantation with a 1 keV Au beam are shown in Figure 2(a). Each IV sweep consists of scanning the gate voltage from 0 V to +50 V, then to – 50 V and back to 0 V. This way, hysteresis is observed when traps are generated in the device since these traps are being filled on the positive sweep while they are being depleted on the negative sweep, leading to a threshold voltage shift. When reducing the ion landing energy, we expect that the ion energy is sufficiently low that no defects are generated by an ion, leading to the absence of hysteresis from defect filling / defilling during the gate voltage sweep, as shown in Figure 2(b). Additionally, we anticipate that Au acts as a p-type dopant, shifting the threshold voltage. However, we do not observe a systematic shift of the threshold voltage, instead the observed changes in threshold voltage are ascribed to lifting and relanding of probes inbetween experiments. Two possibilities may lead to the lack of changes in transfer characteristics; [1] Au lands on the 2D material but does not get incorporated into the lattice and therefore is not electrically active. [2] The ion beam rastering is calibrated for a 1 keV landing energy, at 10 eV the ion optics may lead to a larger area being irradiated leading to an error in the fluence calculation.

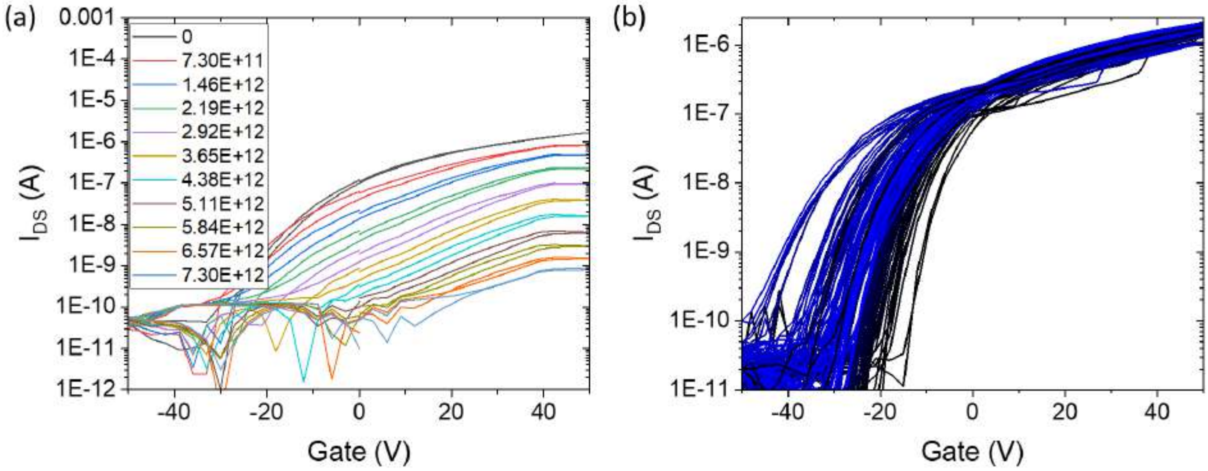

Figure 2. (a) 2D Device under 1 keV Au irradiation; the legend shows fluence in ions/cm^2. The transfer curve becomes hysteretic with increasing irradiation fluence while the drive current collapses simultaneously. (b) 2D Device under 10 eV Au irradiation. The apparent changes in transfer are from changes in contacting from probe lifting and relanding, not a shift in device transfer from ion implantation. Lighter color indicates higher ion fluence. In total 80 traces are shown.

We have demonstrated introduction of hysteretic behaviour of 2D transistors using a 1 keV Au focused ion beam through in-situ electrical transport measurement. A pathway towards implantation at 10 eV is shown, however, we are unsuccessful in modifying the transfer behavior of the device under test, either due to lack of incorporation of Au ions, or because the ion fluence calculation at 10 eV is erroneous. Further experiments will explore higher ion landing energy to attempt ion incorporation as well as higher fluence low energy implantation.

Acknowledgements
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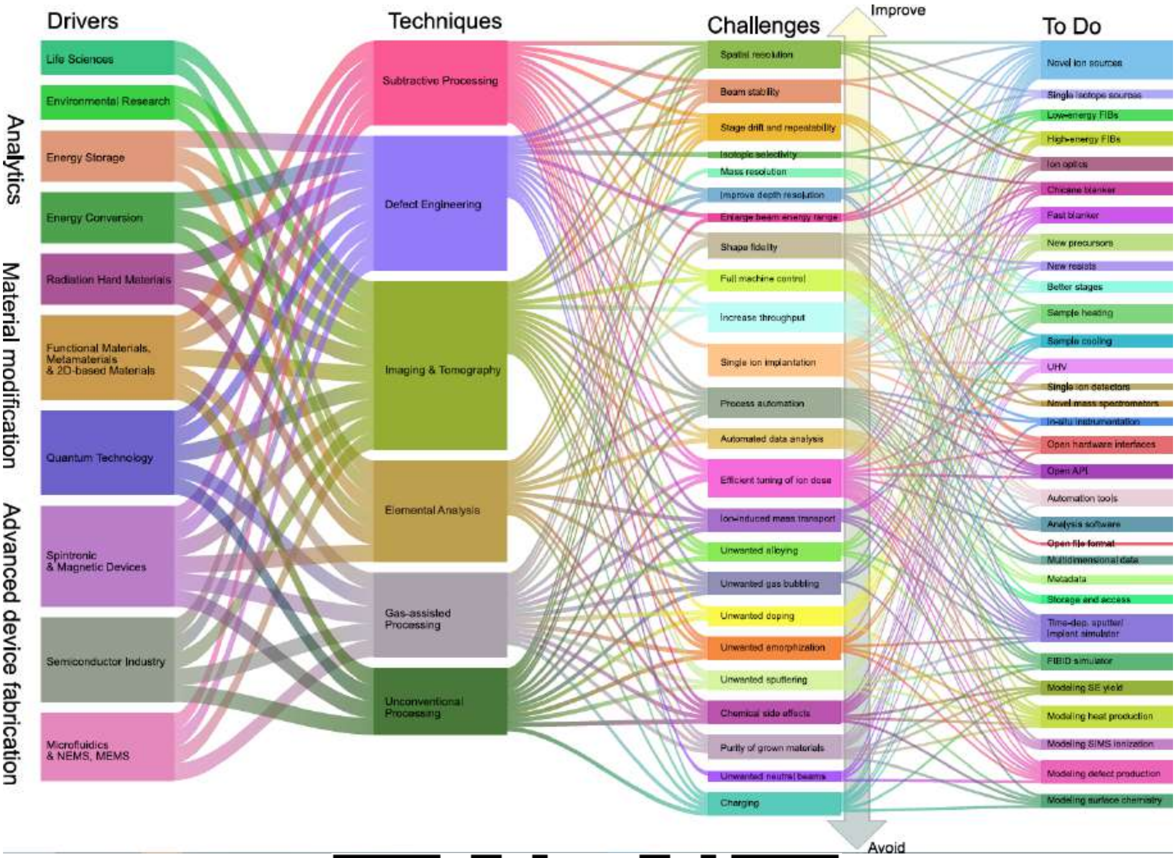
OC11: FIT4NANO FIB Roadmap

K. Höflich

Ferdinand-Braun-Institut gGmbH, Leibniz-Institut für Hönchstfrequenztechnik, 12489 Berlin, Germany
E-Mail: katja.hoeflich@fbh-berlin.de

Presentation of FIT4NANO's 'Roadmap for focused ion beam technologies' accessible at https://arxiv.org/pdf/2305.19631.pdf



OC12: Surface near Helium damage in materials studied with a high throughput implantation method

P. Hosemann[1,2], M. Balooch[1], S. Stevenson[1], and Y. Xie [1]

[1] University of California at Berkeley, Department of Nuclear Engineering, CA, Berkeley, 94720, USA
[2] Lawrence Berkeley National Laboratory, Material Science Division, CA, Berkeley, 94720, USA
E-Mail: peterh@berkeley.edu

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.

OC13: Irradiation with focused helium ion beams as a tool for engineering the superconducting properties of copper-oxide high-*Tc* superconductors on the nanoscale

W. Lang[1], B. Aichner[1], M. Karrer[1], C. Schmid[2], K. Wurster[2], L. Backmeister[1], P. A. Korner[1], R. Kleiner[2], E. Goldobin[2], and D. Koelle[2]

[1] Faculty of Physics, University of Vienna, 1090 Wien, Austria
[2] Physikalisches Institut, Center for Quantum Science (CQ) and LISA+, Universität Tübingen, 72076 Tübingen, Germany
E-Mail: wolfgang.lang@univie.ac.at

Many proposed concepts for superconducting devices rely on the controlled fabrication of vortex pinning centers, predetermined paths of vortex motion, and nonreciprocal propagation. The magnetic coupling of these Abrikosov vortices is a necessary prerequisite, as their spacings must not exceed the London penetration depth at the intended operating temperature. The latter must be significantly lower than the critical temperature T_c to avoid adverse effects from thermodynamic fluctuations. While appropriate fabrication techniques are available for metallic superconductors, the required nanoscale resolution in copper-oxide superconductors is challenging due to their complex atomic structure and susceptibility to environmental influences. The severe constraints on conventional lithographic techniques can be overcome by leaving the material's crystallographic framework intact and tailoring the superconducting properties through the controlled introduction of point defects.

To this end, the focused beam of a helium ion microscope (HIM) is used to fabricate narrowly spaced nanocolumns in thin films of copper-oxide superconductors. Inside these nanocolumns, the critical temperature T_c is reduced or entirely suppressed due to pair-breaking by numerous point defects. The interaction of helium ions with primarily oxygen atoms in the $YBa_2Cu_3O_{7-\delta}$ (YBCO) thin film produces these point defects. Simulations of He^+ ion-target interactions using the SRIM/TRIM code and, in addition, calibrated with experimental data [1] predict that focused 30 keV He^+ ion irradiation will result in well-defined columns of non-superconducting material in the superconducting matrix.

We present the results of several experiments that demonstrate this concept. (i) At an unprecedented high magnetic field of 6 T, vortex commensurability effects in YBCO thin films with a nanocolumn spacing of 20 nm show pronounced maxima of the critical current and corresponding resistance minima. At this matching field B_1, each nanocolumn pins exactly one flux quantum Φ_0. Furthermore, as shown in the Figure, the matching phenomenon persists over a wide temperature range, down to 2 K, far below T_c. (ii) Scalable critical behavior is observed in voltage-current isotherms near the second-order glass melting transition. The latter exhibits a distinct peak at the matching field and a significant increase in the lifetime of glassy fluctuations, which we attribute to the recently discovered ordered Bose glass phase [2]. It can emerge from a vortex Mott insulator when thermal energy and disorder weaken the vortex correlations. (iii) Angle-dependent magnetoresistance measurements in constant Lorentz force geometry point to a substantial increase of anisotropy compared to a pristine reference film when the density of vortices matches those of columnar defects [3]. (iv) Complex pinning landscapes, like quasi-kagomé tiling, exhibit temperature-tunable vortex caging [4] and nonreciprocal transport effects.

Finally, the potential use of focused helium-ion beam modification in the fabrication of more complex circuits such as cellular vortex automata is discussed.

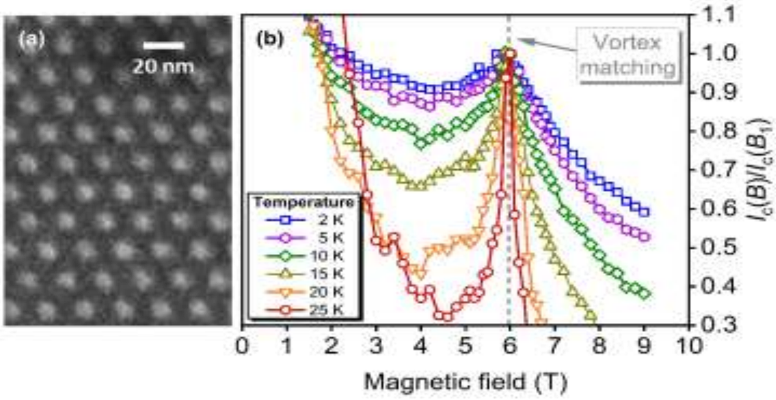


Figure 1. (a) Secondary electron image of a reference sample taken in the HIM to visualize the hexagonal array with 20 nm spacings after irradiation with an excessive dose D = 60 000 ions/dot. The white spots are amorphous blister-forming regions. The samples for the electrical measurements were irradiated with D = 10 000 ions/dot and showed no contrast in imaging. (b) Critical currents at different temperatures, normalized to their maxima at the matching field B_1 vs. applied magnetic field. © by the authors.

Acknowledgments
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OC14: Room Temperature Ferromagnetism in non-magnetic semiconductors

H. Luitel[1, 2]

[1]Nar Bahaduer Bhandari Govt. college, Tadong, Gangtok-737102, India
[2] Ex-research scholar, Variable Energy Cyclotron Centre, 1/AF Bidhannagar, Kolkata-700064, India
E-Mail: luitelhomnath@gmail.com

Defect induced room temperature ferromagnetism in non-magnetic semiconductors has attracted a lot of research attention in the present era due to its potential applications in non-volatile memory storage and spintronics devices [1, 2, 3]. We have carried out both experimental and theoretical studies of the room temperature ferromagnetism in various non-magnetic semiconductors. Defects (atomic vacancies and doping) have been created in the oxide samples by ion beam irradiation technique. The defect profile of irradiation has been simulated using SRIM/TRIM software and experimentally characterized using positron annihilation spectroscopy (LT/CBD) Technique. Strong ferromagnetic ordering at room temperature (~300 K) has been observed in 10 keV B^+ [4], 50 keV N^{4+} [5], 1.2 MeV C^{4+} [6] irradiated oxide samples, TiO_2 and SnO_2 polycrystalline samples with cationic vacancies and TiO_2 with anionic vacancies [7]. Also, room temperature ferromagnetic ordering has been observed in methylammonium lead halide perovskite sample which is a direct band gap semiconductor for the very first time [8, 9]. Ab-initio calculations of ferromagnetic ordering have been performed in various p block elements doped oxides (viz., TiO_2, SnO_2, ZnO) and also with atomic defects [10, 11]. A significant amount of induced magnetic moment has been observed in most of the cases and the primary source of magnetism is the p-orbital electrons of the dopants and neighboring atoms for atomic vacancies. The theoretical calculations are consistent with the experimental observations.

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OC15: Fluxonic and Magnonic Devices Enabled by Focused Ion Technology

Oleksandr Dobrovolskiy

University of Vienna, Faculty of Physics, Nanomagnetism and Magnonics, Superconductivity and Spintronics Laboratory, 1090 Vienna, Austria
E-Mail: oleksandr.dobrovolskiy@univie.ac.at

Next-generation computing systems require new materials and platforms for fast and efficient operations with data. Addressing this challenge, fluxonics [1] and magnonics [2] offer low-energy operations with data carried by magnetic flux quanta (Abrikosov vortices or fluxons) and collective spin precessions (spin waves and their quanta – magnons). Featuring velocities up to several tens of km/s, length scales (vortex lattice periodicity and magnon wavelength) down to few tens of nm and a broad frequency range from rf to sub-THz, the fluxon and magnon dynamics become vibrant avenues of research with application potential for quantum technologies. In both these domains, ion beam technology plays a key role for the realization of various applications, such as single-photon detectors, quantum interferometers, and various microwave devices [3].

In my talk, I will demonstrate three devices explored in my group which all are enabled by focused ion beam technology. First, I will introduce a Nb-C superconductor written with a focused ion beam, which supports 15 km/s vortex velocities due to fast relaxation of unpaired electrons and its high structural uniformity [4]. This makes Nb-C a candidate material for single-photon detectors. Next, I will proceed to a Co-Fe ferromagnet in which a single nanogroove milled by a focused Ga ion beam allows for the realization of a spin-wave phase shifter up to a full phase reversal [5]. This material is relevant spin wave nano-optics and can be used for the steering of spin waves in two or three dimensions. Therein, the phase shift originates from both, the reduced magnetization and conduit thickness in the area exposed to the ion beam. Finally, I will present our results on the Cherenkov-type generation of spin waves in Co-Fe by fast-moving flux quanta in Nb-C [6]. The system acts as a dc-to-microwave converter and, due to the magnon Josephson effect, is a prospective platform for magnon quantum metrology.

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OC16: Tungsten Field Emitters Fabricated by Helium Ion Beam Integrated with Microelectromechanical Systems

E. Gacka[1], G. Hlawacek[2], K. Kwoka[1], and T. Gotszalk[1]

[1] Department of Nanometrology, Wrocław University of Science and Technology, 50-372, Wrocław, Poland
[2]Institute of Ion Beam Physics and Materials Research, Helmholtz-Zentrum Dresden-Rossendorf, 01328, Dresden, Germany
E-Mail: ewelina.gacka@pwr.edu.pl

Due to the development of electronics, new methods of prototyping microelectromechanical systems (MEMS) as sensors, e.g. microcantilevers or microbridges, are being pursued. The leading technology for their fabrication is photolithography, which, however, is limited by the diffraction of visible light. Consequently, new methods of prototyping nanoelectronic devices that simultaneously enable their integration into MEMS are being sought. MEMS-integrated nanoelectronic devices are fabricated to perform a specific function, such as detecting MEMS movement induced by an external factor. Popular methods for detecting MEMS deflection are optical, capacitive, piezoresistive, or tunnel current [1]. However, a method based on field emission, that is, emission/quantum tunnelling of electrons through an insulator induced by an electrostatic field, is constantly being developed. Until now, the popular method for fabricating field emitters was the Spindt method. It is based on the fabrication of a cathode – a metal tip, surrounded by a metal gate electrode (anode). However, this method is a multistep process. This inconvenience could make it difficult to integrate emitters fabricated by the Spindt method into MEMS. As an alternative, emitters can be fabricated using helium ion microscope (HIM) and a selected precursor by the focused ion beam-induced deposition (FIBID) process.

During the presentation, tungsten field emitters deposited on cantilevers will be shown. The cantilevers, fabricated by photolithography, consist of alternating Si_3N_4/SiO_2 layers to minimize leakage currents and stresses. The design of proposed MEMS allows for optical (a mirror on the apex), thermal and electromagnetic (a metallization loop around the edges) actuation, as well as the development of a detection method based on the field emission phenomenon – Fig. 1. To prevent the occurrence of leakage currents resulting from the halo effect during FIBID, neon beam milling of the holes between the electrodes was performed. Nanowires, as field emitters, were deposited at the spots marked by a circle in Fig. 1, between the holes, using the FIBID process. A Zeiss Orion NanoFab HIM and W(CO)6 precursor was applied for this purpose. For the fabricated structures, emission was obtained at about 70 V. The stability of the emission current was verified.

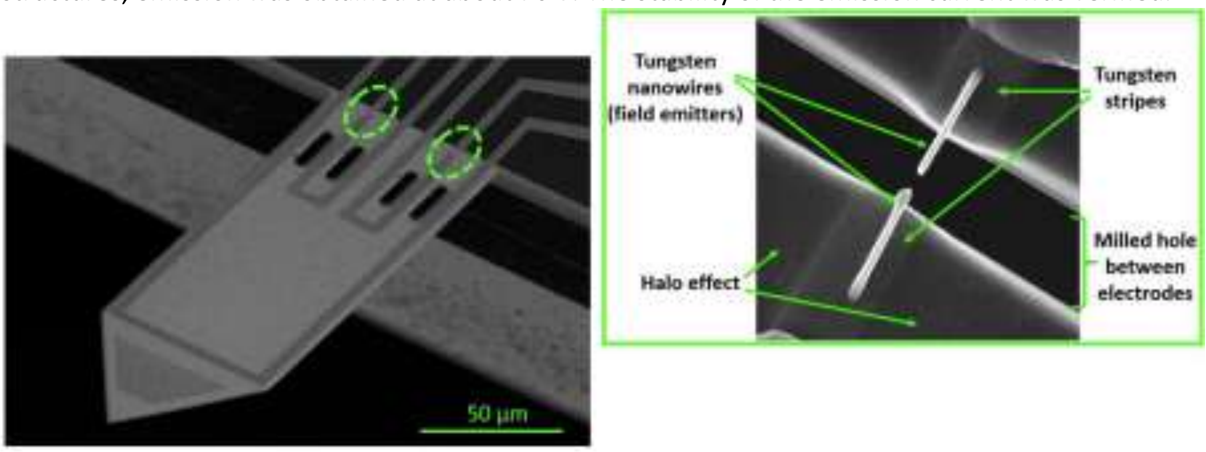

Figure 1. Image of deposited tungsten field emitters on a cantilever.

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OC17: Focused ion beam modification of MEMS cantilevers by stress engineering

B. Pruchnik[1], E. Gacka[1], T. Piasecki[1], D. Cox[2], and T. Gotszalk[1]

[1]Department of Nanometrology, Wrocław University of Science and Technology, 50370, Wrocław, Poland
[2]Advanced Technology Institute, University of Surrey, GU2 7XH, Guildford, UK
E-Mail: bartosz.pruchnik@pwr.edu.pl

Microelectromechanical systems (MEMS') are widely developed as sensors, actuators and tools for nanotechnology. Manufacturing methodology derives from CMOS technology, which allows for precise, repeatable manufacturing of large batches of MEMS devices. For this purpose however, rapid prototyping or small-serial development is often suboptimal. At the same time MEMS' are widely applied in nanometrology, where their features find plenty of applications. However, experimental works often require specialized devices differing in some range of parameters. As stated, classic manufacturing technology doesn't meet this requirement.

In case of MEMS devices, postproduction may serve to adjust given parameters of single devices. Apart from topological modification by milling or depositing in the proximity of prefabricated device, also material of the device can be altered. Localized change in material composition may lead to the stress generation with strain incompatibility due to thermal expansion coefficient mismatch. Direct stress implementation is possible in overconstrained devices eg. membranes, bridges. Then it leads to material modifying phenomena through topological modification [1] up to phase change. In devices with some degrees of freedom, however, stress is followed by strain. If structure is modified asymmetrically, then strain induces deflection.

With use of focused ion beam (FIB) it is possible to modify MEMS locally by surface milling or localized doping. Both operations generate thermal expansion coefficient gradient along cross-section. Therefore bimorph structure is created (fig. 1). For microcantilever, which is simple structure with one degree of freedom, this lead to controlled deflection in one direction. Even stronger effect can be achieved with focused ion beam induced deposition (FIBID), where complementary layer is added on top of existing structure.

Apart from tuning initial stress and deflection of the device, modifications performed with FIB can serve the purpose of enhancement of the devices. Deposition of auxiliary layer not only generates thermal stress', but additionally improves efficiency of thermal actuation. New possibility emerges to develop unitary devices and tune their dynamic properties accordingly to given parameters. We present an approach to modification of thermally actuated microcantilevers in which thermal efficiency of actuation [2] was tuned in a range of two orders of magnitude (fig. 2).

Specifically localized modification can prove useful not only in enhancement of parameters, but also in expanding possibilities of an actuated structure. Local amplification of thermally induced stress' changes harmonic behavior of the vibrating structure, enhancing amplitudes of vibrations in higher eigenmodes. Structures improved in such a manner will find their use in precise nanoscopy with active piezocantilevers.

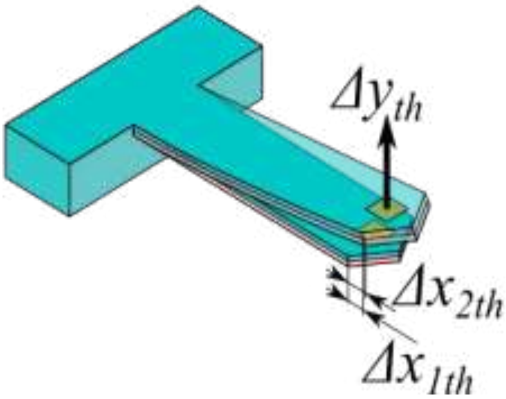

Figure 1. Thermomechanical actuation principle of exemplary bimorph microcantilever [2].

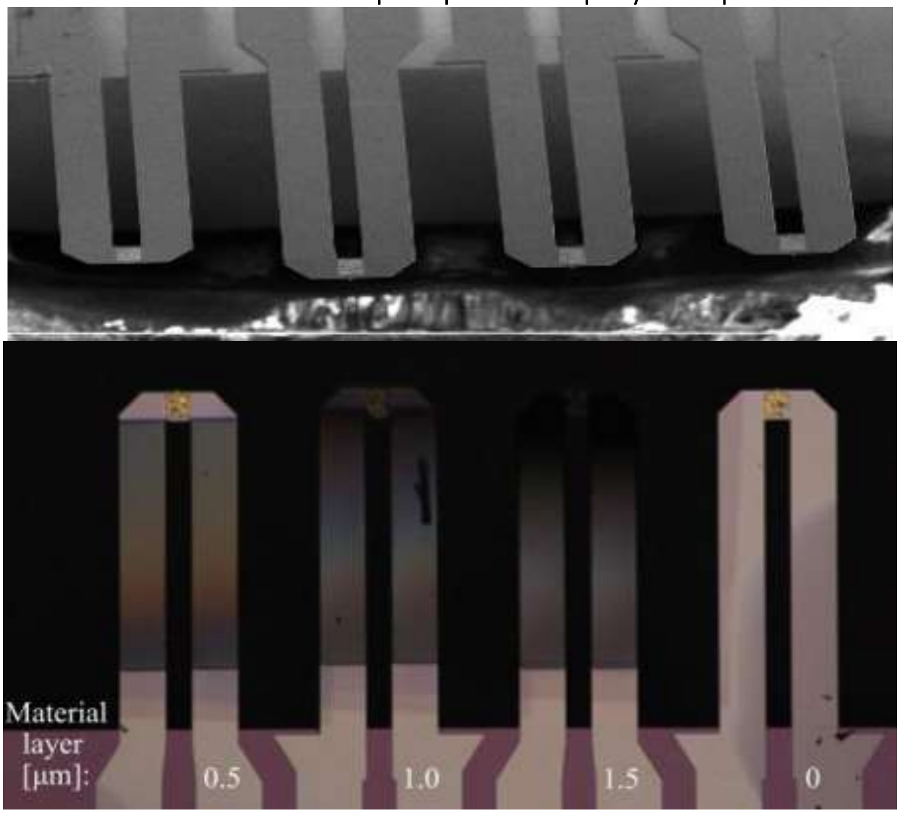

Figure 2. Microcantilevers with deposited FIBID – layers of amorphous carbon.

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OC18: Self-organized nanopatterning of Ge (100) surface under low-energy ion beam sputtering

S. Hans[1, 2], and M. Ranjan[1, 2]

[1] Institute for Plasma Research, Bhat, Gandhinagar, Gujarat, India-382428
[2] Homi Bhabha National Institute, BARC Training School Complex, Anushaktinagar, Mumbai, Maharashtra, India-400094
E-Mail: sukritihans21@gmail.com

One key aspect of ion beam sputtering (IBS) process is the modification of nanoscale surface topography, as it is simple and cost-effective method for fabrication of large-area surfaces for various technological applications [1]. Special attention has been focused on monoelemental semiconductors, as they are simple one-component systems and get readily amorphized during ion sputtering. Oblique incidence ion beam leads to nanoripple like pattern formation on various surfaces. Moreover, the periodicity of these ripple patterns can be tuned from around tens up to several hundred nanometers by varying the ion energy and ion fluence. Several investigations regarding ripple pattern formation have been done on Si and it has been found that ripple formation depends mainly on the ion incidence angle, and the surface remains smooth up to a critical angle. The principal applicability of these ion-induced nanoscale ripple patterns is in microelectronic devices as well as templates in the growth of functional thin films [2].
Recently, it has been observed that nano triangular features superimpose these ripple patterns when Si (100) surfaces are irradiated with broad Ar ion beam at oblique incidence, which limits their use for some potential applications like SERS [3,4]. It is desirable to produce highly ordered ripple patterns to make them suitable from application point of view. It is indeed important to understand the formation of triangular features and the basic underlying physics behind their evolution.
In this work, ion irradiation experiments were performed on Ge (100) surfaces with Xe ions by tuning ion energy, ion incidence angle and ion fluence. In this case also triangular features appear with the onset of ripple formation but with a much better ordering compared to Si surfaces. There are some striking similarities of triangular feature dynamics wrt ion fluence, which is, the base angle of triangular features remains constant with increase in ion fluence whereas the lateral length increases in both the cases. Numerical integrations performed using anisotropic-Kuramoto-Sivashinsky (AKS) equation are in good qualitative agreement with the experimental results and suggest that the formation of triangular features during ion beam sputtering is mainly due to dispersion process which is an essential mechanism along with curvature-dependent sputtering and diffusion processes for the pattern formation [3, 5]. This study shows that exploiting dispersion experimentally can lead to formation of highly ordered ripple patterns [6].

