3rd Workshop of the European Focused Ion Beam Network

Dresden, June 12th–14th, 2019

Sponsored by:

3^{rd} European FIB Network Workshop

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Time	Status	Titel	Presenter	Affiliation
		EuFN Workshop Day 1 (June 12th)		
12:30		Get Together, Registration		
13:00		Official Opening	S. Facsko	Head of IBC, HZDR
13:20	Invited	Multigas Plasma FIB (from cell biology to lens manufacturing)	de Marco, A.	Monash University Clayton
13:55	Exhibitor	in situ TEM Lamella LiftOut for Backside Preparation - LO&Flip	Smith, A.	Kleindiek
14:15	Contributed	FIBing Gallium Arsenide (and others): experiments for understanding and limiting artefacts	Audoit, G.	Leti Grenoble
14:35	Exhibitor	Preparation of TEM samples and their observation by means of an adjustable tilt holder	Hrncir, T.	Tescan
14:55	Contributed	Channeling Effects in Gold Nanoclusters under He Ion Irradiation: A Molecular Dynamics Study	Ghaderzadeh, S.	HZDR
15:15		Coffee Break		
16:15	Invited	Microstructure is the “know-it-all” - classification approaches based on 3D-tomography, data mining and deep learning methods	Mücklich, F.	Uni Saarbrücken
16:50	Exhibitor	AFM-in-SEM LiteScopeTM: Tool for in-situ analysis of FIB modified materials	Novotna, V.	NenoVision
17:10	Exhibitor	In-Situ Correlative AFM/SEM/FIB analysis of FIB-treated samples	Schwalb, C. H.	GETec
17:30	Contributed	Feature Adaptive Sampling for Fast Image Acquisition in FIB and SEM	Pauly, C.	Uni Saarbrücken
17:50	Contributed	Removal of Curtaining Eects by a Variaonal Model from Mathemacal Image Processing	Fitschen, J. H.	Kaiserlautern
		EuFN Workshop Day 2 (June 13th)		
9:00		Get Together		
9:15	Invited	Why FIB is essential for analysis in semiconductor industry	Limbecker, P.	Globalfoundaries
9:50	Exhibitor	Latest advances of JEOL`s JIB-4700F Multi Beam System in focused ion-beam lithography	Harzer, T.	JOEL
10:10	Contributed	Defect production in supported two-dimensional materials under ion irradiation from atomistic simulations: the substrate is crucial	Kretschmer, S.	HZDR
10:30		Coffee Break		
11:00	Exhibitor	Advances in Multiple Ion Species Plasma FIB Technology	Prokhodtseva,A.	Thermo Fisher Scientific
11:20	Exhibitor	An Improved Experimental Setup for the Preparation of Back Side and Planar View TEM samples	Perez Willard, F.	Zeiss
11:40	Exhibitor	Site-specific Atom Probe Tomography Sample Preparation Method by Orthogonal FIB-SEM Column Layout	Onishi, T.	Hitachi
12:00		Lunch		
13:30	Invited	Ion Sources for Focused Ion Beams – Present Status and Prospective Developments	Bischoff, L.	HZDR
14:05	Exhibitor	New Applications in advanced FIB-SEM Nanofabrication with a FIB-centric Lithography System	Stodolka, J.	Raith
14:25	Contributed	SIMPLE – A FIB for Deterministic Single Ion Implantation	Cassidy, N.	University of Surrey
14:45	Exhibitor	3D EBSD and EDS Developments	Larsen, K.	Oxford Instruments
15:05	Contributed	Combined laser and FIB preparation for TEM planar analysis of flash memory cells	Simon-Najasek, M.	FhG IMWS
15:25		Coffee Break		
16:20	Contributed	How FIB induced artefacts influence in situ characterization in the TEM	Berthier, M.	Protochips EMEA
16:40	Contributed	Development of a new integrated instrument for accurate and reproducible physico-chemical characterisation of nanoparticles	De Castro, O.	LIST
17:00	Contributed	TOF-SIMS with highest lateral resolution by pulsing the Ne-GFIS in a HIM	Klingner, N.	HZDR
17:20	Contributed	Combination of the FIB-TOF-SIMS technique with GIS – increasing the ionization probability and sputtering rates of thin films	Priebe, A.	EMPA
		Conference Dinner		
		EuFN Workshop Day 3 (June 14th)		
9:00		Get Together		
9:15	Invited	Ultrastructural changes accompanying the intracellular mineral formation in alga E. huxleyi': a cryo-FIB/SEM study	Bertinetti, L.	MPIKG Potsdam
9:50	Contributed	Avoiding amorphization during ion beam irradiation and critical dimension reduction of nanostructures	Hlawacek, G.	HZDR
10:10	Contributed	Ultra-fast growth of W-C metal nanostructures by Focused Ion Beam Induced Deposition under cryogenic conditions (cryo-FIBID)	De Teresa, J. M.	University of Zaragoza
10:30		Coffee Break		
11:00	Contributed	Using FIB as a broad ion source for nanofabricaton on AIII-BV(InSb) semiconductors	Jany, B. R.	Jagiellonian University
11:20	Contributed	Creating mesoscale ballistic transport devices from ultra-pure quantum materials	Bachmann, M. D.	MPI-CPFS Dresden
11:40	Contributed	Out-of-plane Transport in ZrSiS, ZrSiSe, and HfSiS Microstructures	Shirer, K. R.	MPI-CPFS Dresden
		Concluding Remarks		
		Lunch		