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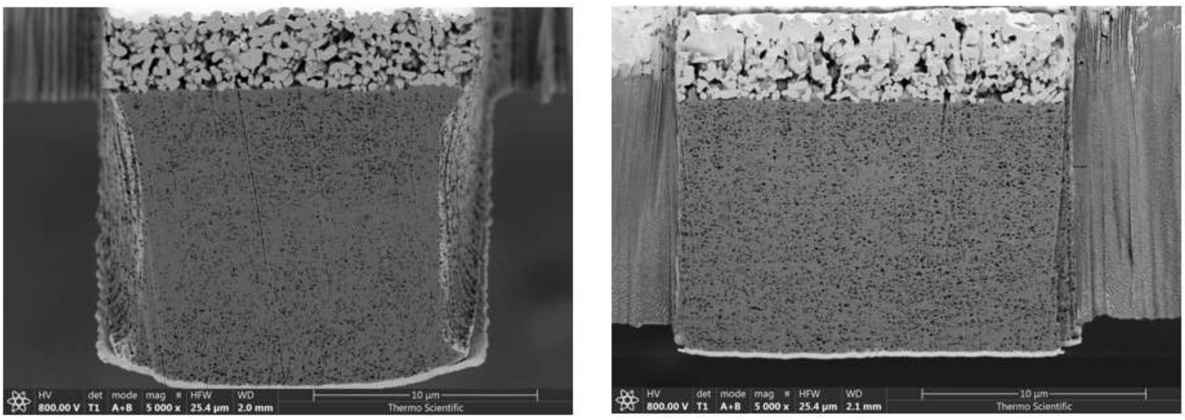
OC19: Damage free 3D characterization and TEM sample preparation of beam sensitive materials using advanced multiple ion source PFIB under cryogenic condition

M. Wu, L. Li, and D. Wu

Thermo Fisher Scientific, Eindhoven, The Netherlands
E-Mail: min.wu@thermofisher.com

Direct quantitative investigation of the inner morphology and structure of materials is of critical importance to provide profound insights for properties evaluation. The scanning electron microscope (SEM) and focused ion beam (FIB), combined known as FIB-SEM or DualBeam, are conventionally recognized as a highly effective method to acquire 3D volume information of materials. FIB-SEM together with integrated automated serial sectioning software has made the 3D data acquisition and analysis possible in an unattended manner. However, slicing using Ga ion beam at room temperature has been found inducing severe damage to beam sensitive materials, resulting in significant deterioration of the 3D data quality. Cutting edge multiple ion source plasma FIB (PFIB) technique with integrated cryo stage provides a fully automated workflow which allows large volume, damage-free ion beam slicing and high spatial resolution SEM acquisition during serial sectioning under cryogenic conditions. With automated 3D reconstruction of the micrograph stacks, we can subsequently recover the comprehensive volume information of such beam sensitive materials. In addition, cryogenic multiple ion source PFIB has been confirmed to be capable of fabricating high quality large area TEM lamellae without damaging the beam sensitive bulk samples. Coupled with cryogenic in-situ nanomanipulator, the TEM samples can be easily lifted out under cryogenic conditions and subsequently transferred to TEM.
In this paper we present a series of large volume 3D imaging results and TEM sample preparation examples of extremely beam sensitive samples. The samples were processed using xenon, oxygen, or argon plasma ion source on Thermo Scientific Helios Hydra Plasma FIB platform under cryogenic conditions. The slicing and imaging acquisition was achieved using Automated Slice and View software and the subsequent data processing was conducted using Avizo 3D analysis and visualization software. The unique technical experiment set up and comprehensive application experience will be discussed in this presentation.



Room Temperature Milled Milled at -125 °C

Figure 1 shows a direct comparison of a commercially available highly beam sensitive PE membrane sample being sliced at room temperature and under cryogenic conditions. Beam damage caused by FIB slicing at room temperature is clearly visible, while under cryogenic condition, the sample has maintained structural integrity and no beam damage is observed.

OC20: Bimetallic precursors for focused particle-based deposition

S. Barth[1, 2], F. Jungwirth[1, 2], F. Porrati[1], N. Jochmann[1, 2], M. Huth[1], D. Knez [3], and H. Plank [3]

[1] Physics Institute, Goethe University Frankfurt, 60438, Frankfurt, Germany
[2] Institute for Inorganic and Analytical Chemistry, Goethe University Frankfurt, 60438, Frankfurt, Germany
[3] Institute of Electron Microscopy and Nanoanalysis, Graz University of Technology, 8010 Graz, Austria
E-Mail: barth@physik.uni-frankfurt.de

In recent years new precursors for charged particle deposition techniques have been synthesized and their performance tested using experimental conditions. In these studies, significant improvements in the understanding of fragmentation processes in the electron-induced fragmentation as well as ion-based process have been gained [1].
In this contribution, we present differences and similarities for FEBID [2] and Ga-FIBID [3] writing of Co/Si-based material as well as currently acquired results for Fe/Si deposits from single source precursors. Deposition parameters have been altered and the changes in composition, growth rate, microstructure etc. have been determined. The electrical transport properties have been determined in two and four-probe configuration and the temperature dependence of the material has been recorded. We trace back differences in FIBID and FEBID-derived materials to composition and microstructure, while discussing also differences in magnetotransport [3].

Acknowledgments
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OC21: Ni, Pd, Cu, and Ag FEBID/FIBID potential precursors' interactions with electrons

I.B. Szymańska[1], A. Butrymowicz[1], and K. Madajska[1]

[1] Faculty of Chemistry, Nicolaus Copernicus University in Toruń, Gagarina 7, 87-100 Toruń, Poland
E-Mail: pola@umk.pl

There is still a lack of appropriate stable and user-friendly compounds to obtain pure nickel, palladium, copper, and silver deposits with a defined 2D and 3D shape and without post-deposition purification [1−2]. The studies on the FEBID/FIBID precursors demonstrate the high complexity of the process and the need to study the mechanisms of interaction of molecules with charged particles on the surface and in the gas phase to find the relationships between the composition and structure of the compounds and their volatility and sensitivity to the electron/ion beam. One class of precursors for focused electron beam induced deposition (FEBID) is the group of silver(I) carboxylates $[Ag_2(\mu\text{-}O_2CR)_2]_n$ (R = perfluorinated or bulky group), which fabricated 2D and 3D deposits (purity up to 76 at.% Ag) [1]. On the other hand, for the copper(II) carboxylate $[Cu_2(\mu\text{-}O_2CC_2F_5)_4]$, 3D growth also was observed, but copper nanocrystallites (around 4 nm) embedded in a C_xF_y amorphous matrix were formed. Therefore, only post-deposition purification processes were allowed to obtain deposits of higher (95 at.% Cu) than initial (23 at.% Cu) purity [1]. In addition, studies of low-energy electron-molecule interactions with silver and copper carboxylate complexes suggest that the formation and release of CO_2 may be an important factor in the formation of deposits [1,3].

Therefore, we extended the scope of research on carboxylate compounds with elements from group 10 (nickel and palladium), and we take into account also carboxylate heteroleptic complexes with N-donor ligands (amidines and amines). Additionally, we used structurally similar to carboxylates ligands such as amidinates and amidates – N,N-donor and N,O-donor, respectively. Finally, we have focused on close to β-diketonates, chelating imidoyloamidinates, which are N,N-donor ligands and O,O-donor ketoesterates (Figure 1).

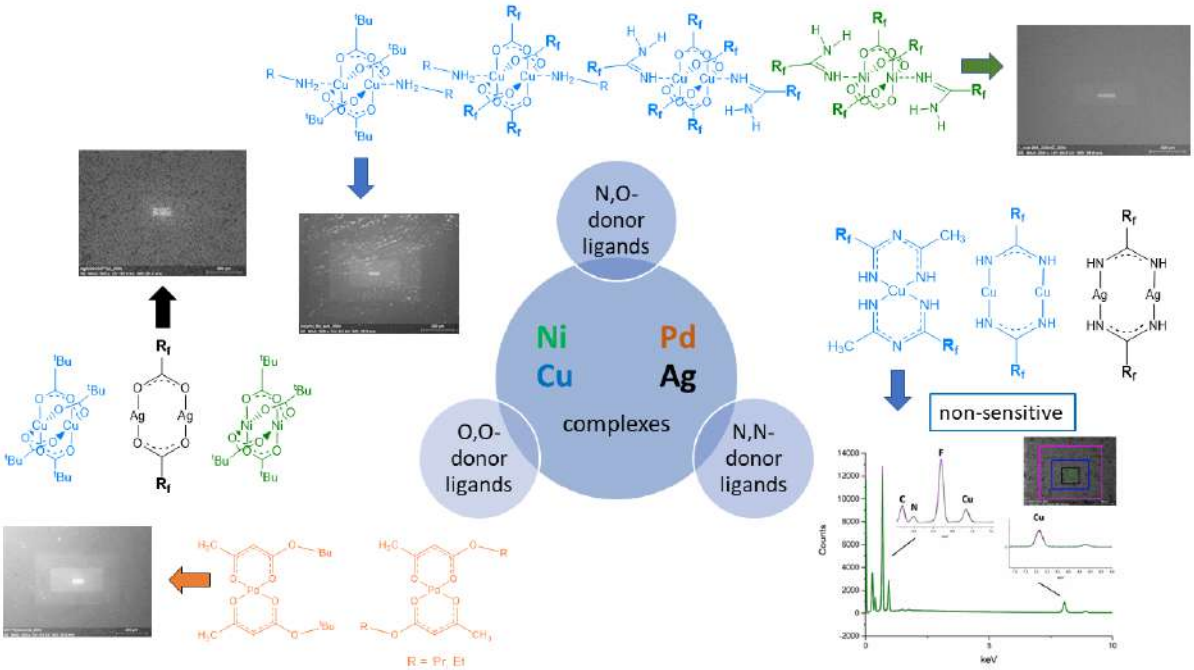

Figure 1. SEM EDS observations for adsorbed on surface potential and used Ni, Pd, Cu, and Ag FEBID/FIBID precursors with O,O- and N,N- donor ligands.

Acknowledgements
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OC22: Analysis of Platin deposition and secondary electron yield on Si, InAs and GaAs at different ion beam voltages and currents with a Cs FIB and a Ga FIB

T. H. Loeber[1], B. Lagel[1], and G. von Freymann[1,2,3]

[1] Nano Structuring Center, Department of Physics, RPTU Kaiserslautern, 67663 Kaiserslautern, Germany
[2] Department of Physics and Research Center OPTIMAS, RPTU Kaiserslautern, 67663 Kaiserslautern, Germany
[3]Fraunhofer Institute for Industrial Mathematics ITWM, 67663 Kaiserslautern, Germany
E-Mail: loeber@physik.uni-kl.de

The deposition of material with an ion beam is essential for FIB applications. For example, the material can be used either as a protection layer in cross sections or as a conductive layer to establish an electric connection. Most important is to reduce damages to the sample surface during the deposition and to achieve a low electrical resistance. The mechanism how the material is deposited with an ion beam from a precursor gas is still not completely understood. The most common theories assume that the deposition is caused by either secondary electrons or excited surface atoms [1].

Here we present the results of Platin (Pt) layers deposited with a Cesium FIB and a Gallium FIB. The former mentioned FIB has a new kind of ion source manufactured by the company ZeroK [2]. Lasers are used to cool down Cs atoms to almost $T = 0$ K which are subsequently ionized with additional lasers. Because the ions have a very low energy spread even at lower acceleration voltages, small ion beam spot sizes can be achieved. Acceleration voltages of 16, 8, 5 and 2 kV are used for the Pt deposition at ion beam currents between 1 and 1000 pA. The Ga FIB is a ThermoFisher Helios 650 NanoLab standard tool and Pt is deposited at voltages of 30, 16, 8 and 5 kV. So far, only one other group reported about Pt deposition with a similar laser cooled ion source, but they are using Rubidium at a low beam current and only one single acceleration voltage [3].

The Cs FIB at the Nano Structuring Center was the first one ever installed at a customer site and the best beam parameters for the deposition had to be found first. Beside the acceleration voltage and the ion beam current, the beam step sizes depending on the beam current and dwell times are set such as to create smooth homogenous deposition layers. The growth rates of the layers deposited with the Cs ion beam are compared with the layers prepared with Ga ions. Beside Silicon also Indium Arsenide and Gallium Arsenide are used as substrates. The deposition and etch rate for the different materials are measured at the above-mentioned acceleration voltages.

Furthermore, we also investigate whether secondary electrons (SE) actually can produce Pt deposition since, in some works, the theory of excited surface atoms is favored [4]. For this, the SE yield was measured at different voltages and ion beam currents for both ion species and then compared with the corresponding growth rates.

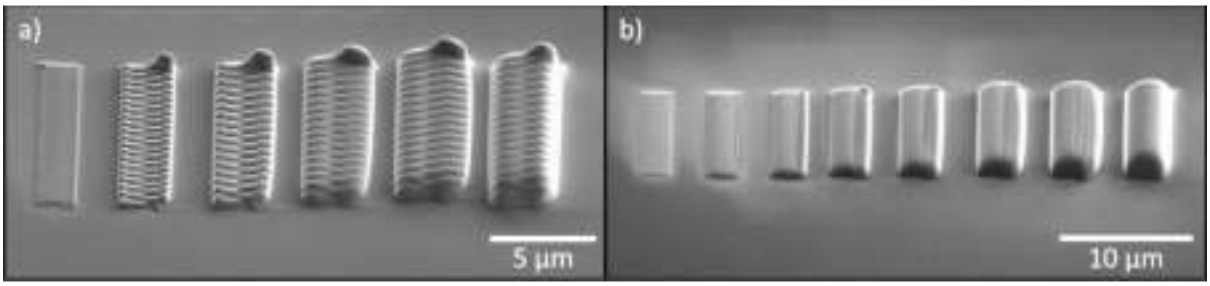

Figure 1. SEM images of Pt layers taken at an angle of 52°. The layers are deposited with the Cs ion beam at a voltage of 8 kV. In each image the ion beam current increases from left to right. Image a) shows inhomogeneous layers before optimization of the beam parameters and image b) with a good beam parameter set.

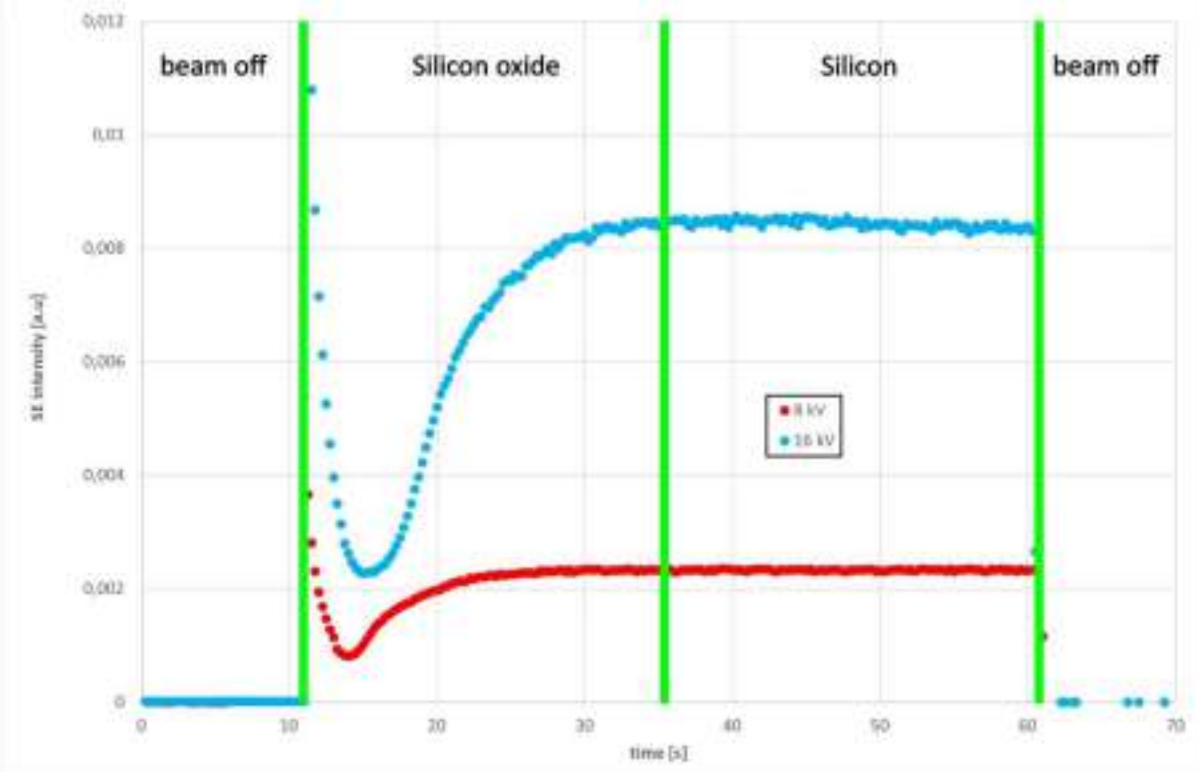

Figure 2. The image shows the course of secondary electron (SE) yields of Silicon recorded with the Cs FIB at voltages of 16 and 8 kV, respectively. The measurements started before the ion beam was turned on. First, the lower SE yield of the native Silicon oxide layer can be seen. After a certain time, this layer is sputtered away and the SE yield of the pure Silicon remains constant. After turning the ion beam off the signal drops back to zero. The SE yield at 16 kV is significantly higher than the SE yield at an acceleration voltage of 8 kV.

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OC23: Method of Testing Decomposition Results of the Metal-organic Precursors for IBID Applications using Gallium FIB

B.R. Jany[1], K. Madajska[2], A. Butrymowicz[2], I.B. Szymańska[2], and F. Krok[1]

[1] Marian Smoluchowski Institute of Physics, Faculty of Physics, Astronomy and Applied Computer Science, Jagiellonian University, Lojasiewicza 11, 30-348 Krakow, Poland
[2] Faculty of Chemistry, Nicolaus Copernicus University in Toruń, Gagarina 7, 87-100 Toruń, Poland
E-Mail: benedykt.jany@uj.edu.pl

We tested Cu and Ag metal-organic compounds [1-2] i.e. $[Cu_2(NH_2NHCC_2F_5)_2(\mu\text{-}O_2CC_2F_5)_4]$, $[Cu_2(\mu\text{-}O_2C^tBu)_4]_n$, $[Cu_2(\mu\text{-}O_2CC_2F_5)_4]$, $[Ag(\mu\text{-}O_2CC_2F_5)]_2$, as new potential precursors for the applications in Ion Beam Induced Deposition (IBID) using Gallium FIB. Metals like Cu and Ag are commonly used in electronic applications and are promising candidates for IBID deposition due to its high electrical and thermal conductivity maintaining moderate costs as compared with much more expensive Pt and Au based compounds. The precursors layers of thickness of few micrometers were deposited by sublimation on the silicon substrate under vacuum (10^{-2} mbar) at earlier determined temperature. Precursor layer decomposition studies were performed using Gallium FIB at FEI's Quanta 3D FEG dual beam SEM/FIB microscope. The decomposition process of the precursor layer results finally in the formation of metal rich structures with a morphology dependent on the irradiation conditions of the Ga FIB beam. The relative changes in the metal content of the irradiated layers were determined by studying the changes in the SEM Backscattered Electrons (BSE) signal intensity, which is a measure of the average atomic number. Chemical composition quantification of the resulted after irradiation structures was performed using combination of SEM EDX together with Machine Learning data processing. First SEM EDX data were acquired in the hyperspectral mode i.e. for each x,y position a full EDX spectrum was recorded. In the next step, the measured hyperspectral data were decomposed by blind source separation (BSS) using non-negative matrix factorization (NMF) [3]. This resulted in separation of EDX signals coming from the silicon support from the EDX signal coming from metal rich structures. Next, the separated signals were quantified by the EDX ZAF method. The metal content of the final structures was successfully determined. It is worth noticing that by this method the tests of new IBID precursors can be successfully performed without the need of expensive equipment like TEM or dedicated GIS systems, here similar results could be obtained by achievable SEM EDX [3]. This also allowed us to study other chemical effects like the effect of gallium implantation into final metal rich structures in a quantitative way systematically for different precursors. The obtained experimental results will be presented and discussed.

Acknowledgements
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OC24: Direct writing of Pd-based micro and nano-structures, electron beam vs Ga^+ beam irradiation

A. Salvador[1,2], L. Herrer[1], S. Sangiao[1,2], P. Philipp[3], P. Cea[1,2], and J.M. de Teresa [1,2]

[1] Instituto de Nanociencia y Materiales de Aragón (INMA), CSIC-Universidad de Zaragoza, 50009, Zaragoza, Spain
[2] Laboratorio de Microscopías Avanzadas (LMA), Universidad de Zaragoza, 50018, Zaragoza, Spain
[3]Luxemburg Institute of Science and Technology (LIST), L-4422, Belvaux, Luxembourg
E-Mail: lucia.h@unizar.es

This contribution aims to present the ability to fabricate metal-enriched micro and nanostructures with excellent control of size, shape and spatial orientation from palladium-based spin-coated films.
To fabricate the structures, the direct writing strategy has been applied by irradiation through an electron beam and a gallium beam. The study carried out and the results are presented here in a comparative manner.
It has been demonstrated that the irradiation of these films with high electron doses, 30 mC·cm^{-2}, results in a really low resistivity for the as fabricated PdNS (palladium nanostructures), namely 145 μΩ·cm, which is only one order of magnitude higher than the value reported for bulk metallic palladium. When the film is decomposed by means of a gallium beam, a dose as low as 30 μC·cm^{-2}is sufficient to produce structures with a metallic Pd content of over 50 % (at.) and an electrical resistivity of 70 μΩ·cm [1, 2].
Both results pave the way for the development of simplified lithographic processes to fabricate micro and nanostructures with: 1) good electrical conductivity, 2) no need for post-treatment steps, 3) almost unlimited freedom of design. A proof of concept for different applications will be presented and the next steps will be outlined.

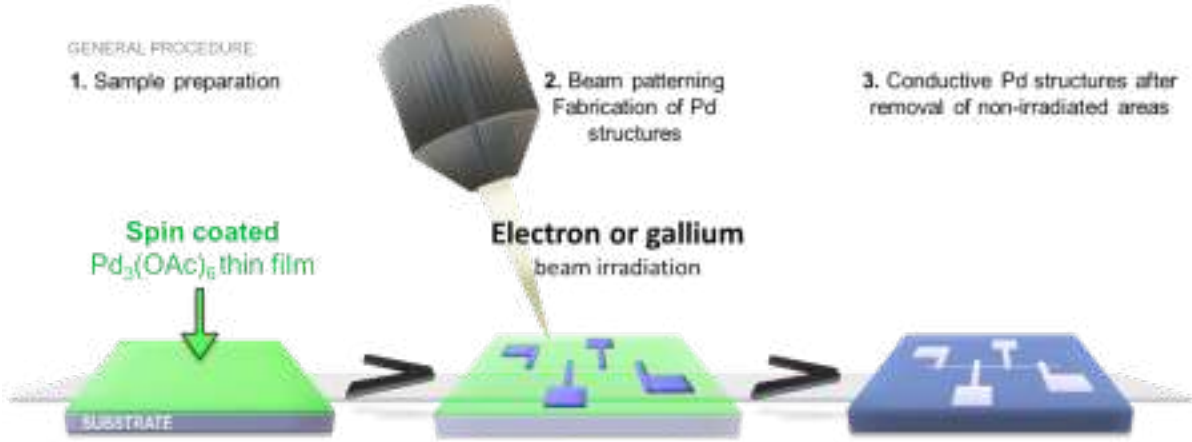

Figure 1. Drawing of the general procedure

Acknowledgments
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OC25: Focused ion beam: from health and bioelectronics to environmental remediation

D. Nunes[1], M. L. Matias[1], T. Calmeiro[1], H. V. Almeida[1], P. Barquinha[1], T. Ferreira-Gonçalves[2,3], C. P. Reis[2,3], E. Fortunato[1], and R. Martins[1]

[1]*CENIMAT|i3N, Department of Materials Science, School of Science and Technology, NOVA University Lisbon and CEMOP/UNINOVA, Caparica, Portugal*
[2]*Research Institute for Medicines (iMed.ULisboa), Faculty of Pharmacy, Universidade de Lisboa, Av. Professor Gama Pinto, 1649-003 Lisboa, Portugal*
[3]*Instituto de Biofísica e Engenharia* Biomédica, Faculdade de Ciências, Universidade de Lisboa, Campo Grande, 1749-016 Lisboa, Portugal
E-mail: daniela.gomes@fct.unl.pt

Focused ion beam (FIB) is a versatile technique largely employed in materials science, mainly in the semiconductor industry and nanotechnology, however, with a growing application in the biological and medical areas. FIB allows imaging, analytical, machining, and sample manipulation for different materials. During FIB experiments, the material surface is irradiated by a focused ion beam of nanometer-order diameter, and accurate patterns can be obtained from a few nanometers to several hundreds of micrometers. Moreover, the precise deposition of specific metals at the nanoscale range is also an essential feature of this technique. This technique is advantageous for scientific research and is generally considered an in-situ nanoscale laboratory. Regarding biological materials, the use of FIB has been increasing lately. However, the nature of biological materials and their interaction with electron and ion beams hinders the analysis of their structures at high resolution. This talk will give an overview of using FIB to investigate different materials and devices. Metal oxide nanostructures will be highlighted, evidencing the sustainability of such materials and their growth on substrates that are flexible, inexpensive, recyclable, and earth-abundant, including plastic, cellulose- and cork-based substrates, and their further investigation using electron microscopy techniques, and associated techniques like FIB and energy dispersive x-ray spectroscopy (EDS). Many studies developed at CENIMAT will be presented. The importance of using FIB for the structural characterization of these materials at the nanoscale integrated with applications ranging from bioelectronics and passing through environmental remediation will be emphasized.

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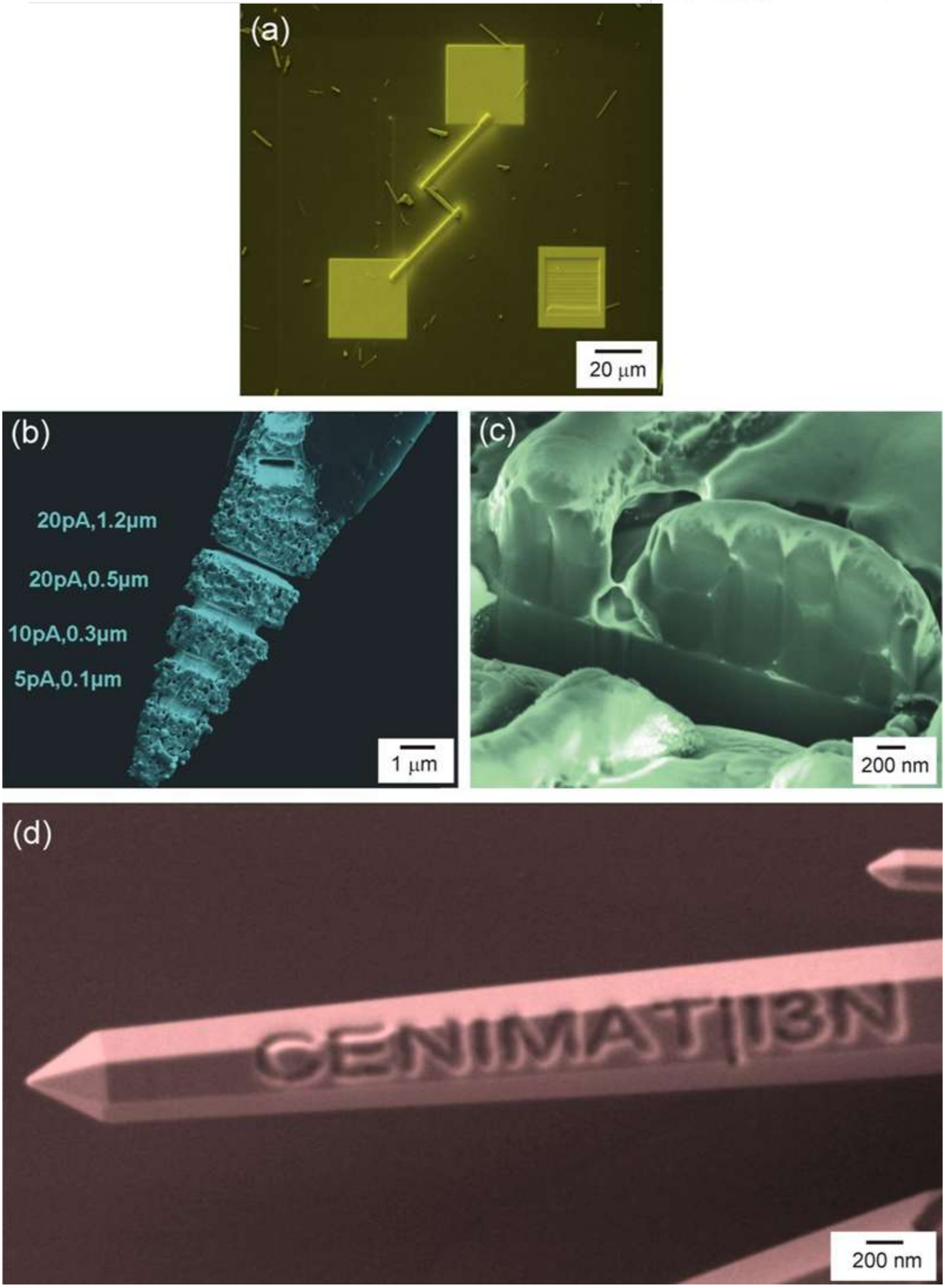

Figure 1. Precise FIB milling and platinum deposition with the gas injection system. (a) ZnO nanowire in a single crystal nano-transistor, (b) Brain electrode modification with several beam currents tested, (c) bacteria internal structure, and (d) an individual ZnO nanowire.

OC26: Magnetic sector SIMS systems for FIB platforms: new developments, applications, and prospects

T. Wirtz, O. De Castro, H.Q. Hoang, A. Biesemeier, and J.-N. Audinot

Advanced Instrumentation for Nano-Analytics (AINA), MRT Department, Luxembourg Institute of Science and Technology (LIST), 4422 Belvaux, Luxembourg
E-Mail: tom.wirtz@list.lu

The structural characterization and the chemical analysis at the nanometer scale are of highest relevance in multiple fields, that can be as diverse as high-resolution imaging of dopant distributions in complex electronic devices, the generation of chemical maps of sub-cellular structures in biological samples to understand the underlying physiological processes, or isotopic ratio measurements at the nano-scale in geological samples. The following key characteristics are required and enabled in our instrument developments: (1) highest spatial resolution, (2) excellent chemical sensitivity, (3) high dynamic range and (4) isotopic selectivity.

Secondary Ion Mass Spectrometry (SIMS) is an extremely powerful technique for analyzing surfaces, owing to its ability to detect all elements from H to U and to differentiate between isotopes, its excellent sensitivity and its high dynamic range. SIMS analyses can be performed in different modes: acquisition of mass spectra, depth profiling, 2D and 3D chemical imaging. Adding SIMS capability to focused ion beam (FIB) instruments offers a number of interesting possibilities, including highly sensitive analytics, highest resolution SIMS imaging (~10 nm), in-situ process control during patterning and milling, and direct correlation of SIMS data with data obtained by other analytical or imaging techniques on the same instrument, such as high-resolution secondary electron (SE) images, back-scattered electron (BSE) images or Energy-Dispersive X-Ray Spectroscopy (EDX) spectra.

In this global context, we developed several generations of double focusing magnetic sector SIMS systems. The latest generation is equipped with a novel continuous focal plane detector. This SIMS system allows for the detection of all masses in parallel for each single pixel, resulting in acquisition times as low as 1 s to obtain a full mass spectrum or 2 min to obtain a 512 x 512 pixel SIMS image with highest signal-to-noise ratio and excellent dynamic range. The advantages over time-of-flight (TOF) systems include the ability of working in the DC mode (providing significantly higher secondary ion (SI) counts for a given analysis duration) and higher overall transmission, resulting in significantly better sensitivity.

This SIMS system is now operating on several multi-modal FIB platforms (Figure 1), including Thermo Fisher DualBeam systems [1], ZEISS ORION NanoFab Helium Ion Microscopes [2-4] and the zeroK SIMS:ZERO platform [5]. The FIB columns of these instruments cover a diverse range of ion species: He, Ne, Ar, Ga, Xe, Cs. Due to their differences in size, mass and chemical reactivity, they lead to differences in sputter yields, fragmentation, dimensions of the collision cascades triggered in the sample and ionization probabilities of the sputtered atoms and molecules.

Here, we will review the performance of the different instruments with a focus on new developments, showcase methodologies for high-resolution 3D chemical imaging, present a number of examples from various fields of applications (nanoparticles, battery materials, photovoltaics, micro-electronics, tissue and sub-cellular imaging in biology, geology, …) (Figure 2) and give an outlook on new trends and prospects.

Figure 1. Different FIB platforms equipped with LIST's magnetic sector SIMS system: ZEISS ORION NanoFab Helium Ion Microscope, Thermo Fisher DualBeam, npSCOPE and SIMS:ZERO (left to right).

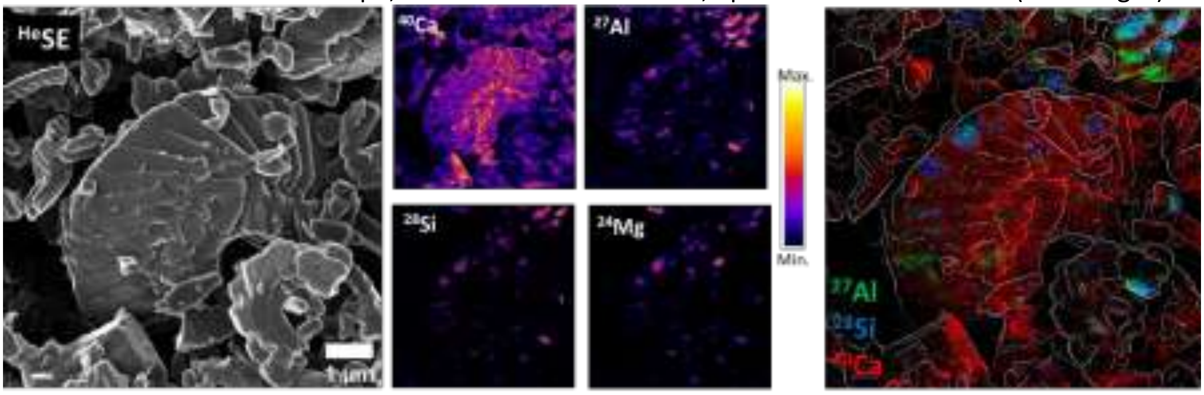

Figure 2. Correlative high-resolution SIMS-SE imaging on a coccolithophore structure on the HIM-SIMS. Left: HIM SE image (25 keV He^+, 0.5 pA, 2048x2048 pixels). Middle: SIMS images (20 keV Ne^+, 3 pA, 512x512 pixels) of ^{40}Ca, ^{27}Al, ^{28}Si and ^{24}Mg. Right: Laplace image fusion of the SE image and the RGB SIMS images of ^{27}Al (green), ^{28}Si (blue) and ^{40}Ca (red).

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OC27: Use for HIM and HIM-SIMS in Correlative Microscopy

F. Vollnhals[1], G. Sarau[2], A. Kraus[1], and S. Christiansen[1,2]

[1] Institute for Nanotechnology and Correlative Microscopy - INAM, 91301 Forchheim, Germany
[2]Fraunhofer Institute for Ceramics Technology and Systems - IKTS, 91301 Forchheim, Germany
E-Mail: florian.vollnhals@inam-forchheim.de

The Helium Ion Microscope (HIM) has revolutionized charged particle microscopy, enabling advances in imaging and nanotechnology applications such as ion beam lithography, nanopatterning, and material modification due to its achievable spot sizes [1]. However, one area where HIM has lagged behind scanning electron microscopy (SEM) is in sample analysis beyond secondary electron imaging. SEMs are commonly equipped with detectors for many signal types generated by the interaction of the electron beam and a sample, ranging from emitted electrons, some carrying specific information such as Auger or diffracted electrons, to light (cathodoluminescence) and X-rays. Such detection systems and other analytical modalities for further characterizing physical or chemical sample properties have been limited for HIM [2].
The HIM community has been making significant efforts to address this limitation by developing ion beam-specific detection tools such as Rutherford backscattering (RBS), Scanning Transmission Helium Ion Microscopy (STHIM), and Secondary Ion Mass Spectrometry (SIMS), which uses the neon ion beam provided by the latest generation ORION NanoFab HIM [3].
In HIM-SIMS, the focused ion beam is scanned across the surface, causing the emission of neutral and ionized atoms or small clusters. The ionized species are then collected and guided to a mass analyzer for detection. This method enables the detection of ions and small clusters ranging from light elements such as hydrogen, lithium, and boron to heavy elements such as lead.
While this new detector enables new experiments and analyses in addition to HIM microscopy, for some samples further information is required to better understand the physico-chemical properties. To achieve this goal, efforts have been made to develop workflows for correlative microscopy using HIM in combination with other analytical modalities, such as atomic force microscopy, optical microscopy, or Raman microscopy, to provide new insights and overcome some of the limitations of individual tools [4]. This is especially useful in the case of sample aspects that cannot be easily analyzed otherwise, such as the Lithium distribution in Lithium ion battery materials, which also require dedicated inert gas workflows.

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OC28: Engineering magnetism in two dimensional phyllosilicates via ion-implantation

M. Z. Khan[1], N. Klingner[2], O. Selyshchev[3,5], S. Valencia[4], G. Hlawacek[2], U. Kentsch[2], F. Kronast[4], C. Teichert[1], G. Salvan[3,5], and A. Matković[1]

[1] Chair of Physics, Department Physics, Mechanics and Electrical engineering, Montanuniversität Leoben, 8700, Leoben, Austria
[2] Ion Beam Center FWIZ, Helmholtz-Zentrum Dresden Rosendorf, 01328, Dresden, Germany
[3] Semiconductor Physics, Chemnitz University of Technology, D-09107, Chemnitz, Germany
[4] Department of Spin and Topology in Quantum Materials, Helmholtz-Zentrum Berlin, Albert-Einstein-Str. 15, D 12489, Berlin, Germany
[5] Centre for Materials, Architecture, and Integration of Nanomembranes (MAIN), Chemnitz University of Technology, 09126, Chemnitz, Germany
E-Mail: muhammad.khan@unileoben.ac.at

The modulation of intrinsic 2D ferromagnetism has attracted massive attention and shown a great potential for spintronics. Although several two-dimensional (2D) van der Waals materials(vdW) exhibit intrinsic magnetism at finite temperatures, lack of ambient stability is still a limiting factor for their integration in 2D device applications. In contrast to the synthetic 2D systems, there is a variety of vdW minerals which offer a wide range of structural and compositional variations. Thus far only our consortium has reported the presence of weak ferromagnetism in 2D iron-rich talc [1] at room temperature, and proposed iron-rich phyllosilicates as air stable platform for magnetic monolayers [2]. These naturally occurring van der Waals (vdW) materials are inherently magnetic and incorporate local moment baring ions of iron (Fe) via substitution of magnesium (Mg) in their octahedral sites. The self inherent capping by silicate/aluminate tetrahedral groups, makes monolayers air-stable.

In order to utilize 2D magnetic minerals in device applications, it is important to exert control over factors such as magnetic ion substitution ratios, magnetic moment baring species and even the process of alloying. Here we further demonstrate an approach to induce magnetic properties by introducing magnetic species (Fe/Co) into non-magnetic talc via focused ion beam (FIB) and broad beam (BB) ion implantation.

From the field of mineralogy, talc ($Mg3Si4O10(OH)2$) is known to be an excellent scaffold to incorporate iron via substitution of Mg in the octahedral sites. In our studies we focus on single crystals, which are possible to mechanically cleave into thin flakes and integrate into heterostructures and devices. Our proposed concept of magnetic ion-implantation into phyllosilicates allows to control and tune the amount of the magnetic species (Fe, Co or Ni) precisely, doping depths, and even alloying in air-stable 2D magnetic minerals. Raman spectroscopy and transmission electron microscopy (TEM) indicates that the implanted magnetic ions (especially at high implantation temperatures) substitute magnesium in talc, without any signature of Fe/Co oxide inclusions. Furthermore, microscopic understanding of the ion implanted structure using superior spatial and spectral resolution of X-rays will yield a strong push towards developing an ion-implantation pathway for 2D magnetic phyllosilicates and their potential applications.

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OC29: FIB for microelectronics: instrument developments and applications

U. Mantz[1], Y. Yu[1], P. Mazarov[1], and T. Richter

[1] Raith GmbH, Dortmund
[2] Raith America, Boston
E-Mail: ulrich.mantz@raith.de

Microelectronics development and production is one of the biggest driver for Focused Ion Beam (FIB) system deployment in industry and represents the largest overall market segment for single FIB or SEM/FIB instruments. Main challenges in microelectronics are not only ever shrinking dimensions but also 3D structures like FinFETs or, more recently, wrap around gate structures. While these trends mostly drive innovation in Ga based automated TEM lamella preparation, innovations in packaging, like multi-chip package integration, require a different, high throughput FIB technology for large area delayering. Both application areas mentioned above, represent FIB based system development and optimization for a very narrow range of applications in high volume semiconductor industry. More recently, 3D NAND tomography is gaining momentum in microelectronics, again leading towards tailored system architectures for in-line frontend and FA lab implementation.
As a result of this situation in semiconductor industry, system capabilities become very specialized for a large portion of the FIB market and often way too expensive or too inflexible for an effective implementation in industrial research, nanotechnology centers or in high-profile universities. Naturally, suppliers focus on industrial equipment with its high margins and high volumes.
As a consequence, the gap between FIB instrumentation in industry versus academic equipment is increasing, making it harder for innovators to generate results which are ready for deployment in industrial workflows. Following the discussion around the Chips Act in Europe and in the US, one prominent goal is exactly, to bridge the valley of death between research and production. To do so, research would need to put more effort into prototype development, which often is not the mission of innovation centers.
Potentially, nanotechnology research and development will help overcoming the challenges listed above. Many nanostructures in research require nanopatterning with high pattern fidelity and uniformity across substrates in order to guarantee reliable nanodevice functionality. Being successful in nanotechnology makes rigor in procedures and processes, known from semiconductor industry, mandatory.
The presentation will highlight aspects of lab to fab challenges for FIB technology and discuss, how development in nanotechnology might help to bridge the gap between research and industrial production. Platforms like the FIT4NANO initiative might serve as an important catalyst to accelerate deployment of significant innovations in industry.

OC30: Polymorph Conversion in Gallium Oxide via Focused Ion Beam

U. Bektas[1], N. Klingner[1], P. Chekhonin[2], M. O. Liedke[3], R. Huebner[1], R. Heller[1], F. Ganss[1], and G. Hlawacek[1]

[1] Institute of Ion Beam Physics and Materials Research, Helmholtz-Zentrum Dresden-Rossendorf e.V. 01328 Dresden, Germany
[2] Institute of Resource Ecology, Helmholtz Zentrum Dresden Rossendorf, Germany
[3] Institute of Radiation Physics, Helmholtz Zentrum Dresden Rossendorf, Germany
E-Mail: u.bektas@hzdr.de

Monoclinic gallium oxide (β-Ga2O3) is the chemically and thermally most stable compound, compared to its other four polymorphs, with an ultra-wide bandgap of 4.9 eV. It is a promising semiconductor material for power electronics, optoelectronics, and batteries. However, controlling the metastable polymorph phases is quite hard, and the fabrication technology at the nanoscale is immature. Our goal is to utilize and understand ion-beam-induced polymorph conversion. Controlling the crystalline structure will allow us to establish new fabrication methods of single-phase polymorph layers, buried layers, multilayers, and different nanostructures in Ga2O3 using focused ion beams (FIBs). The research aims to better understand and control the polymorph conversion, emphasizing spatially resolved modifications by utilizing focused ion beams.
Most of the semiconductor materials transform into an amorphous phase under a high dose of ion irradiation, however, gallium oxide is an exceptionally radiation-tolerant material even at high fluences. In a previous study, Kuznetsov et.al. [1] demonstrated the ion-beam-induced β-to-κ phase transformation in Ga2O3 as shown in Fig.1. However, later, Garcia Fernandez et.al. [2] showed that the monoclinic β-phase actually transforms into the cubic γ-phase. Additional experimental and simulation results suggest that the formed γ layer is not only stable up to several hundred degrees but can also tolerate high fluences of additional ion irradiation. The conversion from the stable to the metastable phase seems to be enabled by the formation of a defective spinel structure in which the oxygen lattice remains unchanged [3].
Here, we used Helium Ion Microscopy (HIM) and liquid metal alloy ion source (LMAIS) FIBs to locally irradiate (-201)-oriented β-Ga2O3 substrate with different ions (Ne, Ga, Co, Nd, Si, Au, In) to induce the polymorph transition. The successful conversion into γ- Ga2O3 under Ne+irradiation (Fig.2(a)) has been confirmed by Electron Backscatter Diffraction (EBSD) and analyzing the Kikuchi patterns (Fig.2(b)). Furthermore, Doppler broadening variable energy positron annihilation spectroscopy (DB-VEPAS) and Rutherford Backscatter Spectrometry (RBS) were performed for neon-broad-beam-irradiated implants to better understand the fluence-dependent creation and distribution of defects. Transmission Electron Microscopy (TEM) images provide information about the interfaces between different polymorphs of Ga2O3. The first results indicate that the damage/strain created by the Ne+, Co+, and Si+FIB irradiation leads to a local transformation of β- Ga2O3 to γ- Ga2O3, and the structure maintains its crystallinity up to high-fluence FIB irradiation instead of being amorphized.

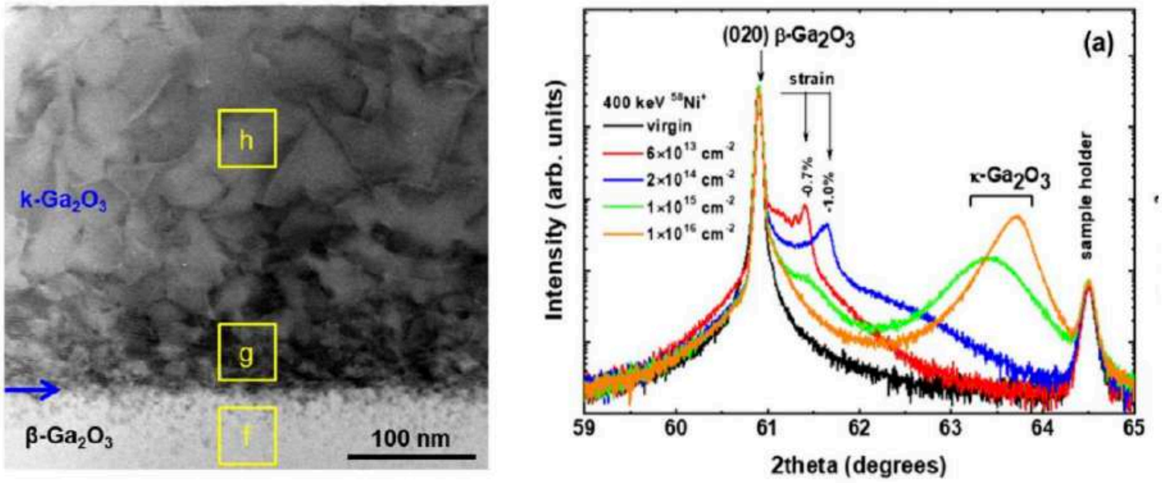

Figure 1. (left) ABF-STEM image of ion-beam-induced β-to-κ phase transformation in Ga_2O_3 under 400 keV $^{58}Ni^+$ broad-beam irradiation with a fluence of $1x10^{16}$ Ni^+/cm^2, (right) XRD data of samples implanted with 400 keV $^{58}Ni^+$ions as a function of ion dose [1].

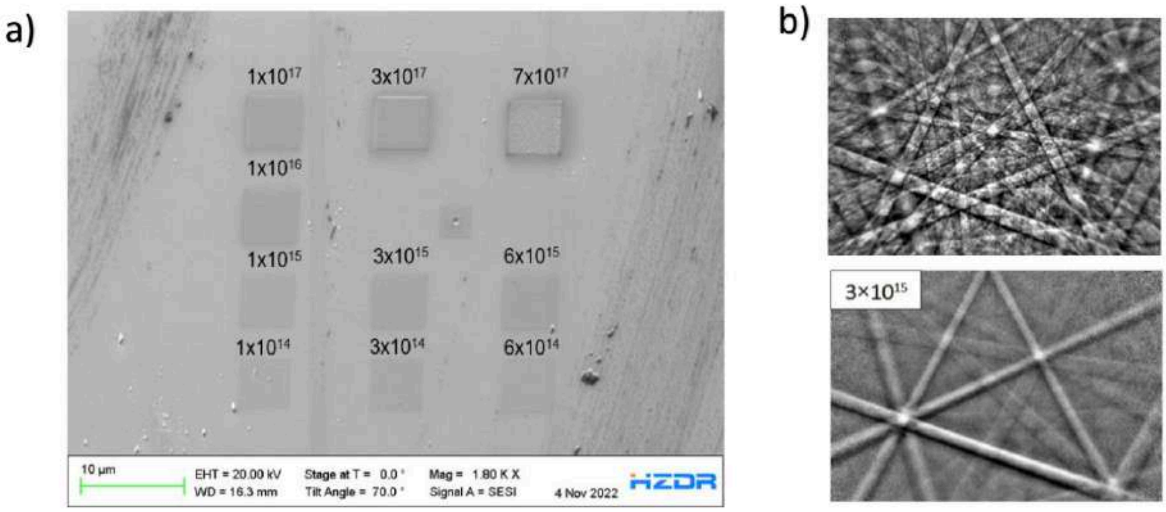

Figure 2. (a) β-Ga_2O_3 substrate irradiated with 25 keV Ne^+ ions at different fluences. Each irradiation area has a size of 5 µm x 5 µm. (b) (top) Kikuchi pattern from the unirradiated area (β-Ga_2O_3), (bottom) Kikuchi pattern of transformed area (γ-Ga_2O_3) after 25 keV Ne^+irradiation with $3x10^{15}$ Ne^+/cm^2ion fluence

Acknowledgements
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OC31: On demand spatially, controlled fabrication of single photon emitters in Silicon by liquid metal alloy ion source focused ion beam implantation

N. Klingner[1], M. Hollenbach[1], L. Bischoff[1], G. Hlawacek[1], and G. V. Astakhov[1]

[1]Institute for Ion Beam Physics and Materials Research, Helmholtz-Zentrum Dresden-Rossendorf, 01328 Dresden, Germany
E-Mail: n.klingner@hzdr.de

Single photon emitters (SPE) are fundamental building blocks for future quantum technology applications. However, many approaches lack the required spatial placement accuracy and Si technology compatibility required for many of the envisioned applications. Here, we present a method to place single or few SPEs emitting in the telecom O-band1. The successful integration of these telecom quantum emitters into photonic structures such as micro-resonators, nanopillars and photonic crystals with sub micrometer precision paves the way toward a monolithic, all-silicon-based semiconductor-superconductor quantum circuit for which this work lays the foundations.
To achieve our goal, we employ home built AuSi liquid metal alloy ion sources (LMAIS) and an Orsay Physics CANION M31Z+ focused ion beam (FIB). Silicon-on-insulator substrates from different fabrication methods have been irradiated with Si++ 40 keV ions in a spot pattern of 6 to 500 ions per spot.
For the analysis and confirmation of the fabrication of true SPEs a home build photoluminescence setup has been used. G-centers formed by the combination of two carbon atoms and a silicon atom with a zero phonon lines (ZPL) at 1278 nm have been created in carbon rich SOI wafers. In ultra clean SOI wafers W-centers, a tri-interstitial Si complex has been created with a ZPL at 1218 nm. The achieved lateral SPE placement accuracy is below 50 nm in both cases and the success rate of SPE formation is more than 50%.
Finally, we give an overview on possible other applications and give an outlook on our ongoing project for single ion implantation.

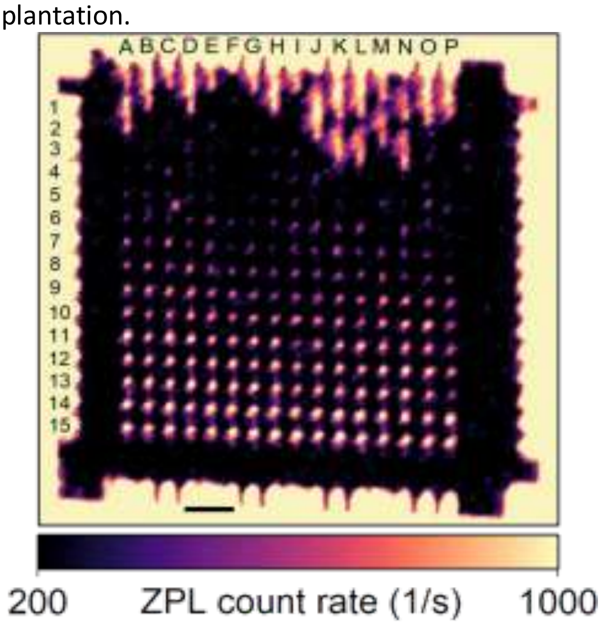

Figure 1. Intensity map of locally created G centers on a silicon on insulator wafer. The number of ions per spot increases logarithmically from nominally 6 (row 1) to 570 (row 15). The pattern frame is created with a fluence $\Phi = 1 \times 10^{11} cm^{-2}$. The scale bar is 20 µm.

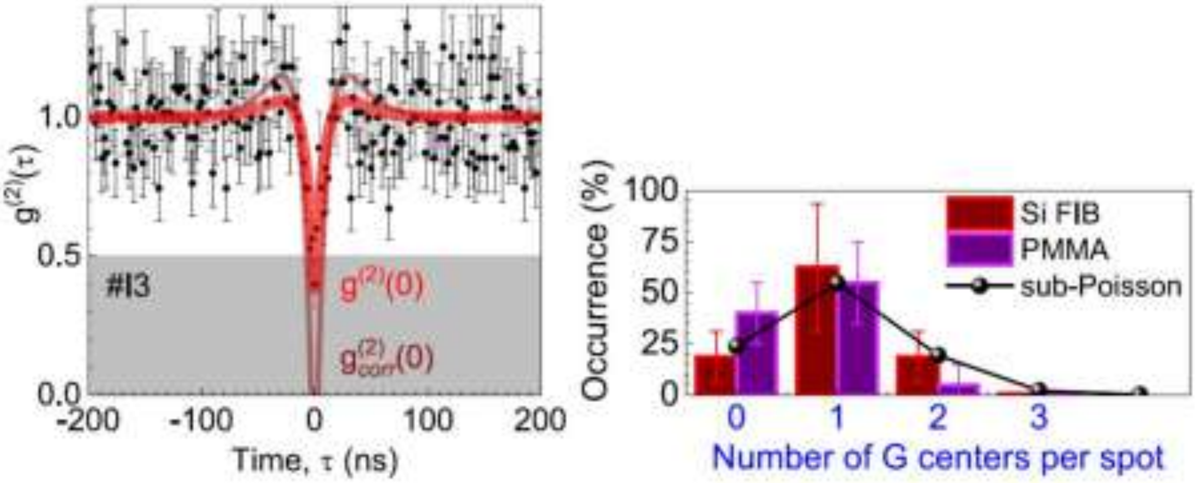

Figure 2. Second-order autocorrelation function $g^{(2)}(\tau)$ obtained with no background correction. The red solid line is a fit, yielding $g^{(2)}(0) = 0.36 \pm 0.06$. The thin solid line is the fit with background correction. The error bars represent standard deviation.

The occurrence probability of G centers for FIB implantation and Si broad-beam implantation (fluence $1 \times 10^{12} cm^{-2}$) through PMMA holes. The solid line represents the sub-Poisson distribution with μ=4.

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OC32: Detection efficiency enhancement for deterministic single ion implantation

K. Stockbridge[1], D. Cox[1], G. Arresta[2], R. Webb[1], S. Clowes[1], and B. Murdin[1]

[1] University of Surrey, GU2 7XH, Guildford, UK
[2] Ionoptika Ltd, SO53 4BZ, Eastleigh, UK
E-Mail: k.stockbridge@surrey.ac.uk

Techniques for deterministic implantation of single ions are currently of high interest for quantum technology 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 expect 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%. Although 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.

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OC33: NanoSQUIDs for SQUID-on-lever scanning probe microscopy

T. Griener[1], S. Koch[1], S. Pfander[1], J. Ullmann[1], C. Bureau-Oxton[2], D. Jetter[3], A. Vervelaki[3], K. Bagani[3], U. Drechsler[2], R. Kleiner[1], M. Poggio[3], A. Knoll[2], and D. Koelle[1]

[1] Physikalisches Institut, Center for Quantum Science (CQ) and LISA+, Universität Tübingen, Germany
[2] IBM Research Europe – Zürich, Rüschlikon, Switzerland
[3]Department of Physics and Swiss Nanoscience Institute, University of Basel, Switzerland
E-Mail: timur.griener@uni-tuebingen.de

Strongly miniaturized nanoscale superconducting quantum interference devices (nanoSQUIDs) can reach ultra-low levels of magnetic flux noise. Together with their small size, this makes them promising candidates for detectors of local sources of magnetic field with high spin sensitivity in scanning probe microscopy. We describe here our attempts to integrate nanoSQUIDs on atomic force microscopy (AFM) Si cantilevers. This approach shall provide ultrahigh-resolution scanning SQUID microscopy (SSM), combined with simultaneous topographic imaging in conventional AFM mode [1]. We will focus on nanoSQUIDs fabricated from single layer Nb thin films, that are patterned by focused ion beams, using Ga, Ne and He. We will discuss the status of the project and challenges that have to be met on the way to combined SSM and AFM imaging.

Acknowledgements

Financial supported by the European Commission under H2020 FET Open grant “FIBsuperProbes” (Grant No. 892427) is gratefully acknowledged.

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OC34: Imaging of the perovskite solar cell comprising nanostructured electron transfer layer

V. Mandić[1], I. Panžić[1,2], and F. Radovanović-Perić[1]

[1] Faculty of Chemical Engineering and Technology, Marulićev trg 20, 10000 Zagreb, Croatia
[2] Materials Center Leoben Forschung GmbH, Roseggerstraße 12, 8700 Leoben, Austria
E-Mail: vmandic@fkit.unizg.hr

Among emerging thin-film solar cells, the highest efficiencies, highest mobilities of charge carriers, tunable band gap, etc. are found in perovskite solar cells (PSCs). PSCs are still not commercial recognized due to susceptibility to both moisture and oxygen, limiting their long-term stability. Many strategies were proposed to address the early degradation of the PSCs, such as configuration modifications, where the importance of various nanostructured electron transfer layer (ETL) became obvious.

In this work we prepared PSCs in n-i-p configuration comprising a rubidium-caesium-methylammonium-formamidinium lead iodide/bromide perovskite absorber, interfaced with nanostructured zincite nanorod or mesostructured titania ETL. We aged the PSCs at ambient conditions or at elevated temperatures using an in-situ setup, comprising synchrotron grazing incidence diffraction and Raman spectroscopy.

The only way to reliably investigate the development of the interfaces was to employ dual beam microscopy for preparing of the lamellas, and to investigate these using scanning transmission electron microscopy (STEM). STEM imaging revealed that the origin of the degradation lay in the zincite/absorber interface. For the case of the nanostructured zincite, the perovskite absorber contained many defects, nullified the advantages initially achieved by nanostructuring, leading to the conclusion that the investigated quadruple perovskite absorber showed limited compatibility with zincite nanorod ETL. Exchanging of zincite nanorod with mesostructured titania improved the stability parameters of the PSCs.