3rd Workshop of the
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Exhibitors

June 12th - 14th, 2019
Dresden, Germany

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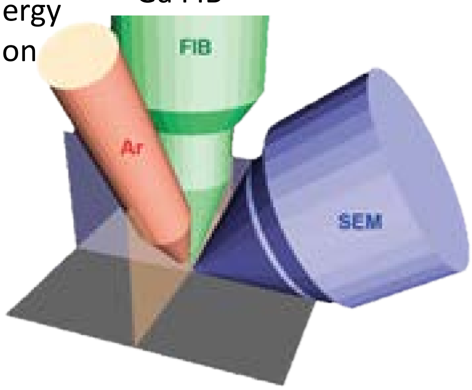

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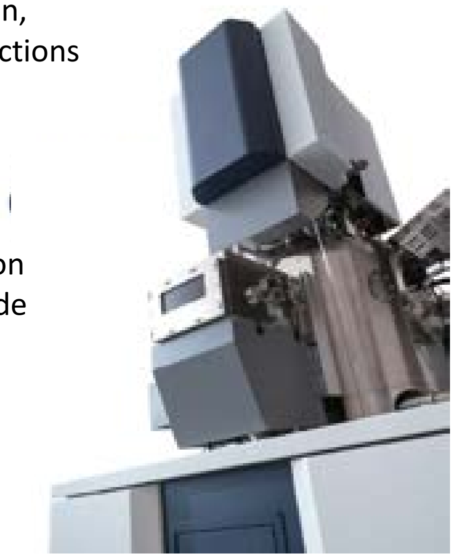

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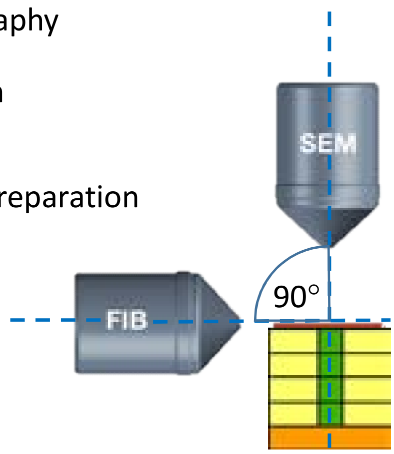