Acknowledgments
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OC35: Gate-controlled critical current in W-C superconducting nanowires grown by FIBID

A. Arroyo-Fructuoso[1], G. Hlawacek[2], and R. Córdoba[1]

[1] Universitat de València (UV), Institute of Molecular Science (ICMol), Catedrátic José Beltrán Martínez, Paterna, Spain
[2] Helmholtz-Zentrum Dresden-Rossendorf (HZDR), Bautzner Landstraße 400, Dresden, Germany
E-Mail: alba.arroyo@uv.es

The gating effect is a term used to define a physical phenomenon which consists of the modulation of the charge carrier density of a material applying an external electric field. This effect has a special relevance at the field-effect transistor (FETs) in the semiconductor industry, particularly, metal oxide semiconductor FETs (MOSFETs). Moreover, recently this interest has been extended to superconductors by the observation of notable changes on their superconducting properties by applying a gate voltage [1–4].

In the present work, the critical current modulation with the applied gate voltage of nanowires with different width values, ranging from 55 nm to 295 nm are presented. The nanowires were prepared through the Focused Ion Beam Induced Deposition technique, using W(CO)6 as precursor molecule. Likewise, to study a more localized effect of the gate voltage, electrodes of different widths and different distances between the electrodes and the nanowire were studied from 164 nm to 2.30 µm and from 80 nm to 590 nm respectively. Dependence resistance with temperature (TC ~ 4.70 K), applied magnetic field (BC2 ~ 9 T), and biased current were studied. The modulation of the critical current of nanowires with the gate voltage was studied by measuring the four-probe resistance of each sample as function of the current at different values of gate voltage and at the superconducting state.

Thanks to the capability of the gate to modulate the critical current, these superconducting nanowires can be extremely attractive for application as switches in superconducting electronic devices due to their high switching speed and low power consumption.

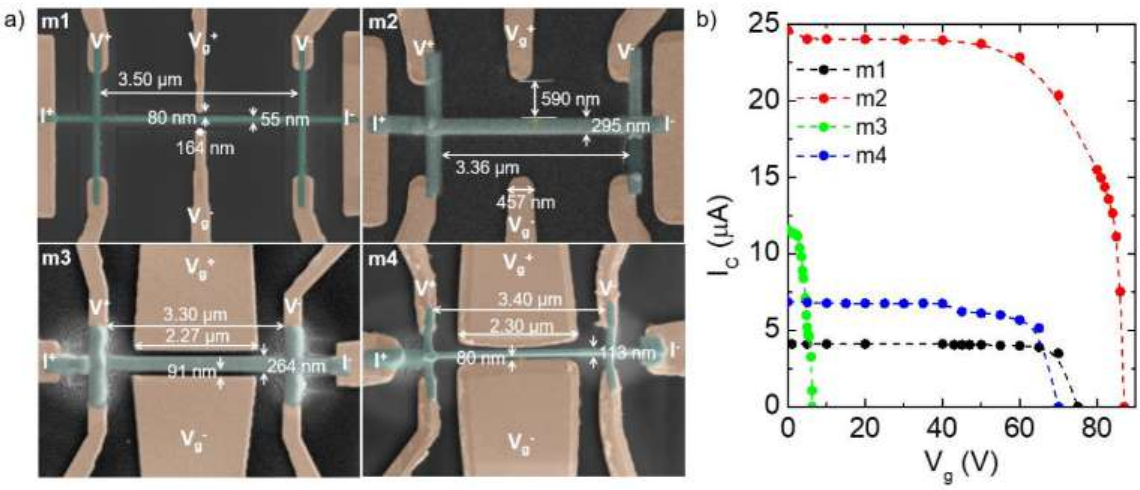

Figure 1. a) Artificially coloured SEM images of different samples (m1, m2, m3, and m4). Colour code: W-C nanowires (green) and Cr/Au contacts and gate electrodes (light brown). b) Modulation of the critical current as a function of the gate voltage at 1.9 K, below the critical temperature of each sample. Dashed lines are a guide for the eye.

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Poster Presentations

fit4nano

17 - 19 July, Lisbon
Faculty of Pharmacy, UL

Workshop 2023

P1: Microstructural characterization of layered Cu-Te structures synthesized by focused ion beam

N. Braun[1], V. Roddatis[2], A. Mill[1], S. Cremer[1], H. Bryja[1], L. Voß[3], S. Sun[4], L. Kienle[3], W. Zhang[4] and A. Lotnyk[1,5,6]

[1] Leibniz Institute of Surface Engineering e.V. (IOM), Leipzig, Germany
[2] GFZ German Research Centre for Geosciences, Potsdam, Germany
[3] Institute for Materials Science, Faculty of Engineering, University of Kiel, Kiel, Germany
[4] Center for Alloy Innovation and Design (CAID), State Key Laboratory for Mechanical Behavior of Materials, Xi'an Jiaotong University, Xi'an 710049, China
[5] Laboratory of Infrared Materials and Devices, The Research Institute of Advanced Technologies, Ningbo University, Ningbo, Zhejiang 315211, China
[6] College of Physics and Optoelectronic Engineering, Harbin Engineering University, Harbin 150001, China
E-Mail: nils.braun@iom-leipzig.de

Specimen preparation for transmission electron microscopy (TEM) using focused ion beam (FIB) is a commonly used method [1]. An advantage over other methods is site specific specimen preparation that only damages a small area of the sample. However, it also suffers from many disadvantages. Artifacts induced by FIB range from ion implantation and resulting amorphization to thermal effects like phase transitions [2,3]. In this work, we prepared different nanoscale Cu-Te phases from Cu (electrodes) - Sb_2Te_3 (thin films) system [4] using FIB method. Since the formation of the Cu-Te phases is highly dependent on settings used during FIB preparation as well as the layer system, it is an interesting model system to study FIB induced artifacts. Copper chalcogenides have shown promising thermoelectric properties, e.g. high efficiency and tunability, which is another motivation for research on these material systems [5].

Sb_2Te_3 thin layers with thicknesses of 17 or 100 nm are epitaxially grown on p-type Si (111) substrates [4]. Samples with a 24 nm thick polycrystalline Sb_2Te_3 thin film are grown on a SiO_2 coated Si(100) wafer using pulsed laser deposition (PLD). Cu, Pt/Cu and Cr layers are deposited by magnetron sputtering on top of the Sb_2Te_3 layers. TEM specimens are prepared at varied beam currents using a standard cross-section FIB preparation method using a Ga FIB [1]. Ion beam parameters are varied and lowered up to 15 kV, 250 pA for the trenching step before lift out. FIB specimens are investigated using advanced methods of aberration-corrected scanning TEM such as atomic-scale HAADF imaging and atomic-scale chemical analysis (EDX and EELS). In situ x-ray diffraction heating of the Cu-Sb_2Te_3 thin film is performed to confirm structural changes. To evaluate the viability of observed structures theoretical calculations are being done. Dependent on beam current used during FIB lamella preparation and Sb_2Te_3 layer thickness, hole formation in the Cu layer, thickness change and chemical changes of the Sb_2Te_3 layers are observed (Fig. 1). Layer thickness after FIB specimen prepared at normal milling parameters are 28 nm and 23 nm for reduced parameters in a sample with originally 17 nm Sb_2Te_3. Hole formation is also decreased indicating less material transport. The structural changes are confirmed by in situ x-ray diffraction heating in addition. Samples with a 17 nm Sb_2Te_3 layer showed uniform intercalation of Cu while a sample with 100 nm thick Sb_2Te_3 layer exhibited differential intercalation behavior. In specimen prepared from the in situ heated samples Sb_2Te_3 and Cu-Te grains are observed. The introduction of a Pt layer between the Cu electrode and Sb_2Te_3 layers hindered structural changes caused by FIB. The same effect is observed in the heated sample where a Sb layer was formed between the Cu and Sb_2Te_3 layer (Fig. 2).

Moreover, Cr-Sb_2Te_3 and a Cu-GeTe layer systems showed no modifications of Sb_2Te_3 and GeTe thin films during FIB preparation. In polycrystalline Sb_2Te_3 specimen the intercalation of Cu and formation of new Cu-Te phases was also observed. Here the formation of a tetragonal rickardite Cu_2Te phase is found.

This work shows that structural changes in Sb_2Te_3 thin film systems can be induced during FIB specimen preparation of Cu (electrode)-Sb_2Te_3 (thin films) systems. The influence of different factors such as ion beam current, layer stacking sequence, Sb_2Te_3 structure and layer thickness on the formation of Cu-Te phases are evaluated. The changes are thermally induced transitions caused by FIB process, while re-deposition of Cu plays a minor role. Similar thermal effects can be observed in heated samples. Other studied systems show no modifications.

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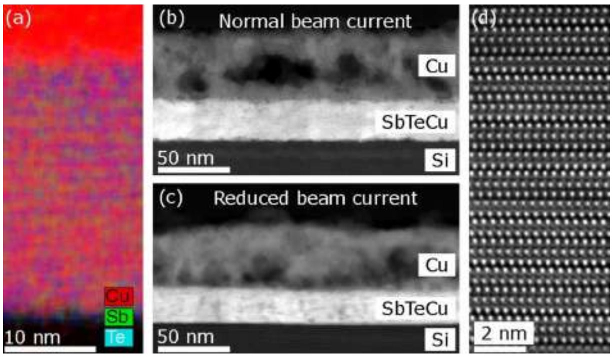

Figure 1. (a) EDX Map of a lamella prepared with FIB, (b-c) Overview HAADF-STEM images of specimen prepared with normal and reduced FIB beam currents. (d) Atomic-resolution HAADF-STEM image showing new Cu-Te phases.

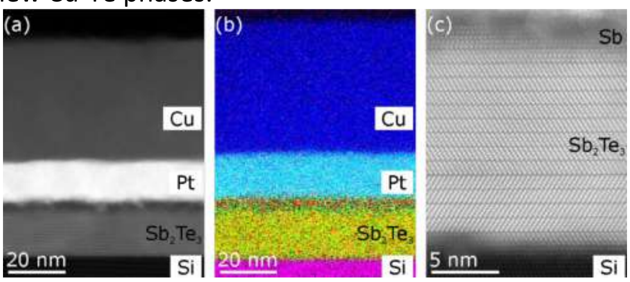

Figure 2. Cu/Pt/Sb_2Te_3 layer stack. (a) Overview HAADF-STEM image. (b) Overview EDX elemental map. (c) Atomic-resolution HAADF-STEM image, showing initial Sb_2Te_3 structure.

P2: FIB-SEM tomography - tool for determining the oxidation mechanism of Sanicro 25 steel at a temperature of 700°C

G. Cempura, and A. Kruk

Faculty of Metals Engineering and Industrial Computer Science, AGH University of Science and Technology
31-423 Kraków, Poland
E-Mail: cempura@agh.edu.pl

Sanicro 25 is a high-temperature austenitic stainless steel developed by Sandvik. It offers exceptional creep resistance, along with high resistance to corrosion, oxidation, and stress corrosion cracking. It finds wide applications in heat exchangers, boilers, and superheaters in the chemical and power generation industries.
The oxide scale and near-surface area of the samples, subjected to 40,000 hours of oxidation, were characterized in three dimensions using FIB-SEM tomography. The tomographic reconstruction data was collected using the slice-and-view technique on the ZEISS CrossBeam 350 microscope. Scanning electron microscope (SEM) images were acquired using both secondary electron (SE) and backscattered electron (BSE) detection methods. The acquired datasets were processed, reconstructed, and visualized in 3D using Fiji and Avizo software.
Obtained results allowed for determination of microstructural changes close to the oxidized surface and allows for understanding of the oxidation process. The FIB-SEM tomography investigations were supported by phase analysis using advanced transmission electron microscopy investigations.

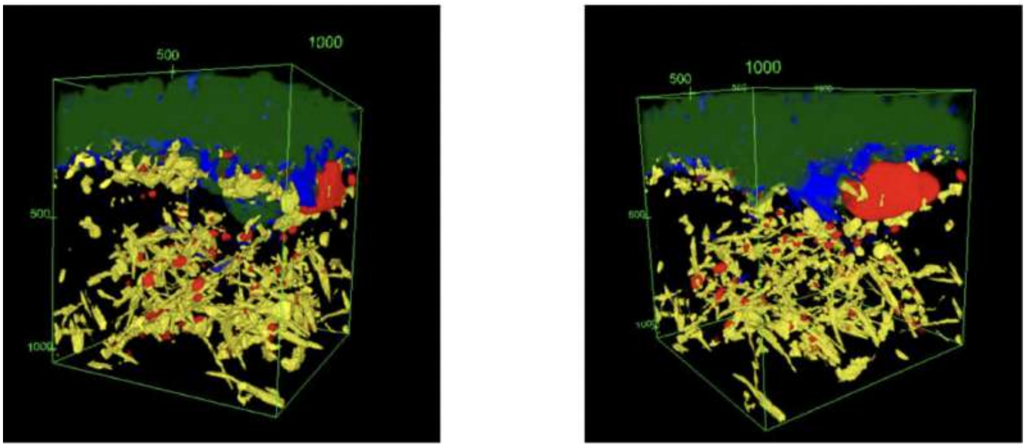

Figure 1. Visualization of the investigated volume, oxide scale at the top and several precipitates.

Acknowledgements
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The authors express their gratitude to Pragya Shekhar, Andreas Schertel, Volker Pusch, and Michał Reimus from ZEISS for their invaluable support during the investigations.

P3: Structural and compositional insights into biological and beam sensitive samples by using a cryo-FIB instrument equipped with three complementary detection modalities

O. De Castro[1], T. Taubitz[1], A. Biesemeier[1], and T. Wirtz[1]

[1] Department of Materials Research and Technology, Advanced Instrumentation for Nano-Analytics, Luxembourg Institute of Science and Technology, Belvaux, Luxembourg
E-Mail: olivier.decastro@list.lu

For studying hybrid samples of organic-inorganic phases, beam sensitive samples and especially the close to native state of life science samples, the technique of electron microscopy under cryo-conditions is well-established. Imaging of samples' surfaces i.e. by scanning electron microscopy (SEM), as well as obtaining 3D volume information by using transmission electron microscopy (TEM) are both approaches routinely incorporated in workflows in order to get structural, and to some extent compositional, data from the samples under inspection [1]. These imaging techniques require investigation chambers operating at high vacuum/ultra-high vacuum atmosphere. To guarantee vacuum compatibility, introducing biological samples in frozen-hydrated condition is beneficial to circumvent the classical way of room temperature (RT) sample preparation, i.e. by chemical fixation known to be a source of undesired artifacts. These beneficial aspects of cryo-EM can be passed on to ion beam induced imaging techniques.
Starting from this context, the development of a cryo-focused ion beam (FIB) instrument has been triggered. It incorporates several investigation modes, namely ion induced secondary electron (SE) im aging, secondary ion mass spectrometry (SIMS) capability and scanning transmission helium ion microscopy (STIM) detection [2]. The instrument makes use of the Gas Field Ion Source (GFIS), which, due to its ultra-high brightness [3], allows to investigate the samples with very finely focused He+ and Ne+ primary ion beams. Sub-nanometer spatial resolution can be obtained when working in SE mode while using the He+ion beam, combined with an excellent contrast and a high depth of field. Moreover, the installed magnetic sector SIMS system provides the capability of extracting sub-15 nm laterally resolved chemical information at high sensitivity, preferentially using the Ne+ beam [4]. Equipped with a continuous focal plane detector, based on a micro channel plate (MCP) - delay line (DL) technology, the SIMS system provides for each irradiated pixel on the sample surface the complete mass spectrum. In addition to these two modes probing the (near)-surface region, the full thickness of samples is accessible through the STIM detection mode. In this way, using a He+ion beam of few tens of keV, thin (about 100 nm thickness) samples undergo STIM investigations at nanoscale [5]. The central feature of the cryo-capability of the FIB instrument, is a high precision 5-axis cryo-stage paired with sample transfer features under cryo-conditions. By combining this cryo-FIB platform with other tools for cryo compatible sample preparation, including a glovebox operating at low humidity nitrogen atmosphere, dedicated workflows can be conceived that are ideal for investigating biological as well as beam sensitive material science samples (e.g. polymer-based samples or batteries) in in-situ correlative manner.
Here, we are presenting proof-of-concept studies performed with beam sensitive samples, such as organic-inorganic hybrid samples, which were investigated both at RT and at cryo-temperature (< -140 °C) (see Figure 1 as an example), demonstrating the benefits of working at low temperature as well as the functionality of all detection modes under cryogenic sample conditions [6].

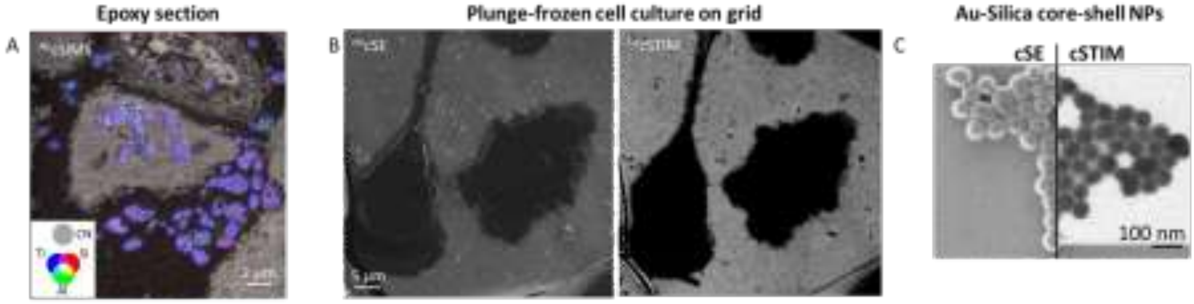

Figure 1. Proof of concept investigation of beam-sensitive samples under cryo conditions. A) Correlative SE-SIMS at -147 °C (false color representation of epoxy cross section of keratinocyte cell culture exposed to Si-Al-TiO_2 nanoparticles (NPs)). B) Investigation of frozen-hydrated A549 cell culture (after plunge freezing and cryotransfer; -147 °C). C) Au-Silica core-shell NPs at -140 °C. cSE, cSTIM, cSIMS: SE, STIM and SIMS performed under cryo conditions (down to -147 °C).

Acknowledgements
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P4: Growth and applications of FIBID superconducting deposits and of FEBID magnetic tips

F. Sigloch[1], A. Sáenz[1], A.T. Escalante-Quiceno[1], S. Sangiao[1,2], C. Magén[1,2], and J. M. De Teresa[1,2]

[1] Instituto de Nanociencia y Materiales de Aragón (INMA), CSIC-Universidad de Zaragoza, 50009 Zaragoza, Spain.
[2] Laboratorio de Microscopías Avanzadas (LMA), Universidad de Zaragoza, 50018 Zaragoza, Spain.
E-Mail: deteresa@unizar.es

Thanks to Focused Ion Beam Induced Deposition (FIBID) and Focused Electron Beam Induced Deposition (FEBID), it is possible to grow 2D and 3D high-resolution nanostructures, with applications in various fields such as nanoelectronics, plasmonics, magnetism, superconductivity, sensing, etc. In this contribution, we will show two examples carried out in our group that illustrate the potential of FIBID and FEBID for the growth of high-resolution superconducting and magnetic nanostructures, respectively.
The first example corresponds to the growth of nanoSQUIDs on Si substrates. FIBID is known to be a very useful technique to grow 2D and 3D superconducting nanostructures [1]. In our case, we have used Ga+-FIBID in combination with the W(CO)6 precursor to grow nanoSQUIDs on flat Si-based substrates [2]. This kind of direct-write lithography technique is promising for niche applications in the field of quantum technologies [3].
The second example corresponds to the growth of magnetic tips, which are useful not only for high resolution magnetic force microscopy with tailored strayed magnetic fields [4], but also for other techniques such as magnetic resonance force microscopy, nanowire magnetic force sensing, etc. [5]. Moreover, we have recently demonstrated that Fe-based magnetic tips grown by FEBID are robust and do not degrade with time in magnetic force microscopy measurements [6].

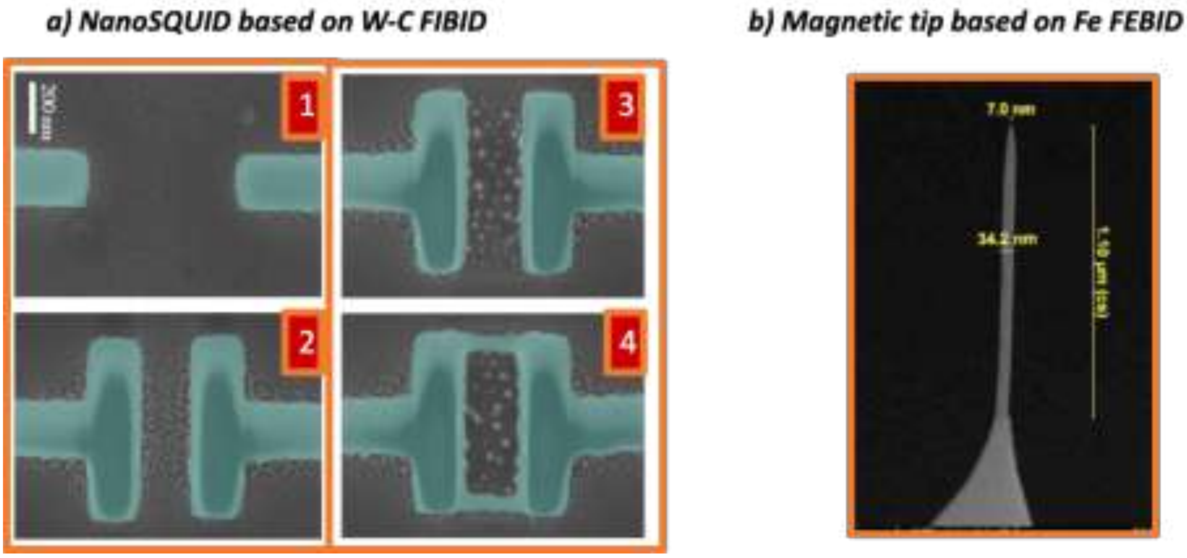

Figure 1. a) W-C FIBID nanoSQUID grown on a flat Si//SiO_2 substrate; **b)** Fe FEBID tip for magnetic force microscopy.

Acknowledgements
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P5: Compatibility Analysis of Focused Ion Beam Induced Deposition of Cobalt-based Deposits under Cryogenic Conditions onto Different Substrates

A.T. Escalante-Quiceno[1], A. Salvador-Porroche[1,2], S. Sangiao[1,2], M. Barrado[2], P. Cea[1,2,3], C. Magén[1,2], and J.M. De Teresa[1,2]

[1] Instituto de Nanociencia y Materiales de Aragón (INMA), CSIC-Universidad de Zaragoza, 50009 Zaragoza, Spain.
[2] Laboratorio de Microscopías Avanzadas (LMA), Universidad de Zaragoza, 50018 Zaragoza, Spain.
[3] Departamento de Química Física, Facultad de Ciencias, Universidad de Zaragoza, 50009 Zaragoza, Spain.
E-Mail: aescalante@unizar.es

Focused Ion Beam Induced Deposition (FIBID) is a direct-write, resist-free nanolithography technique that enables the growth of high-resolution nano- and micro-structures. The deposition process involves a gas precursor which interacts with the ions, dissociating the gas molecules to produce the deposition of solid material on the substrate. Nevertheless, the dissociation process of the molecules relies on the continuous adsorption and diffusion of the gas molecules in the substrate surface, which makes FIBID growth a slow process compared with other techniques. Furthermore, the long exposure time to the ion beam irradiation leads to side effects such as implantation, amorphization and milling. To overcome these issues, a possible solution is to decrease the temperature of the substrate during irradiation below the condensation temperature of the precursor gas (-100°C), a technique called cryo FIBID. In this approach, when the precursor gas is injected, a condensed precursor layer is created (Fig. 1a), and then irradiated with the ion beam to produce the decomposition of the molecules (Fig. 1b). Finally, when the sample is heated up to room temperature, causing the evaporation of the unexposed condensed precursor, and the irradiated pattern remains as the final deposited structure (Fig. 1c).
The development of cryo-FIBID has allowed the growth of W-C [1], Pt-C [2], and Co [3] nanostructures at -100°C with liquid nitrogen as a cooling agent, as well as cryo-deposition of W-C using a thermoelectric plate at -60°C [4]. Cryo-FIBID has a significantly higher efficiency than room-temperature FIBID in terms of resolution, irradiation dose and processing speed [5]. It also provides deposits with high metal content and low resistivity, suitable for circuit editing, electrical contacts, and mask repair applications. We present here a compatibility analysis of Co deposits grown by cryo-FIBID on a series of technologically relevant substrates, such as Si, SiO_2, Au, Al, graphene, $LaAlO_3$ and $SrTiO_3$, using different irradiation doses from 5 to 55 $\mu C \cdot cm^{-2}$. We also analyzed the resolution limit of Co-based deposits grown by cryo-FIBID, where we found that thin lines of up to 20 nm thickness and width can be deposited. These results open up the possibilities for the use of cryo-FIBID Co deposition for different applications.

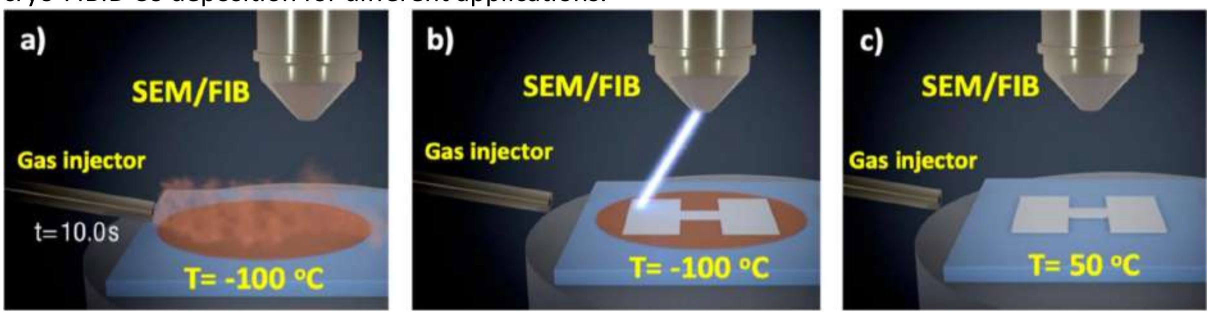

Figure 1. Schematic images of the cryo-FIBID process. a) Formation of the condensed layer on the substrate. b) Irradiation pattern on the condensed layer. c) Heating of the substrate causing the evaporation of the unexposed condensed precursor, revealing the deposited structure. Adapted and reprinted from De Teresa et al., Micromachines 2019 [5].

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P6: Revealing Plasma focused ion beam (O-PFIB) surface interactions on polypropylene using Secondary Electron Hyperspectral Imaging

N.T.H. Farr[1,2], A. de Marco[3,4], and C. Rodenburg[1,2]

[1] Department of Materials Science and Engineering, Sir Robert Hadfield Building, Mappin Street, University of Sheffield, Sheffield S1 3JD, UK
[2] Insigneo Institute for in silico Medicine, The Pam Liversidge Building, Sir RobertHadfeld Building, Mappin Street, Sheffield, UK
[3] Biomedicine Discovery Institute, Department of Biochemistry and Molecular Biology, Monash University, Melbourne, VIC, Australia
[4] ARC Centre of Excellence in Advanced Molecular Imaging, Monash University, Melbourne, VIC, Australia
E-Mail: n.t.farr@sheffield.ac.uk

Background: The development of Focused Ion Beam – Scanning Electron Microscopy (FIB-SEM) systems have provided significant advances in the processing and characterization of polymers [1]. Plasma-FIB (PFIB) and FIB systems are routinely used to prepare polymer specimens for SEM or TEM analysis, creating thin slices, cross-sections and polished surfaces. For these examples there is a requirement that the finished material surface is clean, smooth and undamaged by the FIB process. The Oxygen Plasma FIB (O-PFIB) process has been developed to create fine cross-sections, which can then be imaged by the electron beam or other techniques (such as cellular FIB-SEM tomography). Despite FIB-SEM being routinely applied for micro-structural analysis of polymers, a fundamental understanding of ion-sample interaction is still not fully established. To progress in this field in-situ analysis of carbon bonding within the SEM chamber (thus without the need for an Ultra High Vacuum (UHV) that allows for multiscale scale surface sensitive analysis is required. Additionally, such in-situ analysis should be carried out at low beam energies to reducing the prospect of sample modification. All these requirements are satisfied via the use of Secondary Electron Hyperspectral Imaging (SEHI) and the accompanying application of Secondary Electron spectroscopy (SES) [2-3]. Data presented in the study provides evidence of SEHI's ability to characterize polymer surfaces post PFIB etching. This study highlights that SEHI can reveal oxygen plasma FIB interactions on the surface of polymers and can also monitor serial FIB surfaces in-situ.
Results: To better understand the effect that O-PFIB has on the surface of PP, three different O-PFIB etch conditions were applied to cryo-faced and gold coated polypropylene (PP). The resulting SEHI derived secondary electron specta (SES) are presented in Figure 1A/B with accompanying SE images presented in Figure 1 C-E. It can be confirmed that all O-PFIB conditions applied resulted in spectra showing the effective removal of the gold coating from the sample. Observable in Figure 1A is a notable variation of the resulting spectra for different etching condition, and also for the when the same etching conditions are applied to different regions of the specimen. Figure 1B further indicates this variation within SE peaks is in the range 3 – 4.5 eV formed from sp2/CHx. The largest variation in spectra collected from different regions is apparent after O2 2 KeV 3.8 nA (conditions commonly applied as a polishing step). From the SEHI data provided, this variation indicates polymer chain fragmentation and variation within the surface cross-linking density [4]. Such variation appears consistent with SE images provided (Fig 1C-E) which show large morphological surface variation for O2 2 KeV 3.8 nA compared to that of other conditions.

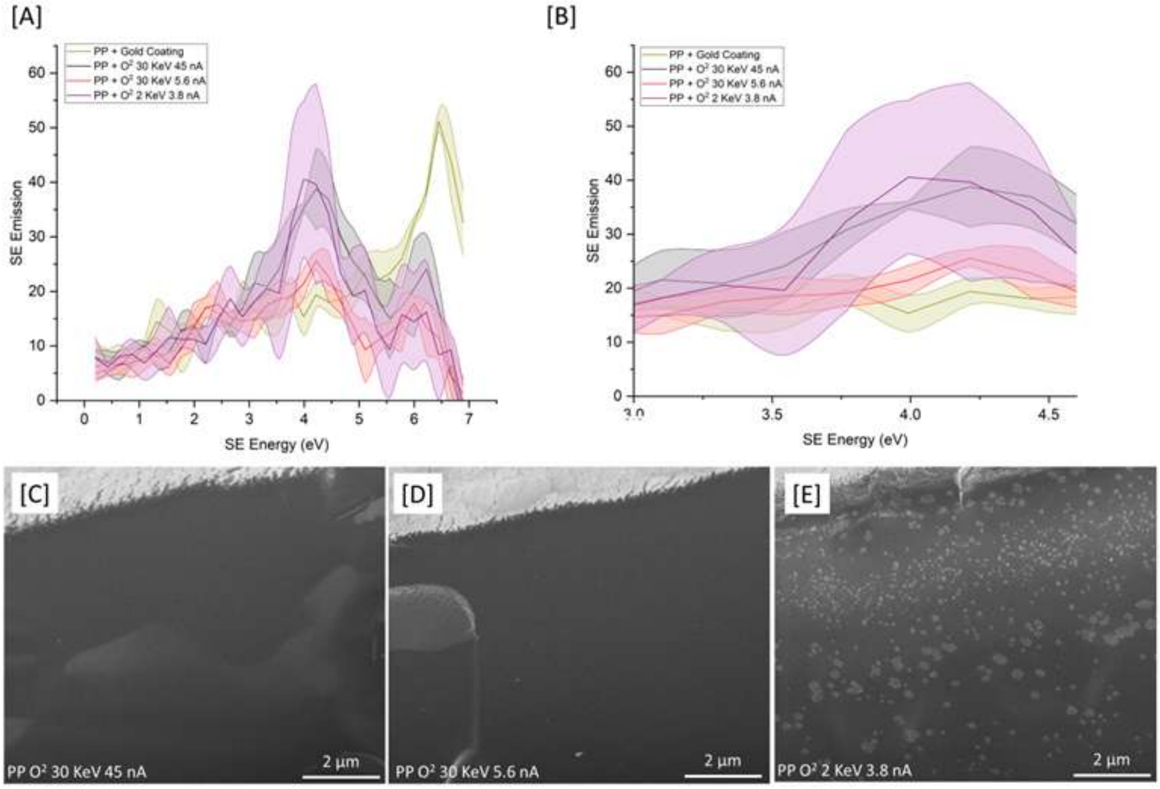

Figure 1. (**A**) SES of cryo-faced and aged PP + Gold Coating (n=3), PP + O2 30 KeV 45 nA (n=3), PP + O_2 30 KeV 5.6 nA (n=3), PP + O_2 2 KeV 3.8 nA (n=3). (**B**) SES of samples highlighted in (A) plotted from 3 eV to 4.5 eV. (**C-E**) SE images of PP post O-PFIB etch.

Conclusion: The data provided in this study provides evidence of SEHI's ability to be a valuable tool in the characterisation of polymer surfaces post PFIB etching. This study highlights that SEHI can reveal oxygen plasma FIB interactions on the surface of polymers and can also monitor serial FIB surfaces in-situ.

Acknowledgments
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P7: The stability of the bistable carbon defect under proton irradiation

T. Fernández Bouvier[1], V. Jantunen[1], and F. Djurabekova[1]

1 Department of Physics, University of Helsinki, P.O. Box 43, FI-00014, Finland
E-Mail: tomasfbouvier@gmail.com

Bistable carbon defects (also known as G centers) have recently acquired significant importance due to their implication in the manufacture and enhancement of optoelectronic devices. They can be found in different configurations which ultimately will determine their optical properties. G centers can be synthesized by irradiation of carbon doped silicon with MeV protons.
While the quantum properties of the latter centers and their formation mechanisms have been studied widely, only few investigations have been carried on the survival of those centers with time or their eventual annihilation under such irradiation conditions. In this work we address this gap.
In order to explore the different breaking mechanisms and stable configurations of the defect we have built a free energy surface map using enhanced sampling (metadynamics). We have obtained the most relevant energy barriers using nudge elastic band. Finally we have performed molecular dynamics simulations of primary recoilsions target to the center and surroundings of the system.
Overall we find high energy barriers for the breaking of the center. Irradiation simulations confirm that by showing that only direct impact in this energy attempt to break the defect whereas nearby impacts mostly leave them intact.

P8: Morphological characterization of gold nanoparticles as versatile tools for photothermal therapy

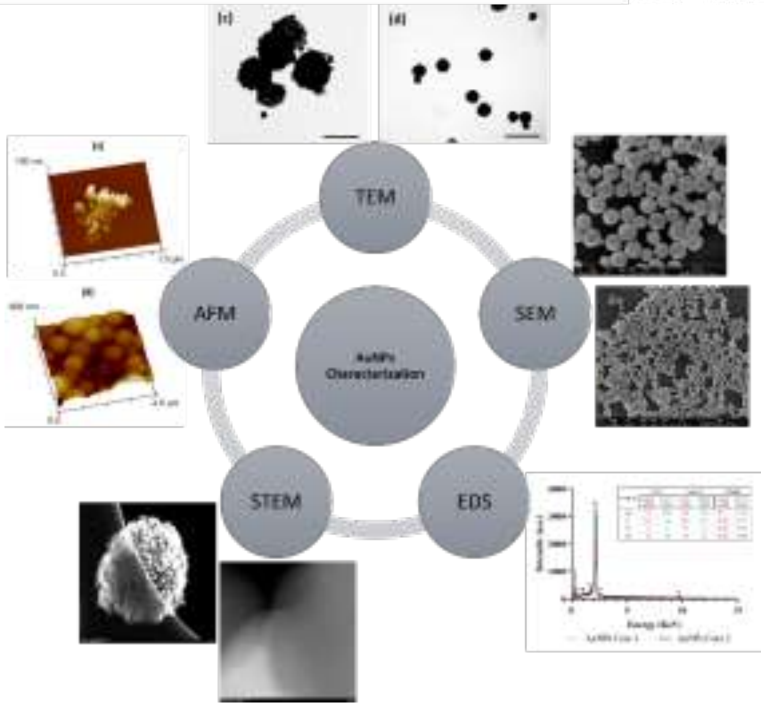
T. Ferreira-Gonçalves[1,2], A. S. Viana[3], L. Ascensão[4], P. Ferreira-Santos[5,6], D. Nunes[7], E. Fortunato[7], R. Martins[7], M. M. Gaspar[2], D. Ferreira[8], H. A. Ferreira[2], and C. P. Reis[1,2]

[1] Research Institute for Medicines (iMed.ULisboa), Faculty of Pharmacy, Universidade de Lisboa, Av. Professor Gama Pinto, 1649-003 Lisboa, Portugal
[2] Instituto de Biofísica e Engenharia Biomédica, Faculdade de Ciências, Universidade de Lisboa, Campo Grande, 1749-016 Lisboa, Portugal
[3] Centro de Química Estrutural, Faculdade de Ciências, Universidade de Lisboa, Campo Grande, 1749-016 Lisboa, Portugal
[4] Centro de Estudos do Ambiente e do Mar Lisboa (CESAM Lisboa), Faculdade de Ciências, Universidade de Lisboa, Campo Grande, 1749-016 Lisboa, Portugal
[5] Department of Chemical Engineering, Faculty of Science, University of Vigo, As Lagoas s/n, 32004 Ourense, Spain
[6] Centre of Biological Engineering, Universidade do Minho, Campus de Gualtar, 4710-057 Braga, Portugal
[7] Department of Materials Science, NOVA School of Science and Technology, Campus de Caparica, i3N/CENIMAT, 2829-516 Caparica, Portugal
[8] Comprehensive Health Research Centre (CHRC), Departamento de Medicina Veterinária, Escola de Ciências e Tecnologia, Universidade de Évora, Pólo da Mitra, Ap. 94, 7002-594 Valverde, Évora, Portugal
E-Mail: taniag1@edu.ulisboa.pt

Gold-based materials have been used by the humanity for centuries in different areas. More recently, they have also been applied in biomedicine [1]. Clear differences were seen between gold materials at macroscale and nanoscale. In particular, gold-based nanoparticles (AuNPs), have been attracting special attention. Depending on the synthetic method used, AuNPs with variable shapes, sizes and surface properties can be attained. Those intrinsic features are known to result in variable physicochemical and optical properties which make them versatile tools for several biomedical applications. One particular application in which AuNPs can be key elements is photothermal therapy (PTT), a minimally invasive therapy. The efficacy of PTT depends greatly on how deep the radiation can reach and on the amount of heat generated. AuNPs have the exquisite ability to convert light energy into heat and for this reason, the combination of AuNPs with radiation within the optical therapeutic window range (650 to 900 nm) constitutes a potentially powerful strategy to enhance PTT efficacy and outcome [2].

Herein, the main physicochemical and morphological properties of AuNPs prepared using modified versions of a recently developed synthetic method reported by this research group, in which commonly used cytotoxic reagents were replaced by more biocompatible and environmentally friendly options, such as ascorbic acid and rosmarinic acid [3]. A primary characterization of the AuNPs was carried out by dynamic light scattering (DLS) and transmission electron and atomic force microscopies (TEM and AFM, respectively). A predominance of spherical-like AuNPs with sizes ranging between 100 and 570 nm was observed depending on the stoichiometry used on the syntheses, even though populations of larger planar structures were also identified. Moreover, the microscopical analyses evidenced some surface roughness of the AuNPs. Later on, the particles were also observed by scanning electron microscopy (SEM) and dispersive X-ray spectroscopy (EDS), which confirmed the morphology of the AuNPs previously observed and showed the predominant abundance of the Au element on the AuNPs constitution. At the last, the AuNPs were analyzed by scanning transmission electron microscopy (STEM) to further provide some insight about the Au atoms conformation, having shown a face centered cubic (FCC) structure.

This extensive characterization was crucial to select the most suitable type of particles for future application.



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P9: Correlative SEM/AFM Microscopy - Combining Two High-Performance Methods for Nanoscale Measurements

H. Frerichs[1], S. Seibert[1], L. Stühn[1], M. Wolff[1], D. Jangid[1], and C.H. Schwalb[1]

[1] Quantum Design Microscopy GmbH, DE-64293 Darmstadt, Germany
E-Mail: frerichs@qd-microscopy.com

The combination of different analytical methods for a qualitative and quantitative evaluation of material properties is nowadays an important part of the development of characterization methods. Especially the correlative microscopy represents an important technique to analyze materials with spatial resolution. In this context, scanning electron microscopy (SEM) and atomic force microscopy (AFM) are powerful tools to study even the smallest features of a sample with high resolution. However, combining these methods is not easy and remains challenging regarding the instrument setups required. In most cases, both methods are used separately and the obtained results cannot be truly spatially correlated.

In this contribution, we will demonstrate that a correlative in-situ analysis strategy using a combination of SEM and AFM is possible and that it can provide new ways to study material characteristics with nanometer resolution [1-2]. In addition to the possibility of adding true height information to SEM data, the enhanced capabilities of a probe-based method like AFM are particularly beneficial for imaging material properties that would be otherwise not "visible". In this context, we will present a series of investigations that demonstrate the benefits of an interactive, correlative in-situ characterization method for various materials and nanostructures. Results from the in-situ characterization of grain boundaries using electrostatic force microscopy (EFM) [3], measurements of mechanical properties of thin films using mechanical measurements, and the analyses of multilayer and steel structures using magnetic force microscopy (MFM) will provide insights into the correlative analysis approach. In order to measure special material characteristics, the cantilevers and their tips must provide special properties. It is therefore beneficial to use advanced tip fabrication techniques like the focused electron beam induced deposition (FEBID) to fine-tune the tip properties [4]. We would therefore like to highlight some results regarding the use of advanced cantilever probes, used to perform the advanced AFM measurements described here.

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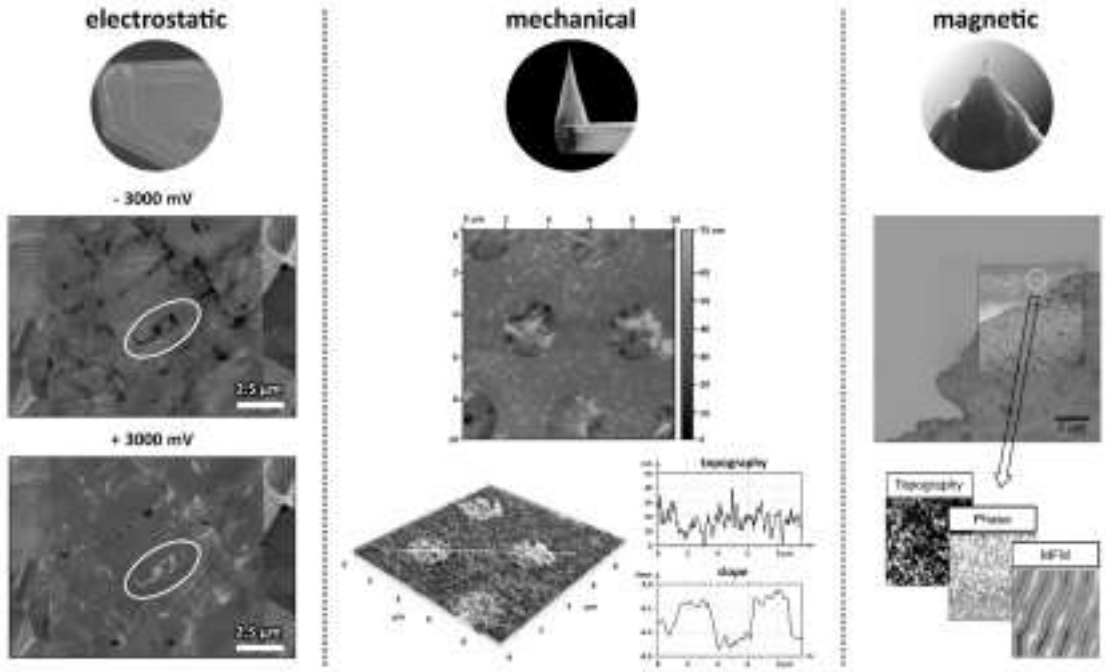

Figure 1. Overview of the different measurements related to electrostatic (EFM), mechanical and magnetic (MFM) measurements using correlative microscopy (SEM/AFM).

P10: The formulation and hemolytic assessment of biomimetic polysaccharide electrospun nanofibers

A.-T. Iacob[1], O.M. Ionescu[1], M. Dragan[1], F. Lupascu[1], M. Apotrosoaei[1], I. Vasincu[1], and L. Profire[1]

[1] Department of Pharmaceutical Chemistry, University of Medicine and Pharmacy, 700115, Iasi, Romania
E-Mail: panzariu.andreea.teodora@gmail.com

The aim of this paper is the formulation of novel electrospun nanofibers based on hyaluronic acid and polyethylene oxide (HA/PEO) incorporating as active substances: propolis (P), insulin (I) and infusion of *Calendula officinalis flos* (C) as new dressing materials in the treatment of wounds.
The preparation of HA/PEO matrices was carried out in three stages: (i) the dissolving of HA and PEO in physiological brine by stirring at room temperature (rt), then, (ii) the active substances were added and stirred until a homogeneous solution is obtained [1]; for the last step (iii) an INOVENSO nanospinner was used and then different values of flow-rate, applied voltage and also different distances from the tip of the syringe to the collecting plate were applied [2]. The 3 nanofibrous matrices developed were: HA/PEO/P (A); HA/PEO/PI (B); HA/PEO-PC (C). After the formulation was optimized the polymeric formulations were subjected to the evaluation of hemolytic effect by the capacity of human erythrocyte membrane stabilization on hypotonicity-induced lysis [2].
All the formulations had a hemolytic index below 4%, which demonstrates a good bio-compatibility for the wound dressings designed.
Conclusions: After analyzing the data obtained, it was concluded that nanofibers with propolis and Calendula officinalis flos infusion obtained the smallest hemolytic index of 2.8%.

Acknowledgments
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P11: A overview on the biological effects of hyaluronic acid nanofibers for wound healing applications

O.M. Ionescu[1], A. Iacob[1], F. Lupascu[1], M. Apotrosoaie[1], I. Vasincu[1], M. Dragan[2], and L. Profire[1]

[1] Department of Pharmaceutical Chemistry, University of Medicine and Pharmacy Gr. T. Popa, 700115, Iasi, Ro mania
[2] Department of Medicine industry and Pharmaceutical biotechnologies, University of Medicine and Pharmacy Gr. T. Popa, 700115, Iasi, Romania
E-Mail: oana-maria.ionescu@umfiasi.ro

Electrospinning approach has grown to be widely used for processing biomaterials and serves a variety of applications. The purpose of this paper is to investigate the biological effects of hyaluronic acid nanofibers and optimum electrospinning conditions.
Hyaluronic acid and polyethylene oxide were used in ratio that permits a final solution concentration of 13% (wt/v). By using distilled water there was no need for additional steps to remove any harmful solvent residue. The yield rose with a flow rate of up to 0.7 mL/min by adding active substances (manuka honey, L-arginine, propolis, and Calendula off. extract) to the solution and maintaining a relative humidity over 20%. Microscope images (LEICA) and SEM micrographs were taken to demonstrate nanosize. According to the guidelines and protocol approved by the Ethics commission, the nanofibrous mats with the best characteristics were tested on a surgically excised acute wound model on 6–8-week-old Wistar male rats over the course of 18 days (dressing changes were made every three days).
The rise in electric charges, which could lead to the fiber stretching under the electromagnetic field, may be the reason why the diameter of the fibers did not surpass 300 nm (Fi bermetric), only matching this number if the active component was also included. The test groups presented superior effects compared to the positive control group.
The goal of the approach is additionally aided by the synergistic interaction between the polymer matrix and active component. The dressing may be useful for further research in this area given the low cost of the active chemicals in comparison to the positive control.

Acknowledgements
This work was funded by the following projects: PN-III-P4-ID-PCE-2020-2687, Contract no. 244/2021 and Net4Science, cod SMIS: 154722, contract No. 24867 from 28/10//2022, pro iectul PN-III-P1-1.1-MC-2017-2462.

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P12: Towards hybrid spin-mechanical systems in silicon carbide with helium ion implantation

N. S. Jagtap[1, 2], Y. Klaß[3], F. David[3], P. Bredol[3], E. Weig[3], M. Helm[1, 2], G. V. Astakhov[1], and A. Erbe[1, 2]

[1]Institute of Ion Beam Physics and Materials Research, Helmholtz-Zentrum Dresden - Rossendorf, 01328 Dresden, Germany
[2]Dresden University of Technology, 01062 Dresden, Germany
[3]Chair of Nano and Quantum Sensors, Technical University of Munich, 85748 Munich, Germany
E-Mail: n.jagtap@hzdr.de

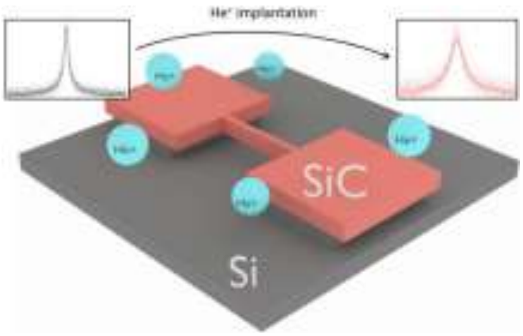

Figure 1. He$^+$implantation scheme on resonators in 3C-SiC (insets show change in the resonance spectrum).

Silicon carbide (SiC) is a suitable candidate for studying hybrid spin-mechanical systems due to its established use as a sensor material [1] and the capability of its hexagonal polytype (4H-SiC) to host highly coherent spin-centers [2], such as silicon vacancies (V_{Si}). To realize such a system, spin resonances associated with V_{Si} need to be coupled to the mechanical modes, which will allow more sensitive magnetic field sensing [3]. To achieve this, we will fabricate mechanical resonators in 4H-SiC and create V_{Si} by helium ion implantation.

To study the influence of created V_{Si} on the mechanical properties, we first considered a system with 3C-SiC (grown on Si) as shown in the figure, which provides higher-quality mechanical resonators compared to 4H-SiC grown on 4H-SiC. In our preliminary experiments, we implanted the resonators with broad beam He$^+$implantation to create ensembles of V_{Si}. We intend to show how the mechanical properties can be modified varying fluence, in terms of resonance frequencies, mechanical quality factors and, stress. In future, we plan to use focused He$^+$ implantation to study the positional dependence and number of V_{Si} on the modification of the material. Finally, we will employ an Optically Detected Spin-Mechanical Resonance (ODSMR) scheme to characterize the coupling of spins and phonons [3].

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P13: Combining binary collision approximation with molecular dynamics for more accurate radiation damage modeling

V. Jantunen[1], T. Bouvier[1], and F. Djurabekova[1]

[1] Department of Physics, University of Helsinki, 00014 University of Helsinki, Helsinki, Finland
E-Mail: ville.jantunen@helsinki.fi

Binary collision approximation (BCA) is a widely used simulation technique for predicting damage from focused ion beams. Popular codes such as SRIM [1] have been available for a long time and have been widely used by theorists and experimentalists alike. However, BCA can not be accurately used in the lower energy collisions (sub 1 keV) from e.g. from secondary recoils where many-body effects become important. This is why molecular dynamics (MD) is often used as it describes the many-body effects that are important in lower energies. This however, comes with a computational cost, and calculating the full damage from high fluence of highly energetic ions can become out of scope for MD. This is why the combination of these methods is better suited for the accurate prediction of total damage.

In this presentation, we present one approach of combining BCA with MD by collecting recoils up to certain energies in BCA and combining this with MD to get the total damage. Using MD we study damage overlap and the annealing of previous damage from new cascades. We also take a look into cascade overlap within individual large cascades. We also discuss the relevance of the crystal structure and irradiation direction for cascade shape and distribution of defects.

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P14: *In-situ* monitoring of focused electron beam induced deposition with platinum-trimethylcyclopentadienylmethyl using mass spectrometer method

C.S. Jureddy[1], J. Jurczyk[2], L. Pillatsch[3], J. Whitby[3], J. Michler[1], P. Hoffmann[4], and I. Utke[1*]

[1] Laboratory for Mechanics of Materials and Nanostructures, Empa-Swiss Federal Laboratories for Materials Science and Technology, Feuerwerkerstrasse 39, CH-3602 Thun, Switzerland
[2] Institue of Nanoscience and Materials of Aragon – CSIC, Edif. I+D, C/ Mariano Esquillor Gómez, 50018 Zaragoza, Spain
[3] TOFWERK AG, Schorenstrasse 39, CH-3645 Thun, Switzerland
[4] Laboratory for Advanced materials processing, Empa-Swiss Federal Laboratories for Materials Science and Technology, Feuerwerkerstrasse 39, CH-3602 Thun, Switzerland
E-Mail: ivo.utke@empa.ch

Focused Electron Beam Induced Deposition (FEBID) is a deposition technique capable of direct writing of 3D nanostructures. It is often used for deposition of metals. It involves the focused electron beam dissociating the metalorganic molecules adsorbed on the substrate to deposit the desired material. However, the deposited structures often suffer from the low metal content in the deposit. One of the main cause is the dissociation of organic ligands and their incorporation into the deposit when electrons interact with precursors on the substrate. Hence, it is important to understand the exact dissociation pathways under electron beam irradiation because it can help tailoring the process parameters as well as designing new precursors to deposit material with high purity.
The fragmentation processes have long been studied in gas phase where single precursor molecules interact with the electrons and in condensed phase where electrons interact with the condensed film of the precursor on the substrate, usually under ultra-high vacuum in cryogenic temperatures [3]. Their results are useful in FEBID, but the experimental conditions of these methods largely differ from the one used in FEBID, unlike in the gas phase studies, in FEBID the precursor molecules interact with substrate. Moreover, in FEBID, the substarte temperature is stabilized such that no condensation takes place which is in contrast to condensed phase fragmentation studies. Hence, it is important to monitor the fragmentation processes directly during FEBID. An approach to solve this problem is to probe the fragments produced during FEBID through Mass Spectrometry [1]. In the Focused Electron Beam induced Mass Spectrometry (FEBiMS) approach, the charged fragments produced by electrons are extracted and studied using the Time-of-flight mass spectrometer (ToFMS), see Figure 1.
Both positively and negatively charged fragments can be extracted depending on the mode of operation. Fragments produced can be studied as a function of time during the FEBID process. Recently FEBID using $W(CO)_6$ was monitored using this technique [1].
Through the same FEBiMS approach we tried to understand the dissociation the precursor trimethyl platinum- methylcyclopentadienyl ($Me_3(MeCp)Pt$) during FEBID. The positively charged fragments were extracted and compared with the gas phase fragments produced in dissociative ionization using 70 eV electrons (see Figure 2). The discussion of the results includes the energy-dependent cross-section of fragments (as FEBID involves electrons with broad energy spectrum), multiple collision conditions, the contribution from surface reactions, ionization of volatile products produced through surface reactions and background gas ionization which can be seen clearly from relative intensity of the water molecule. The gas phase studies [2] revealed that CH_3^+ is ~0.4% of the highest peak ($MePtCpMe^+$) and that no CH_4^+ fragments were observed. In condensed phase studies, CH_4 is one of the volatile products. In the FEBID study we observed CH_3^+ (~41% of the highest $MePtCpMe^+$ peak) and a clear CH_4^+ signal distinguishable from the O_2^{+2}/O^+ signal (Figure 3). This clearly indicates that the spectrum has contributions from gas phase and adsorbed phase

which needs to be differentiated to understand the reactions in FEBID process. This requires further investigation which will be discussed during the conference.

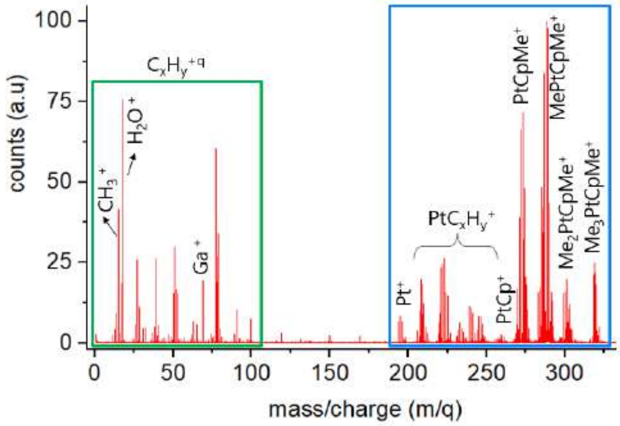

Figure 1. Schematic of the FEBiMS approach for monitoring the fragments produced in FEBID process.

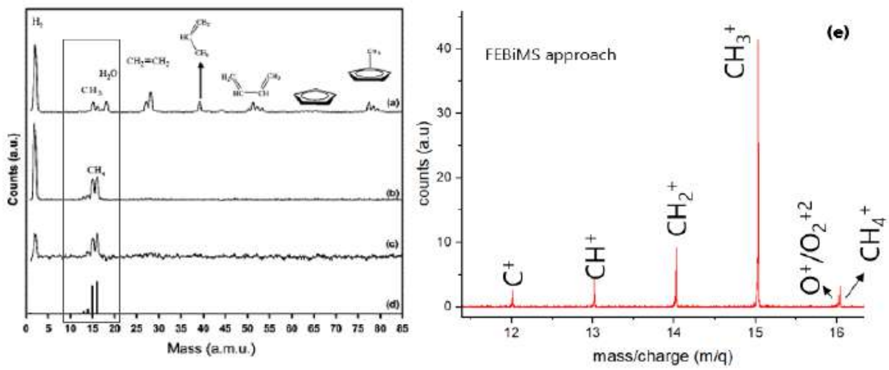

Figure 2. a) Fragments produced when 70 eV electrons interact with precursor in gas phase [Engmann, Sarah, et al. 2012]. b) The fragments produced during the FEBID processing of Me3(MeCp)Pt.

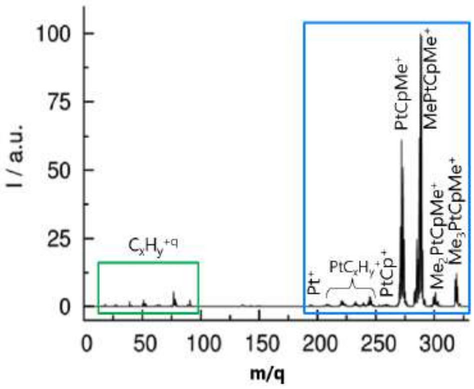

Figure 3. Mass spectrum (0-85 amu) (a) to (d) from ref[3] (a) gas phase $MeCpPtMe_3$ using 70eV electron beam and during the electron irradiation of (b) $MeCpPtMe_3$ and (c) $CpPtMe_3$ films adsorbed onto Au at ~180 K. (d) Reference mass spectrum of CH_4(g) [3]. e) Mass spectra from 12-16 amu observed during the FEBID processing of $MeCpPtMe_3$ through FEBiMS approach.

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P15: 4D STEM of Cryo-FIB Milled Cell Lamellae

K. Khakurel

ELI Beamlines Facility, The Extreme Light Infrastructure ERIC, Dolní Brezany, Czech Republic
E-Mail: Krishna.Khakurel@eli-beams.eu

Four-dimensional STEM has emerged as a tool to probe samples with sub-atomic details and to localize light elements in the specimen. While this method has been extensively applied to samples from material science domain, it's application to biological specimen has been sparse. Here, we present our efforts to perform 4D STEM of cryo-FIB milled cellular lamellae. We will also present the enhanced information scribed in the 4D STEM as compared to traditional STEM and bright filed imaging. The potential of using plasma-FIB to extend the current work to 4D STEM of tissues shall be discussed. Finally, we will show how our current works will pave the way forward for cryo-tomo-Ptychography of FIB milled cells and tissues.

P16: Niobium nanoSQUIDs for scanning SQUID-on-cantilever microscopy, patterned by focused Ne and He ion beam milling

T. Griener[1], S. Pfander[1], S. Koch[1], J. Ullmann[1], C. Bureau-Oxton[2], D. Jetter[3], A. Vervelaki[3], K. Bagani[3], U. Drechsler[2], R. Kleiner[1], M. Poggio[3], A. Knoll[2], and D. Koelle[1]

[1] Physikalisches Institut, Center for Quantum Science (CQ) and LISA+, Universität Tübingen, Germany
[2]IBM Research Europe – Zürich, Rüschlikon, Switzerland
[3]Department of Physics and Swiss Nanoscience Institute, University of Basel, Switzerland
E-Mail: koelle@uni-tuebingen.de

Superconducting quantum interference devices (SQUIDs), that consist of a superconducting loop intersected by one or two weak links (Josephson junctions), are the most sensitive detectors for magnetic flux. Their flux sensitivity improves with decreasing loop inductance, i.e. with decreasing loop size. This motivates the miniaturization of SQUIDs, to form nanoSQUIDs with submicron lateral size. One promising application of ultrasensitive nanoSQUIDs is in scanning probe microscopy: Scanning a nanoSQUID in close distance from a sample surface enables imaging of magnetic flux structures on the nanoscale. Simultaneously, the temperature dependence of the critical current of the Josephson junctions intersecting the SQUID loop provide the possibility for thermal imaging of dissipative processes.

In the project “FIBsuperProbes”, we develop tools that combine magnetic and thermal imaging with topographic imaging – this shall be achieved by placing nanoSQUIDs on silicon cantilevers that can be used for atomic force microscopy (AFM) [1]. Here, we report on the development of Nb nanoSQUIDs fabricated on plain Si surfaces (as test devices) and on commercial and on custom-made AFM silicon cantilevers. The Nb thin film structures are nanopatterned by milling with a focused neon ion beam. In addition, we use a focused He ion beam for recutting those structures, to trim the electrical transport properties of the devices. We present the performance of test devices and nanoSQUIDs fabricated on cantilevers, and we discuss further strategies to improve device performance, in particular with respect to improved flux sensitivity and spatial resolution.

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P17: Design and Optimisation Method for Obtaining Pioglitazone and Curcumin-Loaded Chitosan Nanoparticles

F. Lupascu [1], A. Iacob [1], M. Apotrosoaei [1], I. Vasincu [1], O. M. Ionescu[1], B. Profire [2], and L. Profire [1]

[1]Department of Pharmaceutical Chemistry, Faculty of Pharmacy, “Grigore. T. Popa” University of Medicine and Pharmacy of Iași
[2]PhD student, Department of Internal Medicine and Toxicology, Faculty of Medicine, “Grigore. T. Popa” University of Medicine and Pharmacy of Iași
E-Mail: florentina-geanina.lupascu@umfiasi.ro

Diabetes mellitus (DM) is one of the most serious chronic diseases, which is considered an important public health problem and a social-economic one due to the increased prevalence from last decades [1]. Worldwide, diabetes causes nearly 4 million deaths annually, while another 1.5 million new cases are diagnosed and it is estimated that in 2040 one in ten people will suffer from this disease [2]. Aim: The aim of the present study was to design and optimise the method used for preparation of CS NPs loaded with pioglitazone (P) and curcumine (C), as a new multi-target polymeric nanosystem, for DM therapy. Material and methods: CS NPs loaded with poiglitazone, curcumin and pioglitazone-curcumin were prepared using ionic gelation method. After setting the optimal parameters, the developed nanosystems (CS_P NPs, CS_C NP and CS_P_C NPs) were characterized in terms of morphological aspects, using Scaning Electronic Miscroscopy (SEM) and of average particle size and polydispersity index, using Dynamic Light Scattering (DLS) measurements. In addition, the zeta potential values, an important parameter for physical stability of the NPs, was also studied. Results and discussion: The following parameters were set as optimal, which assure the obtaining of NPs with proper characteristics: CS 0.1% (wt/v): TPP 0.1% ratio of 3:1, Tween 80 0.05% (wt/v), DMSO as solvent, sonication during 30 min, rotational speed of 1400 rpm. Conclusions: The developed CS_P_C NPs are stable nanosystems with proper physical characteristics (PS = 215 nm, PI = 0.300, ZP = 11.13 mV), which support to continue our studies, using in vitro and in vivo assays, to prove their efficiency as multi-target polymeric nanosystem.

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P18: Comparative study of focused electron & ion beam induced deposition (Ga+, Xe+) with Cu(II)(hfac)2·xH2O precursor

K. Maćkosz[1], P. Le Gal[2], A. Delobbe[2], I. Utke[1], and G. Goupil[2]

[1] Empa, Swiss Federal Laboratories for Materials Science and Technology, Laboratory for Mechanics of Materials and Nanostructures, Feuerwerkerstrasse 39, 3602 Thun, Switzerland
[2] Orsay Physics, TESCAN Orsay, 95 Avenue des Monts Auréliens, ZA Saint-Charles 13710 Fuveau, France
E-Mail: krzysztof.mackosz@empa.ch, pierre.legal@orsayphysics.com

In this study we extend our analytical work on focused ion beam induced deposition (FIBID) and focused electron beam induced deposition (FEBID) with the copper precursor copper hexfluoroacetylacetonate precursor Cu(II)(hfac)2·xH2O. Copper is a widely used metal to make via and lateral interconnects in complex high density 3D packaged electronic circuits on the circuit boards.
The Cu(II)(hfac)2·xH2O precursor is known for its notoriously low copper content <≈10at.% during room temperature FEBID deposition [1], which can only be marginally improved by vacuum annealing [2]. The FEBID material contains large amounts of carbonaceous matrix (with minor amounts of oxygen and fluorine from the ligand elements) embedding the copper nanocrystals. Extending from FEBID to FIBID using not only gallium ion beams but also the noble gas ion beam of xenon we were able to increase the metal content in the deposits. We found the metal content in deposited squares to be dependent on the type of focused charged particle beams used for deposition being highest for xenon (up to 68 at.% of Cu) and lowest for electrons, Xe+ > Ga+ > e-. Morphological changes were observed in cross sections of Xe ion deposited squares pointing to a more granular structure. Similarly, a dependence on the hydration state of the precursor was observed, which could be mitigated by increasing the precursor flux of the dehydrated precursor by changing the temperature of evaporation. We will discuss the findings and propose a route for high copper content deposition using FEBID & FIBID.

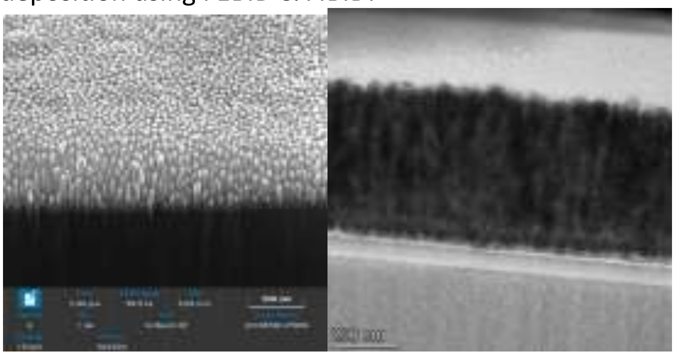

Figure 1. Xe plasma FIB deposits with Cu(hfac)2. SEM (a) and TEM (b) image of cross section square deposit showing a grainy structure.

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P19: Precise fabrication of plasmonic nanostructures using helium ion beam milling of mono crystalline gold microplatelets

J. Meier[1], R. Kullock[1], L. Zurak[1], T. Feichtner[1], and B. Hecht[1]

[1] Nano-Optics and Biophotonics Group, Experimental Physics 5, University of Würzburg, Germany
E-Mail: jessica.meier@physik.uni-wuerzburg.de

Field hotspots in nanometer-sized gaps of plasmonic antennas are commonly employed to control and enhance light-matter interaction on the nanoscale and are therefore interesting for spectroscopic and optical sensing applications as well as to boost nonlinear processes such as higher harmonic generation and photoelectron emission [1]. Alongside large field enhancement, these phenomena often require control over the field distribution in the hotspot, which can be defined by the shape of the antenna gap and is therefore challenging considering the nanometer length scales. Plasmonic antennas that are fabricated via focused ion beam milling of mono-crystalline gold microplatelets with gallium ions (Ga FIB) showed superior optical properties compared to their poly-crystalline counterparts [2]. However, even though complex structures with connectors and several active as well as passive elements were realized, additional fabrication steps were needed to further determine the shape of the antenna gap [3]. Focused helium ion beam milling (He-FIB) of evaporated gold films already surpasses Ga-FIB and reaches sub-10 nm precision [4]. Here, we apply He-FIB to structure mono-crystalline gold microplate lets resulting in even smaller feature sizes which leads to high fabrication accuracy and reproducibility.
To demonstrate the resulting advanced control over the optical properties we study the nonlinear response of electrically connected nanoantennas, in particular second harmonic generation (SHG) [5]. In the bulk of centrosymmetric materials like metals the second-order dipole response vanishes, which is why SHG is usually quenched in plasmonic devices when excited with a linearly polarized plane wave. To enhance SHG in gold plasmonic antennas the centrosymmetry can be broken by an asymmetric gap geometry. We employ a three-step milling approach (see Figure 1): First Ga-FIB is used to create a rough outline of the antenna, followed by He-FIB to precisely define the antenna shape and finally the gap. With this combined Ga-/He-FIB approach we are able to realize asymmetric-gap antennas with ultra-sharp tips exhibiting a radius of curvature at the apex down to 3 nm for a gap size of 8 nm. The strength of the second harmonic intensity is adjusted by altering the geometric asymmetry in the gap region and is therefore employed to demonstrate the precision and variability of our Ga/He-FIB approach.

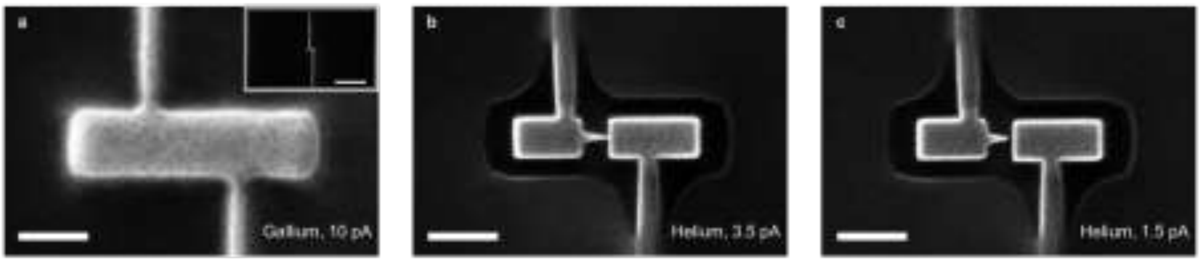

Figure 1. Helium ion microscope images illustrating the three-step milling approach based on Ga-/He FIB. **a**, Gold antenna after Ga-FIB, **b**, after the first He-FIB step, and **c**, after the second He-FIB step. Scale bars, 100 nm. The inset in **a** shows an overview of the antenna and the surrounding (4 × 4) µm glass window. Scale bar, 1 µm.

Acknowledgments
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P20: The effect of 3D hydrogel structure and properties on the escape of single cells from cancer spheroid

S. Metwally, and M. Lekka

Institute of Nuclear Physics Polish Academy of Sciences, Radzikowskiego 152, 31-342 Kraków, Poland
E-Mail: sara.metwally@ifj.edu.pl

Understanding the relationship between structural, mechanical, and biological properties is crucial in designing scaffolds for biomedical applications. The 3D hydrogels gained a lot of interest due to their unique properties, such as high porosity and water content, tunability of mechanical properties, and biocompatibility. Cell spheroids serve as an excellent tumor model used extensively in biomedical studies as they can mimic the complex physiological properties of living tissues [1]. It has been shown that shear stresses affect cancer cell migration and invasion in connection with changes in the mechanics of surrounding tissue, scaffolds' 3D microstructure, pore size, distribution, and interconnectivity [2,3].
The study aims to analyze the influence of shear strains on cell migration from the spheroid surface to the surrounding 3D collagen-hyaluronic acid (Col/HA) hydrogels. Spheroids were formed from non malignant cell cancer of the ureter (HCV29) and human bladder cancer (transitional cell carcinoma, T24) cells. Deformation of the samples was performed using a rotational rheometer, working in an oscillatory mode, by applying shear strain at the level mimicking physiological mechanical forces. Col/HA hydrogel structure was characterized using a scanning electron microscope (SEM), revealing the fibrillar collagen structure with high hierarchical porosity. Single-cell migration from the spheroid surface to the hydrogel interior was assessed using an optical microscope. Significantly higher cell migration was observed for T24 cells, due to the high invasiveness of these cells. Using cryo-FIB-SEM for cell-scaffold interface imaging allows for a detailed analysis of spheroid integration with the hydrogel structure at the submicron level. The result might help us understand the mechanical microstructure relationship and its impact on cancer cell migration.

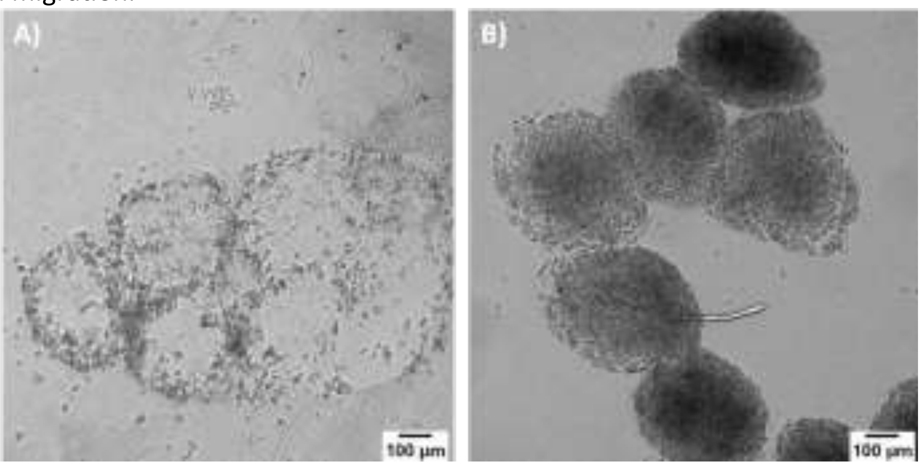

Figure 1. Cancer cells escaping from spheroids surface into Col/HA hydrogel environment under applied shear strain: A) transitional cell carcinoma (T24 cells) and B) non-malignant cell cancer of the ureter (HCV29 cell)

Acknowledgments
3D hydrogels and cell escape from spheroids were realized within the project financed by National Science Centre (Poland), no. UMO-2021/41/B/ST5/03032.

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P21: Generation and scaling of qubit arrays in diamond by focused single ion implantation and lithographic methods

D. Reinhardt[1,3], P. Räcke[1,2,3], T. Lühmann[1], J. Heupel[4], C. Popov[4], D. Spemann[2,3], and J. Meijer[1,3]

[1] Applied Quantum Systems, Felix Bloch Inst. for Solid State Physics, Universität Leipzig, 04103 Leipzig, Germany
[2] Tools, Leibniz Institute of Surface Engineering (IOM), 04318 Leipzig, Germany
[3] Leibniz Joint Lab "Single Ion Implantation", 04318 Leipzig, Germany
[4]Technological Physics, Inst. of Nanostructure Technologies and Analytics, Universität Kassel, 34132 Kassel, Germany
E-Mail: dominic.reinhardt@uni-leipzig.de

Color centers, especially the nitrogen-vacancy centers (NV centers) in diamond, have an increasingly important role in applications such as magnetic field sensors, single photon emitters or as quantum bits for the use in quantum computers. Precisely for this type of application, however, an exact positioning of the color centers is required to allow quantum mechanical coupling between the centers. This cannot be achieved by conventional ion implantation processes. We show the current production process of color centers by ion implantation with optical (UV) lithography masks for the positioning of the color centers in the sub-micron range. In order to be able to achieve an even more precise placement in the nanometer range in the future, new methods are presented, which should provide the possibility of placing NV centers with a precision of 50 nm in the diamond lattice using electron beam lithography and single ion implantation.
As an alternative to the use of lithographic masks, nitrogen ion implantation using a focused beam (FIB) can be utilized. For this application, a conventional gallium FIB was modified and equipped with an electron beam ion source (EBIS). An ionization of atoms in the gas phase up to complete ionization is possible and thus gives a variety of charge states. This enables the ions to be accelerated from just a few to several hundred keV. The direct writing with a three-dimensional nanoscale placement accuracy has already been demonstrated with argon ions. For the creation of NV centers in ultrapure diamond we are currently optimizing the focused nitrogen ion beam in this implanter. An adjustable and measurable beam current down to a range of just a few femtoamperes enables the implantation of individual ions. In order to realize deterministic single ion implantation we are furthermore investigating single ion detection techniques.
With the use of the masked and focused implantation methods, we are aiming for an upward scaling, to achieve four coupled NV centers as the next milestone. Here, the current status of this work in progress is presented.

P22: Focused ion beams from GaBiLi LMAIS for nanofabrication and nano-analytics

T. Richter, and U. Mantz

Raith GmbH, Konrad-Adenauer-Allee 8, 44263 Dortmund, Germany
E-Mail: torsten.richter@raith.de

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 established 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 are well suited for sample imaging due to their low sputter yield and surface sensitive properties.

GaBiLi ion sources unifies light and heavy ions in a single Liquid Metal Alloy Ion Source to fulfil requirements for both nanofabrication as well as ion imaging [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 both nanofabrication processes and nano analytics.

In this contribution we present workflows employing a vertical GaBiLi FIB on a lithography platform to facilitate unique Mill & Image processes for 3D ion microscopy. The selection of various milling paths or looping strategies with Bi ions circumvent selective sputtering and redeposition while digging into the sample. Intermittent highly surface sensitive imaging with light Lithium ions prevents further sputtering and allows 2D sample imaging for mapping the region of interest layer by layer.

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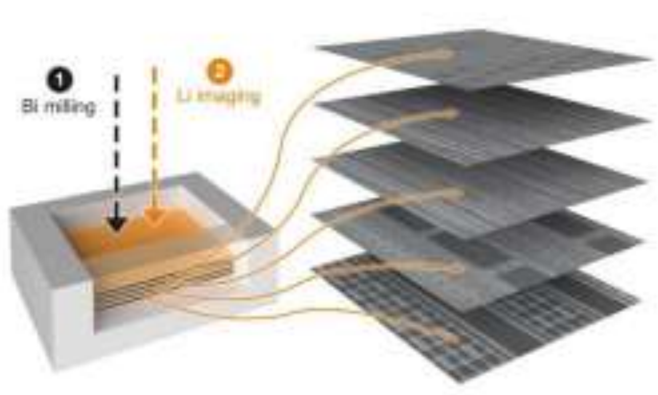

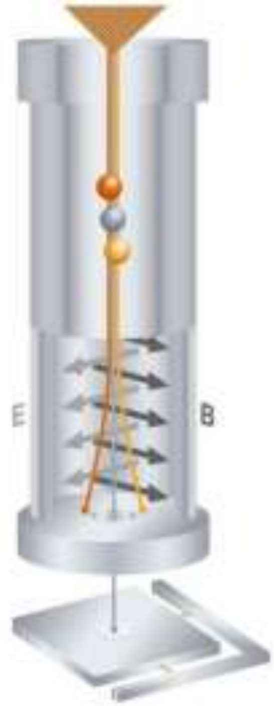

Left: Bismuth ions and Lithium ions are applied alternately for 3D ion microscopy. Appropriate Bi beam milling strategies are effectively employed to ensure smooth and uniform delayering of the sample while milling the substrate. Intermittent scanning ion microscopy with lithium delivers high-resolution images between respective milling steps, which can be combined for a 3D nano-reconstruction of the sample.

Right: Setup of a lithography platform with top-down FIB including LMAIS source and downstream Wien Filter for reliable and fast ion switching on a laser interferometer stage.

P23: Design of an electrostatic lens by differential-algebraic method and genetic algorithm

A. Sabouri, and C. S. Perez-Martinez

London Centre for Nanotechnology, University College London, WC1H 0AH, London, United Kingdom
E-Mail: a.sabouri@ucl.ac.uk

Electrostatic lenses have been widely used in electron/ion microscopes, charged particle lithography and secondary ion mass spectroscopy systems. In order to optimise the efficiency of these lenses, the analysis of optical aberration is required. The aberration can be calculated in three ways: through direct ray tracing of particles, aberration integral, or differential-algebraic (DA) method. The latter method is far more powerful than the traditional method as it evaluates high-order lens aberrations (third, fifth order and etc.), avoiding the painstaking derivation of formulae [1].
The DA method is based on the theories of nonstandard-analysis and formal series. It is an automated differential method to compute arbitrary order derivatives with high accuracy without truncation and rounding errors. This method requires a single ray trace and computes the aberrations by writing the equation of motion with respect to the position on the optical axis as the independent variable [2].
Due to the swift nature of the DA calculation procedure, it is feasible to use a search optimisation method such as a genetic algorithm (GA) to determine the optimum lens design. The GA approach utilises the initial population of different lens configurations, and through the iterative process of crossover and mutation, the optimum lens design can be achieved [3].
This study applies the DA method to compute the third-order aberration of an Einzel lens. This is done by computing the electrostatic field using the finite difference method. This is followed by fitting the axial potentials using the Hermite series. Then the focusing voltage is calculated by solving the equation of the motion for the charged particles, and the DA ray trace is performed to achieve the optical aberrations. The spot sizes of lenses with different configurations are calculated to produce the initial population for the GA method. The diameter of each electrode and the gap between them are varied. New configurations for lenses are produced by applying crossover and mutation to the lens parameters, and the optimum design for achieving the best spot size is obtained.

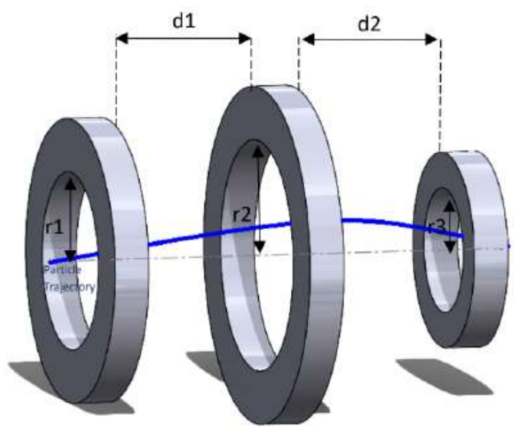

Figure 1. Schematic view of an Einzel lens and the particle trajectory.

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P24: Optimization of tungsten-based deposits by Focused Ion Beam Induced Deposition on Scanning Probe Microscopy cantilevers

A. Sáenz[1], F. Sigloch[1], S. Sangiao[1,2], and J.M. De Teresa[1,2]

[1] Department of Condensed Matter Physics, Instituto de Nanociencia y Materiales de Aragón (INMA), Universidad de Zaragoza- CSIC, 50009, Zaragoza, Spain
[2]Laboratorio de Microscopías Avanzadas (LMA), Universidad de Zaragoza, Spain
E-Mail: asaenz@unizar.es

Superconducting nanostructures exhibit unique properties and thus have many promising applications in the development of high-precision sensors [1]. The present work deals with the fabrication of tungsten-based nanoscale Superconductive Quantum Interference Devices (nanoSQUIDs) on Scanning Probe Microscopy (SPM) cantilever tips as part of the FET-OPEN project (892427). The resulting SQUID on Tip (SOT) sensors would allow SPM to map magnetic fields, magnetic susceptibility and electric currents, giving researchers deeper insight into 2D materials. In the present work nanoSQUIDs are fabricated by means of Focused Electron/Ion Beam Deposition (FEBID/FIBID), direct write techniques that allow the growth of superconducting materials on different substrates independent of their topography.
Extensive research on tungsten-based nanostructures deposited by means of FIBID has been carried out. The resulting deposits generally exhibit superconducting behavior at temperatures below 4-5K [2] and interesting properties such as vortex phenomena [3] and tunable T_c [4]. Tungsten-based SQUIDs presenting current modulation have also been successfully deposited on planar silicon substrates with 300nm thermally grown silicon dioxide [5].
In this work, we optimize the growth conditions of tungsten deposits wires on silicon cantilevers with 50nm thermally grown silicon dioxide by Ga+ FIBID using $W(CO)^6$ as the precursor gas. Results show different growth rates in cantilevers and planar substrates. We have conducted experiments on chemical composition and growth rate under different deposition parameters to optimize the deposition process for future application in tungsten-based SOT fabrication.

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P25: fit4nano for high school teachers: focused ion beam (FIB) technology – infographics, application examples, quiz questions and exercises

S. Schintke[1], S. del Rossi[1], G. Rius[2], C. Hofer[3], A. Berens[4], and G. Hlawacek[4]

[1] Laboratory of Applied NanoSciences (COMATEC-LANS), Department of Industrial Technologies, HEIG-VD // HES-SO University of Applied Sciences and Arts Western Switzerland, CH-1401 Yverdon-les-Bains, Switzerland
[2] Institute of Microelectronics of Barcelona (IMB-CNM-CSIC), ES-08193 Barcelona, Spain
[3] HTL Leoben, A-8700 Leoben, Austria
[4] Helmholtz-Zentrum Dresden-Rossendorf, D-01328 Dresden, Germany
E-Mail: silvia.schintke@heig-vd.ch

Focused ion beam technologies have nowadays gained broad application in science and engineering e.g. for nano- and microstructuring, surface milling, ion implantation, as well as for surface imaging. While high school courses generally address the topic of charged particles in electric and magnetic fields as well as the working principles of electron sources, electron beams and electromagnetic lenses as used in scanning electron microscopes, examples of emerging applications using focused ion beams are rarely addressed.

In order to generate a broader awareness and knowledge about focused ion beam techniques, the COST action FIT4NANO has decided to develop teaching materials for high school teachers, illustrating application examples of focused ion beam technology, as well as quiz questions and exercises, which can be directly integrated within high school physics curricula. To this extend, we have designed interactive infographics, including illustrations, quiz questions and exercises about focused ion beam technology (Fig 1).

Figure 1. *online teaching material platform "fit4nano for high school teachers" (prototype).*

The material is first developed in English and will be translated to further languages (at least to German, French, and Spanish). The content in English will be edited also as a single document, which allows teachers to select and adapt the proposed content according to their needs.

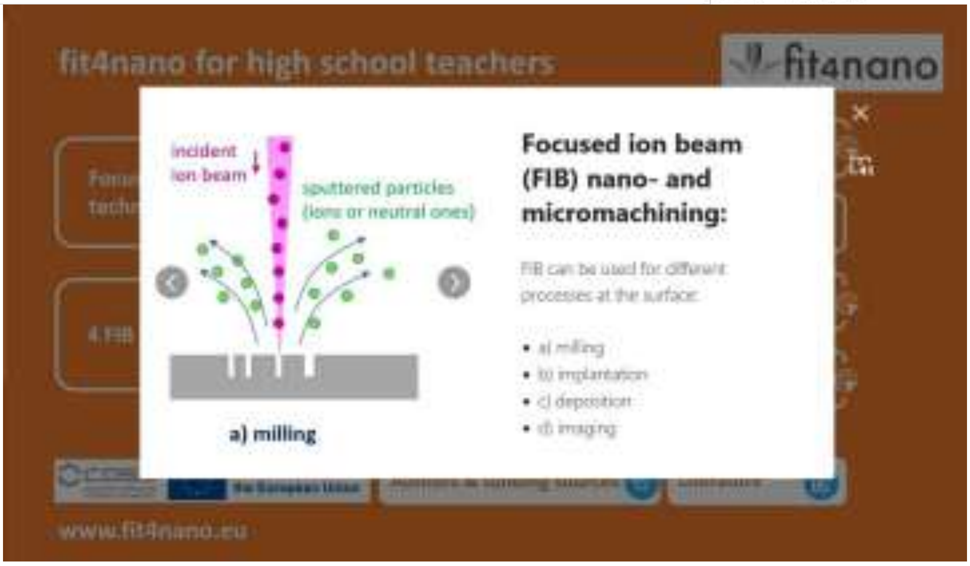


Figure 2. *Example from the online platform "fit4nano for high school teachers" (prototype).*

Illustrations, quiz questions and exercises have been pre-tested in high school physics classes in Germany, and in Engineering curricula with bachelor and master students at the University of Applied Sciences and Arts Western Switzerland in 2022. Further tests are performed with the online material in 2023. We present the development, the finalized online version (in English, German, French, and Spanish), as well as results from teacher feedback.

Acknowledgements

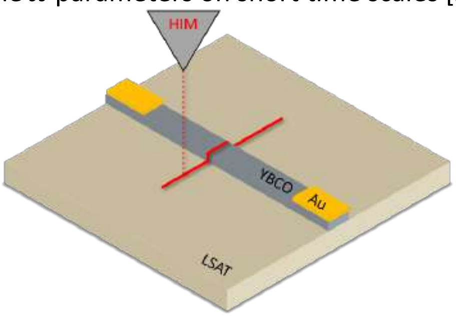
This work has been supported by the COST Action CA19140 Focused Ion Technol ogy for Nanomaterials (FIT4NANO) funded by the European Commission, and HEIG-VD/HES-SO (pro ject NANOFIT, HES-SO IA-EXT20-103-107469).

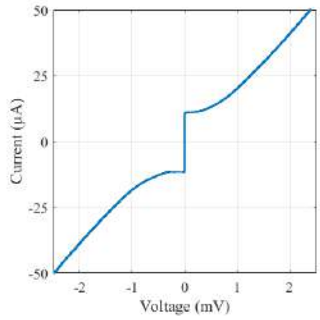
P26: $YBa_2Cu_3O_{7-\delta}$ Josephson junctions written with a focused He ion beam

C. Schmid[1], M. Karrer[1], K. Wurster[1], M. Meichsner[1], N. Hasan[1], R. Manzorro[2,3], J. Pablo-Navarro[2,3], C. Magen[2,3], D. Koelle[1], R. Kleiner[1], and E. Goldobin[1]

[1] Physikalisches Institut, Center for Quantum Science (CQ) and LISA+, Universität Tübingen, 72076 Tübingen, Germany
[2] Instituto de Nanociencia y Materiales de Aragón (INMA), CSIC-Universidad de Zaragoza, Spain
[3] Laboratorio de Microscopías Avanzadas (LMA), Universidad de Zaragoza, Spain
E-Mail: Christoph.schmid@uni-tuebingen.de

We use a focused He Ion Beam (He-FIB) to "write" Josephson barriers across $YBa_2Cu_3O_{7-\delta}$ (YBCO) thin-film bridges, i.e., by He-FIB irradiation we locally turn YBCO into a normal conductor or even insulator. Such He-FIB Josephson junctions (JJs) exhibit current-voltage (I–V) characteristics that are reasonably well described by the resistively and capacitively shunted junction (RCSJ) model. The He irradiation dose determines the critical supercurrent density and the resistivity of the JJs. The dependence of the critical current vs. magnetic field resembles a Fraunhofer pattern [1].
Scanning transmission electron microscopy (STEM) reveals that He-FIB irradiation modifies the YBCO crystal structure on the nanoscale. Applying a high line dose above about 1000 ions/nm leads even to amorphization. Electric transport measurements provide the evolution of the JJ parameters with time or thermal annealing. Those reveal barrier recovery processes and show how to stabilize the JJ parameters on short time scales [2].





Left: Schematic view of a He-FIB YBCO JJ. Right: IV characteristics at T=4.2 K; © by the authors

Furthermore, we investigated the interaction of two and more JJs placed close to each other and biased in series. Using a multi-terminal design layout, we found some unexpected behavior in the IV characteristics the origin of which still has to be resolved. Several approaches to synchronize arrays of He-FIB written JJs are investigated.

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P27: Vortex Chains and Vortex Jets in FIB-Milled MoSi Microbridges

C. Schmid[1,2], V.M. Bevz[3], B. Budinská[1,2], S. Lamb-Camarena[1,2], S.O. Shpilinska[4], M.Y. Mikhailov[5], W. Lang[1], and O.V. Dobrovolskiy[1]

[1] Faculty of Physics, University of Vienna, 1090 Vienna, Austria
[2] Vienna Doctoral School in Physics, University of Vienna, 1090 Vienna, Austria
[3] Physics Department, V. Karazin Kharkiv National University, 61022 Kharkiv, Ukraine 4Faculty of Computer Sciences, University of Vienna, 1090 Vienna, Austria
[5] B. Verkin Institute, National Academy of Sciences of Ukraine, 61103 Kharkiv, Ukraine
E-Mail: c.schmid@univie.ac.at

Fluxonics is a domain of science at the interface between nanotechnology and superconducting electronics [1]. It concerns with structure and properties of Abrikosov vortices and their application in in formation processing. In this regard, additive and subtractive manufacturing by using focused particle beams allows one to realize desired functionalities at the micro- and nanoscale. In our studies, we apply a focused ion beam for the creation of various configurations of magnetic flux quanta in current biased superconductors, which exhibit many properties of a Josephson junction, such as the electromagnetic radiation at overcritical currents I > Ic and steps in the microwave-irradiated current–voltage (I–V) curves [2]. These Josephson effects stem from the periodic motion of magnetic flux quanta (vortices) in the narrowest region of the bridge. According to the Aslamazov and Larkin (AL) theory [3], the I–V curve of such a constriction should exhibit voltage kinks each time the number of vortices in the 1D vortex chain is increased by one. However, in the presence of defects and fluctuations, the inter vortex repulsion stipulates the formation of a 2D vortex jet [4, 5], which goes beyond the 1D AL model. Here, by milling one or two slits across a MoSi thin strip, we make vortices to move in a vortex–jet or a vortex–chain fashion, respectively [6]. Our findings provide a demanded approach for the deduction of the number of vortices and their velocity in superconductors, and the realization of fluxonic devices operated in the one- and few-fluxon regime.

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P28: 3D Nanoprinting of advanced AFM nanoprobes

L. M. Seewald[1], R. Winkler[1], M. Brugger-Hatzl[2], and H. Plank[1,2,3]

[1] Christian Doppler Laboratory - DEFINE, Graz University of Technology, 8010 Graz, Austria
[2] Graz Centre for Electron Microscopy, 8010 Graz, Austria
[3] Institute of Electron Microscopy, Graz University of Technology, 8010 Graz, Austria
E-Mail: lukas.seewald@felmi-zfe.at

Aside of electron / ion beam microscopes, scanning probe techniques have evolved into an indispensable technology pool for surface characterization in diverse research areas from materials science over biotechnology towards life sciences. In that context, atomic force microscopy (AFM) plays a central role due to its low demands on surfaces and the compatibility with various environments. At the same time, lateral resolution in the lowest nanometer range can be achieved for conventional setups, while advanced operation modes provide access to functional surface properties, including electrical, magnetic, thermal, optical or mechanical properties. The latter possibilities, however, require functional nanoprobes, which are mostly achieved by either coating of conventional nanoprobes or by fabrication of a solid functional nanoprobe tip, which not only increases the achievable resolution due to sharper apexes but also eliminates the risk of delamination, which renders such probes useless. Related fabrication approaches include the mounting of specifically grown crystals on pre-structured cantilevers or the application of particle beam induced deposition techniques for direct growth of functional nanoprobes. In that context, focused electron beam induced deposition (FEBID) has evolved into a reliable, additive direct-write manufacturing technique at the nanoscale. Aside controlled fabrication of 3D (nano-)designs, material properties are naturally the decisive element when advanced operation modes are targeted. Aside of specifically designed precursor compounds, post-growth treatments, such as the exposure to temperatures / gases / electron beams in different environments, have considerably expanded the functional tunability towards the intended properties [1].

In this contribution, FEBID-based 3D nanoprinting will be revisited in the context of the here relevant fabrication of advanced AFM nanoprobes. Process aspects to achieve high-fidelity geometrical replication and post-treatment processes to specifically tune materials properties are also included for further discussions of 3D nanoprobe concepts, which are developed in our workgroup in collaboration with industry. We will focus on thermal [2], electrical [3] and magnetic [4] nanoprobes, where we not only highlight the advantages but also demonstrate the superior performance in comparison to alternative, commercially available products. We conclude the contribution with a view on ongoing activities but also discuss remaining challenges, to provide a comprehensive picture of the current status of FEBID-based, 3D nano-printing of advanced AFM nanoprobes.

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P29: Nanostructuring of graphene membranes with focused ion beams: towards 2D Metamaterials

E. Shorubalko[1]

[1] Empa – Swiss Federal Laboratories for Materials Science and Technology, CH-8600 Duebendorf, Switzerland
E-Mail: ivan.shorubalko@empa.ch

Nanostructuring of two-dimensional membranes with focused ion beams is demonstrated to be a promising technique for creating metamaterials. Few-nanometer-small structures with a down to ten nanometer period are demonstrated in graphene. Understanding the interaction between focused ion beams and freestanding 2D materials is crucial for exploiting the method towards novel metamaterials.

One of the important physical length-scales in electronic devices/materials is the charge carrier mean-free path (MFP). Fabricating devices with a characteristic feature size smaller than MFP leads to ballistic transport mechanisms dominating the conventional diffusive transport picture [1]. Consequently, these devices are called ballistic devices and exhibit various novel electronic properties that may survive to the THz regime due to their nature. Ballistic artificial nanomaterials – engineered to have novel properties that may not be found in nature and classified as a type of metamaterials, Fig.1(a) [2]. These materials can be envisioned as THz rectifiers. Electrical rectification in graphene based individual nanodevices has been demonstrated at room temperature [3, 4]. MFP in such typical graphene material is of sub-100nm. A large part of graphene research is devoted to its nanostructuring or modification on the nanometer scale. Except standard electron-beam lithography many other different techniques have been proposed and explored over the last decade. Most advanced method to controllably create true-nanoscale structures and even individual defects in suspended graphene is its irradiation by high energy electrons in transmission electron microscope (TEM). Nevertheless, the throughput and practicality of TEM method are limited. Sputtering by energetic focused ion beams (FIBs) has been envisioned and to a large extend demonstrated as a powerful method to create nanostructures in graphene [5-8]. Here we report on pushing the limits of this technique in several aspects: resolution, reproducibility, homogeneity, and throughput. Essential understanding of the graphene (or any other 2D material) sputtering by FIBs can be done by rather simple binary collision theory (BCT). Periodic arrays of a million of few-nm-small pores with narrow size distribution can be fabricated in free-standing graphene. Such perforated ultrathin membranes already find their applications in the field of quick and energy efficient filtration [9].

Exposing free-standing graphene to the controlled He- and Ga-ions irradiation allows precise determination of the sputtering yield. Graphene shows to be semi-transparent for 10-30keV energetic He ions. 99-97% of He-ions pass graphene without crating lattice defects. In contrast, large Ga-ions spatter carbon atoms from graphene layer in 80-50% incidence cases, depending on kinetic energy 5-30keV. Binary collision theory (BCT) explains well these experimental findings. Raman spectroscopy of damaged graphene reveals the defect formation mechanisms. He-ions create individual carbon vacancies until these defects overlap and amorphiszation occurs. Ga-ions can form mono and double vacancies as expected from BCT. Understanding the physics of the sputtering process allows to fabricate pores in graphene of only a few nm in diameter, ~2nm for He-beam and ~4nm for Ga. Eventually limited by ion beams diameters [8]. A million of nm-pore arrays are fabricated showing narrow pore diameter distribution, Fig.1(b). Pores as small as 10 nm and still resembling its triangular shape can be created in graphene, Fig.1(c). The method can be extended to other 2D membranes. Triangular (symmetry breaking) pores in graphene can be scaled down to 10 nm. This patterning resolution cross several physical length scales in typical 2D materials, such us: charge carriers and phonon mean free path. Presented study opens a way towards electrical rectifier metamaterials, frequency multipliers in THz range, phononic crystals and thermal rectifiers.

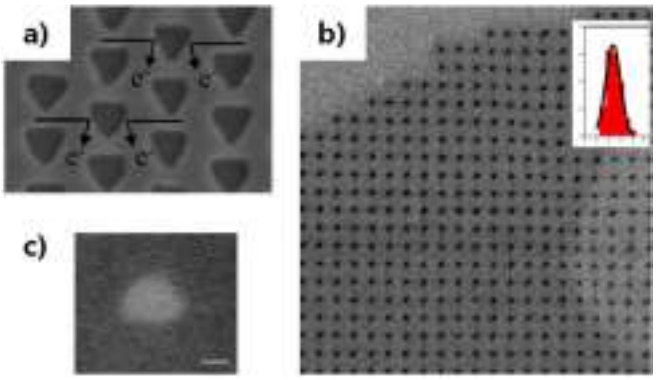

Figure 1. a) Scanning electron microscope image of an artificial functional material. The arrows indicate the typical electron trajectories. The devices operate similar to a bridge rectifier [2]. Triangles base is 200 nm large. B) Helium ion microscope image of perforated graphene membrane with He-FIB, period is 30 nm. Inset shows a typical pore diameter distribution in nm. C) Triangular pore in graphene by He-FIB. Scale is 10 nm.

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P30: Formation of translucent nanostructured zirconia ceramics

K. Smits[1], K. Laganivska[1], D. Olsteins[2], and I. Bite[1]

[1] Institute of Solid State Physics, University of Latvia, Latvia
[2]Center For Quantum Devices, Niels Bohr Institute, University of Copenhagen, Denmark
E-Mail: smits@cfi.lu.lv

Our study aims to investigate the mechanisms that affect the optical transparency of nanostructured translucent ZrO2 ceramic formation using SEM-FIB and TEM [1]. The production of advanced ceramics with desirable properties for various applications, such as 3D printed ceramics, has been a subject of extensive research. Achieving optimal microstructure and properties of ceramics requires a deep understanding of their nanocrystal growth and pore formation. Our study has demonstrated the essential role of ion beam and electron microscopy in providing a detailed analysis of the microstructure of ceramics and identifying changes resulting from different processing methods. These findings can inform the development of more effective techniques for producing high-quality ceramics with desirable properties.

To achieve translucent ceramic samples, we obtained a low agglomeration nanosized powder and sintered it at low pressure and low temperature. Even low pressures caused structural changes and defect creation in the nanocrystals, which we studied through annealing. Our findings highlight the critical role of electron microscopy in examining the microstructure of nano ceramic samples. Specifically, cross-section TEM images show a low quantity of pores in the pellets, with grain growth observed during annealing and the fusion of smaller nanocrystals into edge arrangements. Additionally, SEM images reveal changes in pore abundance and size with varying annealing temperatures. By analysing the SEM images, we determined the percentage and volume of pores and crystalline growth. Significant changes in translucency were observed with an increase in pore size.

Our study provides insights into the mechanisms that affect the optical transparency of nanostructured ceramics and highlights the critical role of electron microscopy in examining the microstructure of these materials.

P31: A Wien filter to separate beams of ionic liquid ions

E. Storey, A. Sabouri, and C. Perez-Martinez

London Centre for Nanotechnology, University College London, WC1H 0AH, London, United Kingdom
E-Mail: alexander.storey.15@ucl.ac.uk

Ionic Liquid Ion Sources (ILIS) are a novel ion source that could be used in material treatment applications, including focused ion beams. Ionic Liquids (ILs) are a type of salt, normally defined as having melting points below 100°C. Ionic liquids are normally synthesized by combining an organic cation with delocalized charge with a smaller anion, preventing the formation of an ordered crystal lattice due to poorly coordinated ions and low symmetry. An example, 1-ethyl-3-methylimidazolium tetrafluorobo rate, EMI-BF_4, is shown in Figure 1(a) [1].
Ion emission in ILIS is achieved by wetting a needle emitter with ionic liquid and biasing the liquid to a high enough electric potential relative to an extractor close to the needle tip, illustrated in Figure 1(b). The surface of the liquid forms into an electric meniscus, due to the balance of surface tension and electrical pressure. The electric field intensifies at the tip of the meniscus becoming large enough to induce evaporation of the ions.
The large variety of ionic liquid chemistries ava110isual make ILIS a versatile tool for materials processing. ILIS produce ions ranging from monoatomic species such as I^- or Cl^-, or kilodalton organic molecules. ILIS are also bright point sources with properties that could make them amenable to operation in a focused ion beam (FIB) column [2]. ILIS have already been used for reactive etching of silicon [3]. Some ionic liquids contain halogenated ions; the halogen species in the beam react with sputtered material from the target and prevent the redeposition of dislocated atoms. In addition, ILIS can provide both positive and negatively charged ion beams, by simply reversing the polarity of the field between the emitter and the extractor. Negative ions can be used when treating dielectric substrates to mitigate charging [4]. ILIS produce ion beams containing a variety of component particle species, and it is desirable to filter these species for materials processing with specific chemistries.
This work will present a design for a Wien filter using perpendicular electric and magnetic fields to separate the different ion species included in the ion beam. COMSOL Multiphysics has been used to calculate electromagnetic fields, trace particle trajectories, and evaluate the effectiveness of the design in selecting the desired ion species, the geometry used and the results of ray tracing through this geometry are presented in Figure 2. The filter has been built and the talk will present experimental results from retarding potential analysis (RPA) comparing the full beam and the filtered beam. An example RPA is shown in Figure 3.

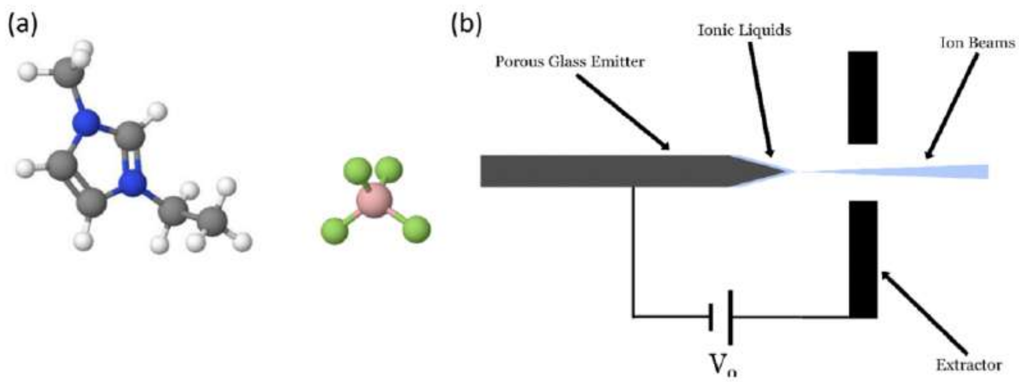

Figure 1. a) A 110isualization of EMI-BF4, created using MolView. B) Diagram of an ILIS setup incorporating a porous emitter, in the negative emission polarity.

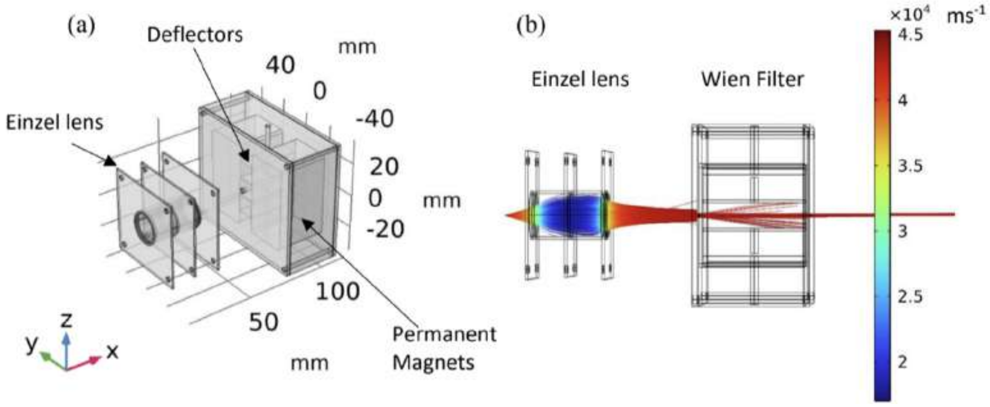

Figure 2. a) Depiction of the Einzel lens and Wien filter geometry constructed in COMSOL, through which ions are simulated to pass through in the x-direction. The lens is a three electrode configuration with the outer electrodes grounded and inner electrode biased. The Wien filter uses orthogonal electric and magnetic fields generated by the deflectors and permanent magnets respectively, to select ions to pass through an aperture based on velocity. B) Example of the raytracing of the monomeric species EMI^+ progressing through the lens and filter. The colour scale indicates the velocities of the ions as they traverse the device.

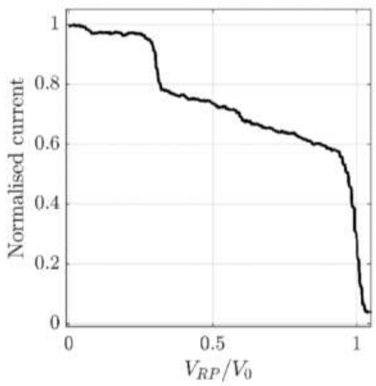

Figure 3. Sample retarding potential analyser curve for the full beam operating in the negative mode. The beam, with energy V_0 (-1540 eV in this example), is passed through a set of grids at potential V_{RP} and the current that makes it past the retarding grids is measured by a collector. For a monoenergetic beam, the full beam current should drop to zero only once VRP exceeds V_0. However, ILIS beams have solvated ions that break up during flight, and these fragmented ions have energies lower than the unfragmented ions. The fragmented ions account for the current drops for $V_{RP} < V_0$.

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P32: Atomic Scale Structure of Cobalt FEBID tips resolved by Atom Probe Tomography and Transmission Electron Microscopy

E. Szkudlarek[1], S. Gerstl[2,3], K. Maćkosz[4], and I. Utke[4]

[1] Academic Centre for Materials and Nanotechnology, AGH University of Krakow, 30-059, Krakow, Poland
[2]Laboratory of Metal Physics and Technology, Department of Materials, ETH Zurich, 8093, Zurich, Switzerland
[3]Scientific Center for Optical and Electron Microscopy, ETH Zurich, 8093, Zurich, Switzerland
[4] EMPA – Swiss Federal Laboratories for Materials Science and Technology, Laboratory for Mechanics of Materials and Nanostructures, 3602, Thun, Switzerland
E-Mail: aleszkud@agh.edu.pl

The study is dedicated to resolving the atomic structure of Co-tips, based on a combined approach using Atom Probe Tomography (APT) and Transmission Electron Microscopy (TEM). The tips were fabricated from the $Co_2(CO)_8$metal-organic precursor directly onto doped Si-based flat-top microtip cou pons, using Focused-Electron-Beam-Induced-Deposition (FEBID). This deposition technique is well suited for prototyping 3D nanodevices and studying various magnetic [1], superconducting [2] and plasmonic [3] phenomena occurring at the nanoscale for a wide class of materials [4]. However, due to the very small volume of deposits only a few microscopic techniques can be used to characterize them. The most common techniques are Transmission Electron Microscopy (TEM) combined with Energy Dispersive Spectroscopy (EDX) to study the crystal structure and chemical composition [5]. Atom probe tomography (APT) enables the mapping of chemical composition with sub-nanometric precision and identifying isotopes of elements with 10s of ppm sensitivity, including light elements such as hydrogen.
In this work, we demonstrate the application of TEM and APT as two complementary techniques to characterize the nanostructure of Co-FEBIP tip depositsto identify the chemical composition, level and types of organic and inorganic impurities, and the morphology of Co crystallites. For both techniques Ga-FIB was used to either mill a TEM lamella or to post-trim the Co-FEBID printed pillar into suitable APT tip shapes. The TEM studies show that cobalt forms nanocrystals in the tip deposits, see Fig. 1A-C. The APT enables near-atomic resolution 3D chemical mappings and the reconstruction of iso-concen tration-surfaces of the detected elements in the deposits, see Fig. 1D. Important additional information on the (non-crystalline) cobalt content of the matrix embedding the nanocrystals is obtained and allows to better understand the very good magnetic and electric performance of 3D cobalt FEBID nanodeposits found in literature.

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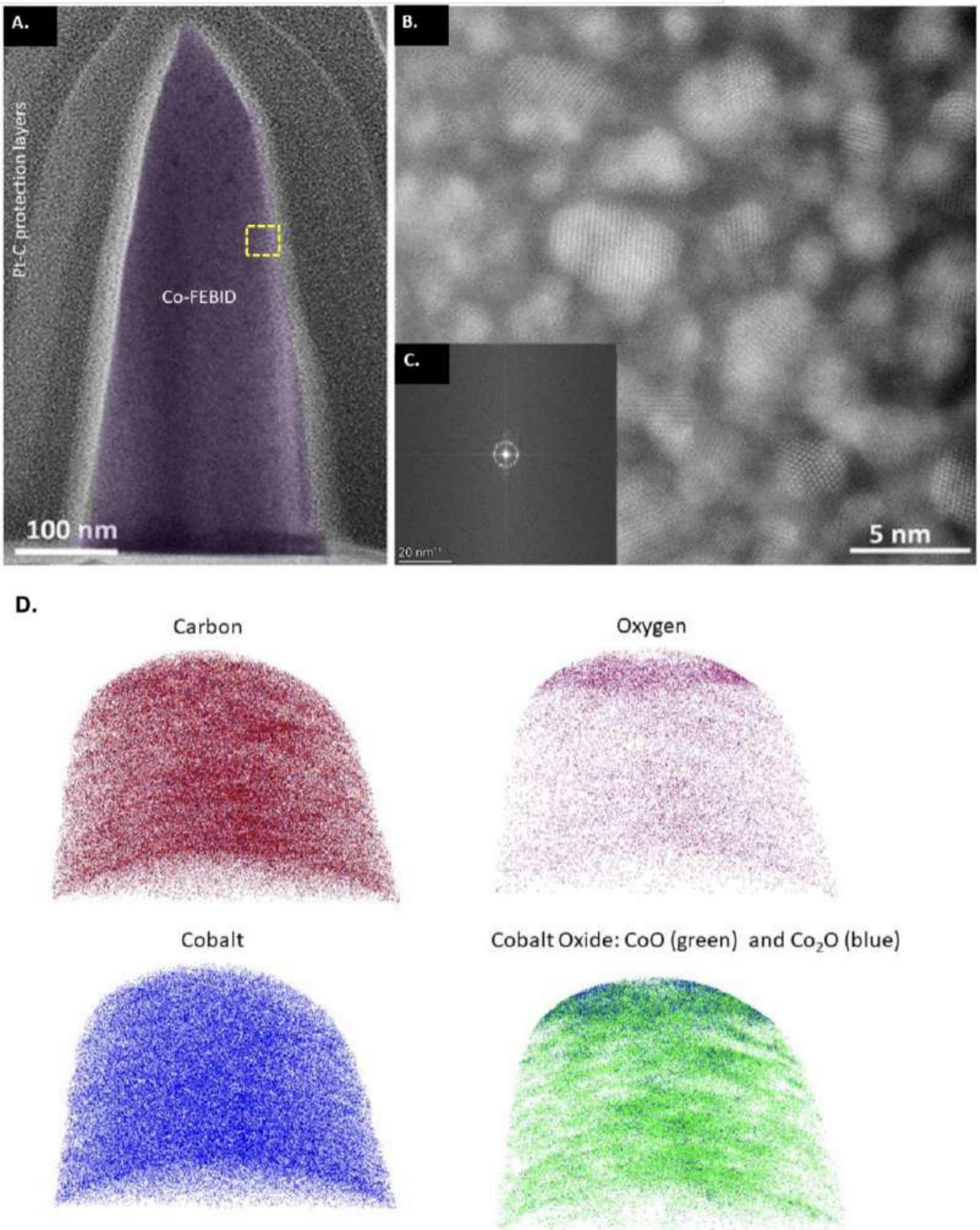

Figure 1 The morphology and chemical composition of Co-FEBID tips obtained with Co2(CO)8 and 25 kV and 50 pA focused electron beam: A) overview TEM dark field image of tip lamella cut by Ga-FIB, B) High Resolution STEM and C) Fast Fourier transform of Electron Diffraction of selected area indicating cobalt nanocrystals, D) compositional mapping using APT of the Co-FEBID tip post-thinned with Ga-FIB revealing localized variations in oxide clusters, correlating well with the nanocrystals observed via TEM.

P33: Towards tunable graphene phononic crystals

Y. Yu[1*], J. N. Kirchhof[1*], A. Tsarapkin[2], V. Deinhart[3], O. Yucel[1], B. Höfer[1], K. Höflich[2], and K. I. Bolotin[1]

[1] Department of Physics, Freie Universität Berlin, Arnimallee 14, 14195 Berlin, Germany
[2]Ferdinand-Braun-Institut gGmbH Leibniz-Institut für Höchstfrequenztechnik, Gustav-Kirchhoff-Str. 4, 12489 Berlin, Germany
[3]Helmholtz-Zentrum Berlin für Materialien und Energie, Hahn-Meitner-Platz 1, 14109 Berlin, Germany
*Authors contributed equally to this work.
E-Mail: aleksei.tsarapkin@fbh-berlin.de

Phononic crystals (PnCs) are artificially patterned media that exhibit bands of allowed and forbidden zones for phonons [1]. Many emerging applications of PnCs, from solid-state simulators to quantum memories, could benefit from the on-demand tunability of the phononic band structure.
Graphene is one of the most promising materials for tunable phononic crystals. It can be directly patterned down to a near-atomic length scale while supporting a variety of phononic lattices, and can withstand tensions as high as 42 N/m (~10% of strain) without breaking [2]. These properties, in combination, suggest that mechanically tunable graphene PnCs can be created [3]. However, the challenges associated with fabricating large-area, uniform graphene membranes, combined with the difficulty of detecting higher-order modes, have prevented the experimental demonstration of tunable graphene PnCs.
Here, we demonstrate the fabrication of suspended graphene PnCs in which the phononic band structure is controlled by electrostatically applied mechanical strain. The device shown in Figure 1 a) consists of a suspended large-area few-layer graphene membrane patterned with uniform honeycomb lattice holes. The honeycomb lattice was milled with a gallium ion focused ion beam. Patterns for ion milling were created using the FIB-o-Mat tool [4] and consist of spirals with optimized pixel spacing to reduce milling time and achieve well-defined holes (see Figure 1 b)). The spirals were consecutively milled from the outside to the inside to ensure a uniform release of mechanical stress in the membrane.
Interferometric measurements with the setup depicted in Figure 1 c), reveal the rich phononic resonance structure with a bandgap for out-of-plane acoustic phonons and its tunability by gating. The experimental data (Figure 1 d)) suggest a phononic band gap at 28-33 MHz in equilibrium (highlighted by the dashed green lines), which can be shifted by 9 MHz under a mechanical stress of 3.12 N/m. This is an important step towards tunable phononics and paves the way for further experiments on phono nic systems based on 2D materials.

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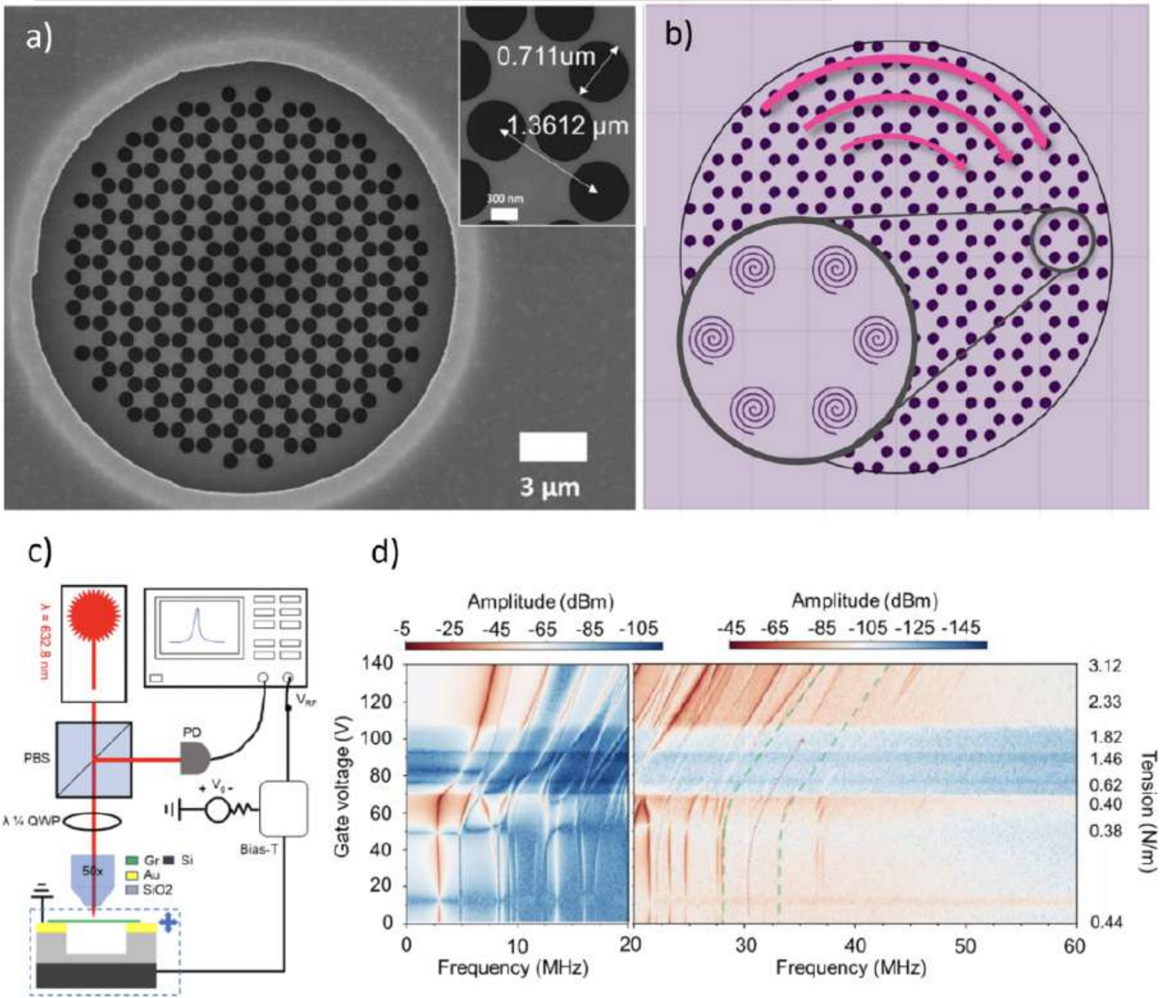

Figure 1. (a) PnC fabricated by FIB milling in a suspended graphene membrane. (b) Geometrical pattern, created by Fib-o-Mat [4]. It consists of the honeycomb lattice of spirals with optimized pitch and dwell time; milling order is from the outside to the inside. (c) Experimental setup for interferometric measurements of phononic spectra. (d) Vibrational spectrum of a phononic crystal device versus applied gate voltage, which controls the voltage. Dashed green lines mark the edges of the phononic band gap.

P34: Rotor stage for Electron BackScatter Diffraction analysis

E. Volobueva[1], Z. Köllö[2], K. Modic[2], and S. Bagiante[1]

[1] Department of Nanofabrication, Institute of Science and Technology Austria (ISTA), 3400, Klosterneuburg, Austria
[2] Department of Thermodynamics of quantum materials at the microscale, Institute of Science and Technology Austria (ISTA), Austria
E-Mail: evgeniia.volobueva@ist.ac.at

Focused Ion Beam system allows to conduct analyzes within the machine itself by means of customization of the tool. One of the main analysis used for studying metals and microelectronics is Electron BackScatter Diffraction (EBSD). This technique is enabling to determine microstructure, grain orientation, texture of materials and etc.
However, positioning of the sample for EBSD analysis inside FIB system can be tricky. Rocking mill stage and Multipurpose stage are limited for rotation and tilting when EBSD detector is inserted in the FIB chamber. Certain correction in sample's placement can be changed only manually, which requires to vent the chamber. Thus the positioning process becomes time consuming.
Nanofabrication Team of ISTA in collaboration with Modic Group (Thermodynamics of quantum materials at the microscale) designed a new type of stage that allows free rotation of the sample for its best positioning.

P35: High Precision 3D Nanoprinting of Sheet-like Structures and their Controlled Spatial Bending via Electron Beam Curing

A. Weitzer[1], M. Huth[2], R. Winkler[1,3], D. Kuhness[3], and H. Plank[1,3,4]

[1] Institute of Electron Microscopy and Nanoanalysis, Graz University of Technology, 8010 Graz, Austria
[2] Physics Institute, Goethe Universität Frankfurt, 60438 Frankfurt am Main, Germany
[3] Christian Doppler Laboratory - DEFINE, Graz University of Technology, 8010 Graz, Austria
[4] Graz Centre for Electron Microscopy, Steyrergasse 17, 8010 Graz, Austria
E-Mail: anna.weitzer@felmi-zfe.at

3D printing via Focused Electron Beam Induced Deposition (3D-FEBID) is one of the very few additive direct-write manufacturing techniques capable of creating highly precise 3-dimensional structures at the nanoscale. While sub-100 nm features can be done on a regular basis with high design flexibility and varying precursor materials, optimized conditions enable structural elements down to the sub-20 nm regime. This technique is meanwhile well-established for mesh-like architectures [1], meaning individual nanowires, which are spatially connected in 3D space. The next logical step is the expansion from wires to sheet-like structures, to extend 3D design possibilities, as recently introduced [2].

One of the main reasons preventing a straight-forward deposition of high-fidelity FEBID sheets is local beam heating. This process strongly influences local growth rates, which, although well-understood for meshed structures, becomes a real challenge for sheet-like architectures of varying element widths. Thereby additional dependencies on the dimensions of built objects as well as on the XY pixel position within the structures arise. Furthermore, electron trajectories are more complex in sheet-like objects, introducing additional proximity effects. To minimize these shape-disrupting effects, we combined 3D

FEBID experiments with finite-difference simulations and developed a Python compensation tool. We were thereby able to stabilize the growth for each XY pixel point in all individual patterning planes by pre-determined parameter adjustments (Fig. 1a). We then expanded our compensation model to wards more advanced structures, such as vertical screws (Fig. 1b), inclined segments and non-rectan gular shapes like trapezoids. All improvements combined led to a “construction kit” tool that is able to build compound structures (Fig. 1c) with very high shape fidelity. We thereby crucially improved FEBID based 3D nanoprinting of closed and consequently mixed objects.

In a next step, we applied post-growth electron beam curing (EBC) [3] to sheet-like 3D objects, to evaluate, whether this approach opens up new possibilities. In the process, structures are again irradiated by electrons, this time, however, without precursor gas present and only at selected areas (Fig. 2a). This impacts the inner structure and thereby the overall volume of exposed regions and, if only applied partially, enables controlled bending deformations (Fig. 2b and c). To gain a greater insight on this manner we performed experimental series and analyzed the resulting structures via SEM, TEM and AFM. We complemented these investigations by Monte Carlo Simulations to explore and identify ideal parameters for smooth, stable and reproducible morphological bending. In our studies, we included a variety of parameters, such as primary electron energy, overall dose, point pitch, dwell time, and beam incidence angle to achieve controlled and reproducible results. The expansion to more complex EBC patterns allows sophisticated bending (Fig. 2d-f), which, beyond morphological implications, is useful for functional imprinting of varying material properties in EBC-treated regions. We thereby extended the post-growth treatment possibilities of FEBID, showing both, flexibility and impact of EBC.

With this combined approach of strongly improved high-precision 3D-FEBID and post-deposition shape adjustments, we were able to open up entirely new design and tuning possibilities for high-fidelity nanostructures, some of which clearly go beyond the capabilities of sole 3D-FEBID (e.g. Fig. 2f).

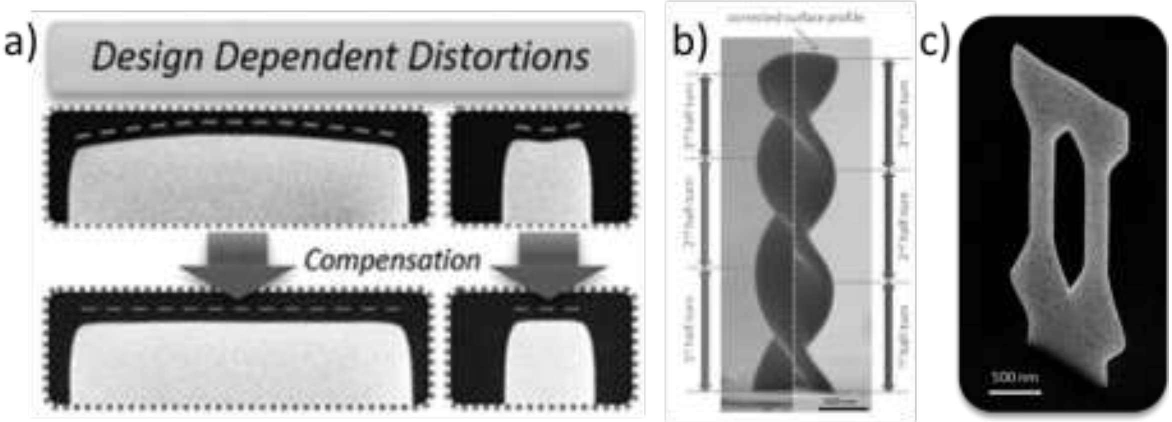

Figure 1: Strongly improved shape-accuracy during 3D-FEBID. (a) Comparison between directly deposited (top) and shape-stabilized wall elements (bottom). (b) Deposition of a twisted wall structure without (left) and with (right) height/temperature corrections. (c) Expansion of the developed compensation tool towards more complex structures with critical bottleneck features.

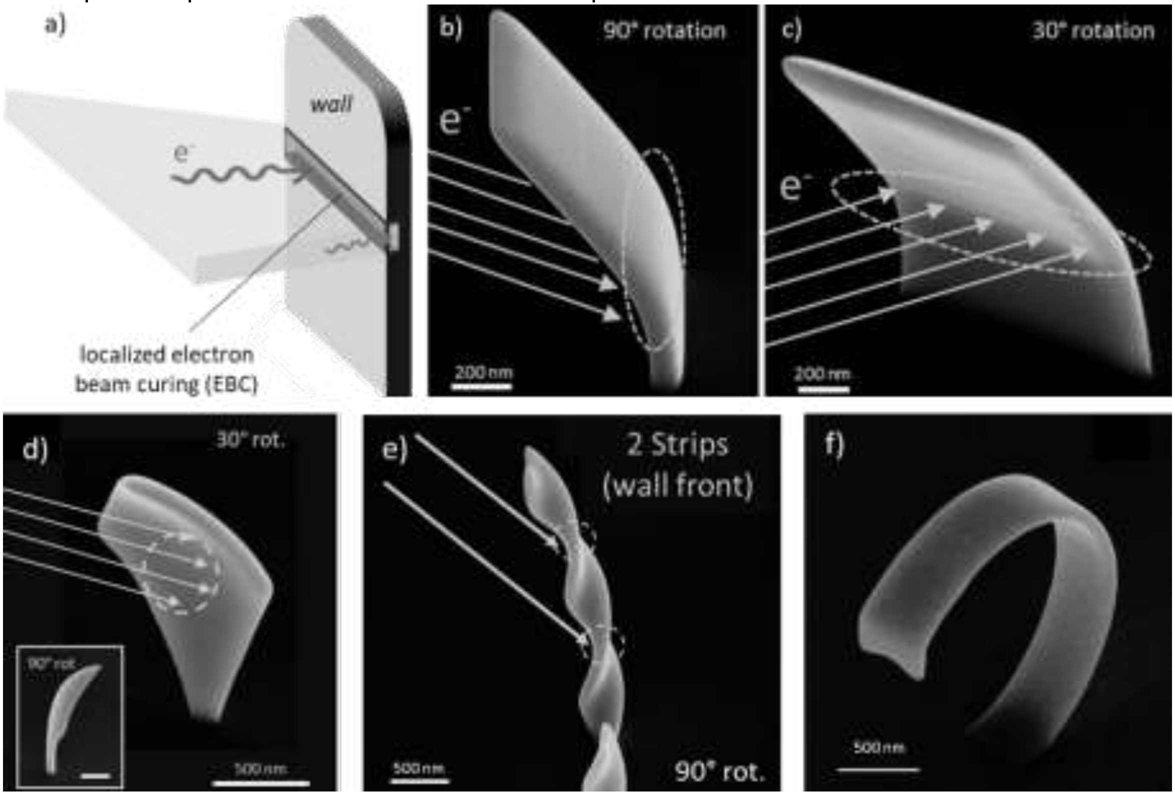

Figure 2: Controlled bending of 3D FEBID structures via EBC. (a) Schematic of the curing process with (b) and (c) the corresponding experimental results. (d)-(f) show more sophisticated deformation processes with (d) a sheet-like diamond element wrapping around a circular EBC area, (e) a screw bent at two curing sites and (f) where a complex overhanging structure was achieved.

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P36: Overcoming Challenges in FIB-TOF-SIMS Mapping with Fluorine Gas Assistance

K. Wieczerzak[1], and J. Michler[1]

[1] Empa, Swiss Federal Laboratories for Materials Science and Technology, Laboratory for Mechanics of Materials and Nanostructures, CH-3602 Thun, Switzerland
E-Mail: krzysztof.wieczerzak@empa.ch

The use of high vacuum-compatible time-of-flight secondary ion mass spectrometry (TOF-SIMS) detectors integrated within focused ion beam instruments (FIB) has become increasingly popular for correlative and complementary studies with other techniques without breaking vacuum conditions. In this work, the practical aspects of FIB-TOF-SIMS analysis enhanced by fluorine gas, delivered with gas injection system (GIS) are discussed, with systematic experiments conducted on high-purity Al and Cu single crystals. Results showed that fluorine-assisted FIB-TOF-SIMS analysis yields significantly higher SIMS signals due to the improved ionization probability of elements. The presence of fluorine gas also reduced the negative effect of preferential reoxidation of the sample surface, enabling high lateral resolution FIB-TOF-SIMS mapping. The importance of adjusting the TOF-SIMS pixel spacing and the role of native oxides and fluorine-based compounds in the ionization process were also demonstrated. The use of fluorine precursor during FIB sputtering was found to partially reduce reoxidation in the region of interest and enhance generation of secondary ions of main alloying elements, but complementary analysis methods may still be necessary for some elements. These findings provide important guidelines for maximizing the potential of TOF-SIMS+GIS setup for chemical characterization.

P37: Functional Imprinting: Local Modification of Beam Induced Deposits

R. Winkler[1], D. Loibner[1], V. Reisecker[2], A. Weitzer[2], and H. Plank[1,2,3]

[1] Christian Doppler Laboratory for Direct–Write Fabrication of 3D Nano–Probes, Institute of Electron Microscopy and Nanoanalysis, Graz University of Technology, 8010 Graz, Austria
[2]Institute of Electron Microscopy and Nanoanalysis, Graz University of Technology, 8010 Graz, Austria
[2] Graz Centre for Electron Microscopy, 8010 Graz, Austria
E-Mail: Robert.winkler@felmi-zfe.at

Additive manufacturing of nanoscale structures on almost any substrate type and surface morphology is one of the major unique selling points of Focused Electron/Ion Beam Induced Deposition (FEBID/FIBID). Beyond the direct-write of bulky, planar and simple pillars geometries, the controlled fabrication of even complex 3D nano-architectures have revolutionized this type of nanofabrication [1] due to the current singular status concerning design complexity, predictability / reliability, feature sizes, and functional variability [2]. The latter, however, is often challenging due to incomplete precursor dissociation with notoriously high carbon contents, which reduce or even entirely mask the intended functionalities right after initial fabrication. To improve the material quality and / or tune them precisely according to application requirements, post-processing steps, such as thermal treatments, exposure to gases and/or irradiation with photons/electrons/ions are extremely useful. While typically applied to the whole FEBID object in the past, we here go the next rational step and introduce a selected area modification, denoted as functional imprinting. That way, functional regions with varying designs can be integrated in the surrounding, pristine material, which serves as scaffold with different properties for different purposes.
In this contribution, we discuss two post-processing approaches, that both use a focused electron beam for variable shape imprinting. The first concept is called electron beam curing (EBC), where deposits are exposed to an electron beam under vacuum conditions. Here, the possibilities range from statistical grain growth for electric conductivity tuning [3] over modification of the carbonaceous matrix with mechanical implications [4] towards asymmetric stress-strain for the controlled bending of 3D objects beyond conventional fabrication possibilities [5]. For the second approach, electron exposure in low-pressure, room temperature water vapor is used to remove residual carbon from original deposits [6]. Here, we evaluate this approach by the local material transfer from pristine AuCX composition into pure gold and explore design possibilities, intrinsic limitations and functional properties of transformed areas. By that, we lay the foundation for advanced local material tuning of FEBID / FIBID materials, which might open up new application possibilities down to the nanoscale.

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P38: Polymer-inorganic hybrids for inducing self-healing functionality in metal oxides

O. Yurkevich[1], E. Modin[1], I. Šarić[3], R. Peter[3], M. Petravić[3], and M. Knez[12]

[1] CIC nanoGUNE BRTA, E-20018, Donostia-San Sebastián, Spain
[2]IKERBASQUE, Basque Foundation for Science, E-48013, Bilbao, Spain
[2] Department of Physics and Centre for Micro- and Nanosciences and Technologies University of Rijeka, 51000, Rijeka, Croatia
E-Mail: o.yurkevich@nanogune.eu

A rapid surge of research works is the commitment to the sustainability of mankind. However, the vast majority of these works are devoted to the self-healing of organic materials. At the same time, there is a growing demand for implementing this functionality to the inorganic materials due to the rapid development in the area of flexible electronics. The few existent examples for inorganic materials rely on liquid healing agents, such as liquid metals or liquid precursors. The progress in this field remains very challenging, mainly because of a lack of feasible healing agents and suitable ways to supply them to the damaged site. In this work, we propose an approach to form self-healing metal oxides (MeO) by applying the vapor phase infiltration (VPI) method [1].
We used VPI as a tool to induce self-healing properties into hybrid organic-inorganic materials. This was achieved by infiltration of metal organics into the polymers which do not possess reactive oxygen containing functional groups. Application of a typical VPI process to a functional polymeric substrate will result in the formation of dispersed metal oxide clusters and nanoparticles (NPs) inside the polymer along with an inorganic thin film of the same MeO on the surface. This hybrid polymer matrix with dispersed NPs can serve as a reservoir with healing agents for a repair of a cracked MeO film. Self healing of inorganic materials and structures was realized also without liquid agents by making use of the mobility of inorganic NPs within polymers, as the spatial distribution of NPs can be tuned by means of harnessing both enthalpy and entropy [2].
After the infiltration process, samples were transferred into the microscope chamber and cut in a controllable way by a Focused Ion Beam (FIB). Usage of FIB and SEM allowed inspecting the ruptured area of the hybrid structure prior to and after its exposure to the ambient atmosphere. X-ray photoelectron spectroscopy (XPS), energy-dispersive x-ray spectroscopy (EDX), and transmission electron microscopy (TEM) were used to analyze the chemical structure and composition of the obtained hybrids. The self healing effect after exposure of the FIB-cut sample to air was observed for zinc and indium metal oxides. Hereby, we introduce an alternative materials architecture and construction framework for designing inorganic materials capable to self-heal.

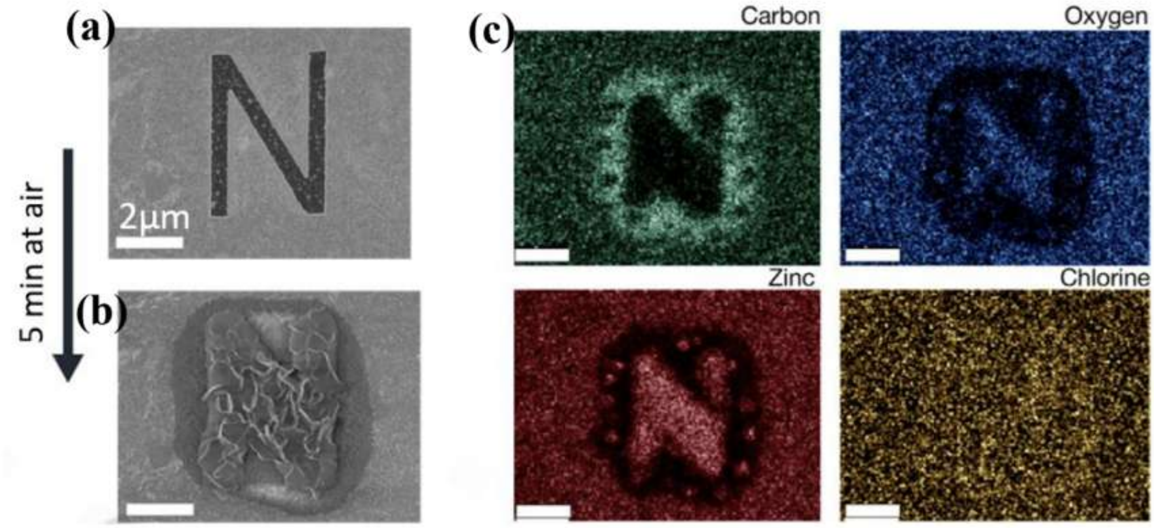

Figure 1. Healing of selectively FIB-etched patterns on the surface of ParyleneC/ZnO (a) before and (b) after exposure to air. (c) EDX maps of the mended ParyleneC/ZnO hybrid structure shown in (b). The scale bar is 2 µm. Reprinted with the permission from Ref [1].

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P39: Piezoelectric-magnetostrictive hybrid nanowires for nano magneto-electro-mechanical systems (NMEMS) fabricated with FIB

W. Zajkowska[1], B. Kurowska[1], M. Bilska[1], M. Chojnacki[1], H. Teisseyre[1,2], K. Fronc[1], J. Turczyński[1], and S. Kret[1]

[1] Institute of Physics Polish Academy of Sciences, Warsaw, Poland,
[2]Institute of High Pressure Physics, Polish Academy of Sciences, Warsaw, Poland
E-Mail: zajkowska@ifpan.edu.pl

The hybrid core-shell nanowires (H-NWs) with piezoelectric material cores (ZnO (0001)) and magnetostrictive alloy shells (FeGa) can be used as nano-devices controlled by external electric and magnetic fields. Both piezoelectric and magnetostrictive properties altered e.g. by mechanical deformation at the core-shell interface can generate a magnetic field around H-NW under the influence of voltage, and vice versa. This means that such nano-objects are sensitive to fluctuations of the environmental electromagnetic field.
We present a self-designed, step-by-step procedure for H-NWs nano-device fabrication: from the crystallization of cores, through FIB transferring & contacting, FeGa sputtering, to testing the final product by TEM examination. Particular emphasis is placed on the procedure of transferring core (ZnO) NW to the chip using Omniprobe needle and contacting it with chip using FIB techniques. Then, the FeGa half-shell and a few nm Pt/Al procreative layer are put on a selected area of contacted ZnO NW using the magnetron sputtering technique. We also test the setup using 4-probe electric measurement and magnetic field of the microscope lens up to 1T in TEM.

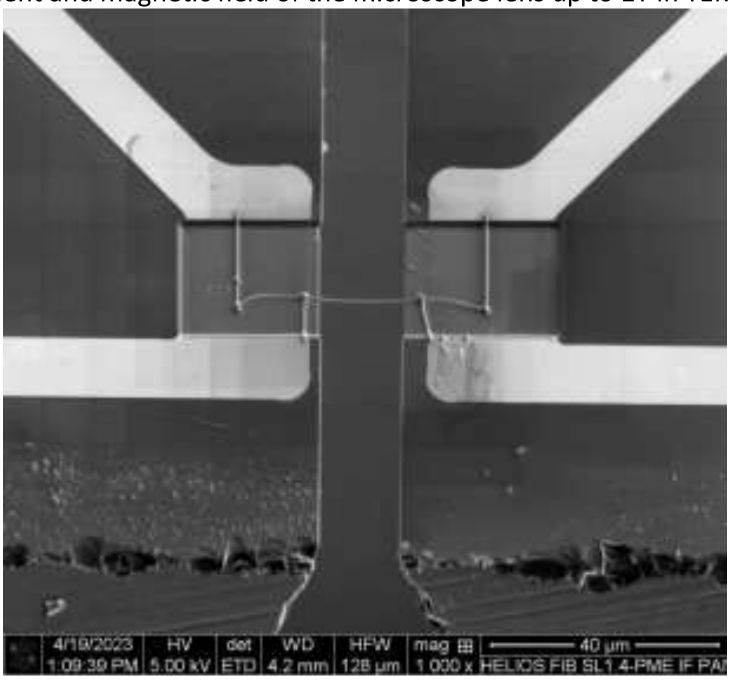

Figure 1. ZnO nanowire mounted to a chip in a 4-probe configuration (tilted by 52°). The NW is placed above 15 µm-wide cut in the chip, which enables TEM imaging.

This work has been supported by the Polish National Science Center, through project No: 2019/35/B/ST5/03434.

P40: USE CASE WITH NANOSPACE: How an analytical FIB-SEM-SIMS tool helps to understand contaminations on a wafer surface

A Zid[1,3], J. Silvent[3], T. Genieys[2,3] , T. Dangngoc[3], and A. Delobbe[3]

[1]Department of Ion Induced Nanostructures, HZDR, Dresden, Germany
[2] CIMAP, UMR 6252 (CEA), Caen, France
[3] ORSAY PHYSICS, Fuveau, France
E-Mail: a.zid@hzdr.de

ORSAY PHYSICS launches NanoSpace, the analytical FIB-SEM (Scanning Electron Microscope and Focused Ion beam) instrument working in an ultra-clean environment (pressure condition below 5.10-10 mbar inside the chamber). NanoSpace has been developed to be a convenient versatile and fully customizable instrument for FIB nanomachining and surface analysis on samples requiring the most rigorous contaminant-free environment. This UHV instrument has been designed with a modular architecture allowing a large choice of SEM and FIB columns, Gas Injection System (GIS), including the latest developed Ga-FIB column. This new gallium FIB allows to reach very good performances at both high currents (120 nA) and low energy (500 eV). In addition, NanoSpace could also be connected to a UHV compatible third party such as a Molecular Beam Epitaxy system or an Atom Probe Tomography system.
NanoSpace can be equipped with an Orthogonal Time Of Flight (OTOF) mass spectrometer for a unique configuration with surface chemical analysis correlated to FIB-SEM imaging, enabling 3D nanoscale chemical mapping of materials on large volumes. The SIMS analysis is performed by the combination of a new secondary ion extraction column called ExOTOF and an OTOF (Orthogonal Time Of Flight) detector which has the advantage of a simultaneous detection of all elements and molecules until about 500u with a dynamic range of 106-107 counts.s-1.nA-1. Both have been completely optimized to enhance the collection of the secondary ions and increase the mass resolution (M/ΔM) of above 4,500 (on the 28Si). Moreover, as NanoSpace is working in UHV, surface analysis and imaging are even more reliable in a contaminant-free environment.
We will present new features developed by ORSAY PHYSICS, including the new gallium FIB column and a use case where the NanoSpace showed interest in these in-situ correlative techniques on a wafer surface contamination case.

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