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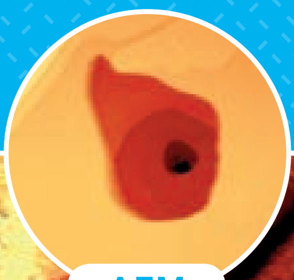
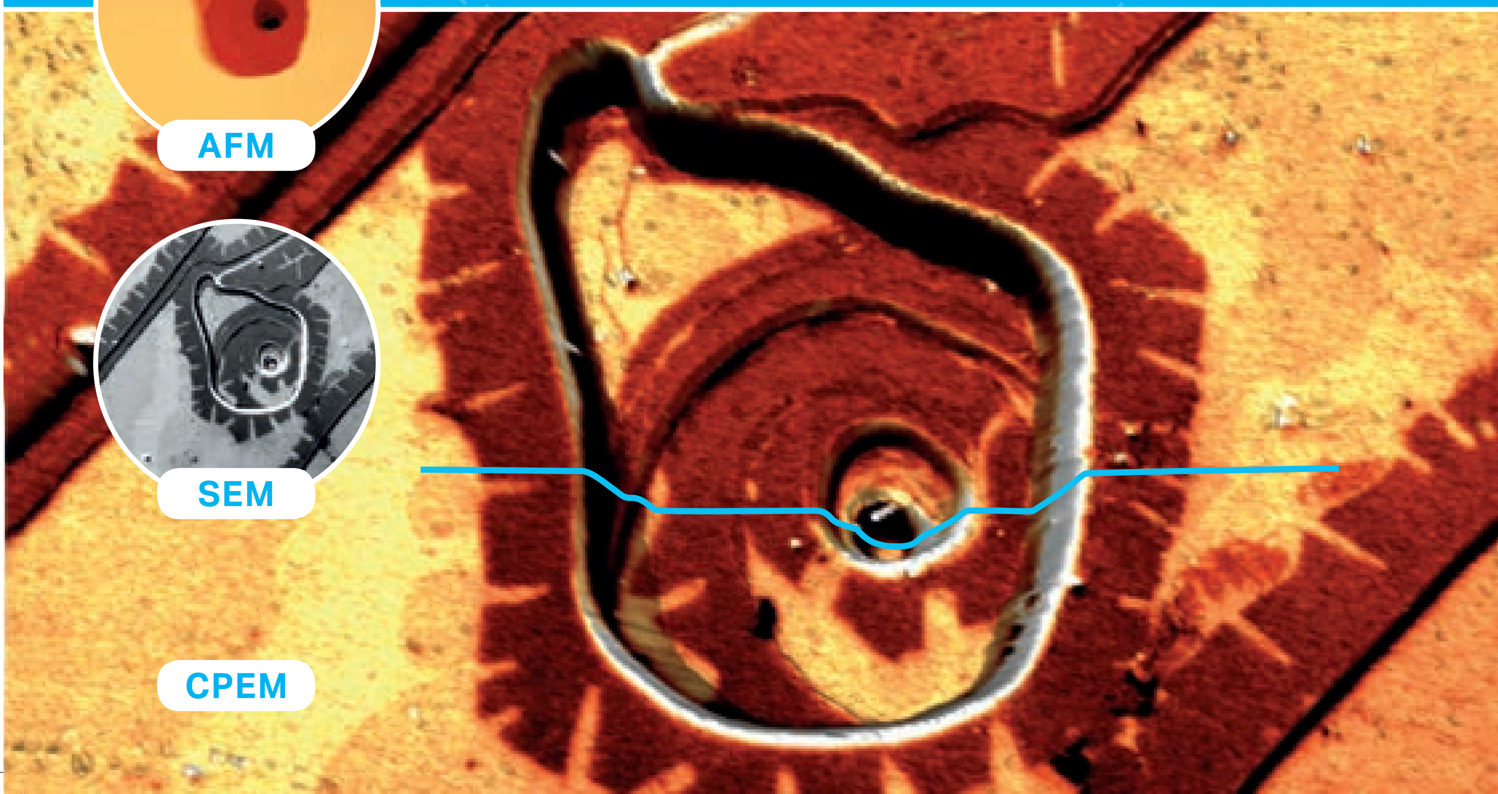
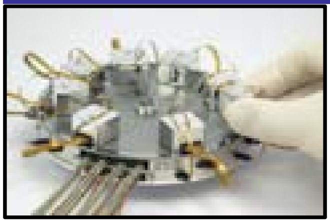




Best Selection for in-situ testing



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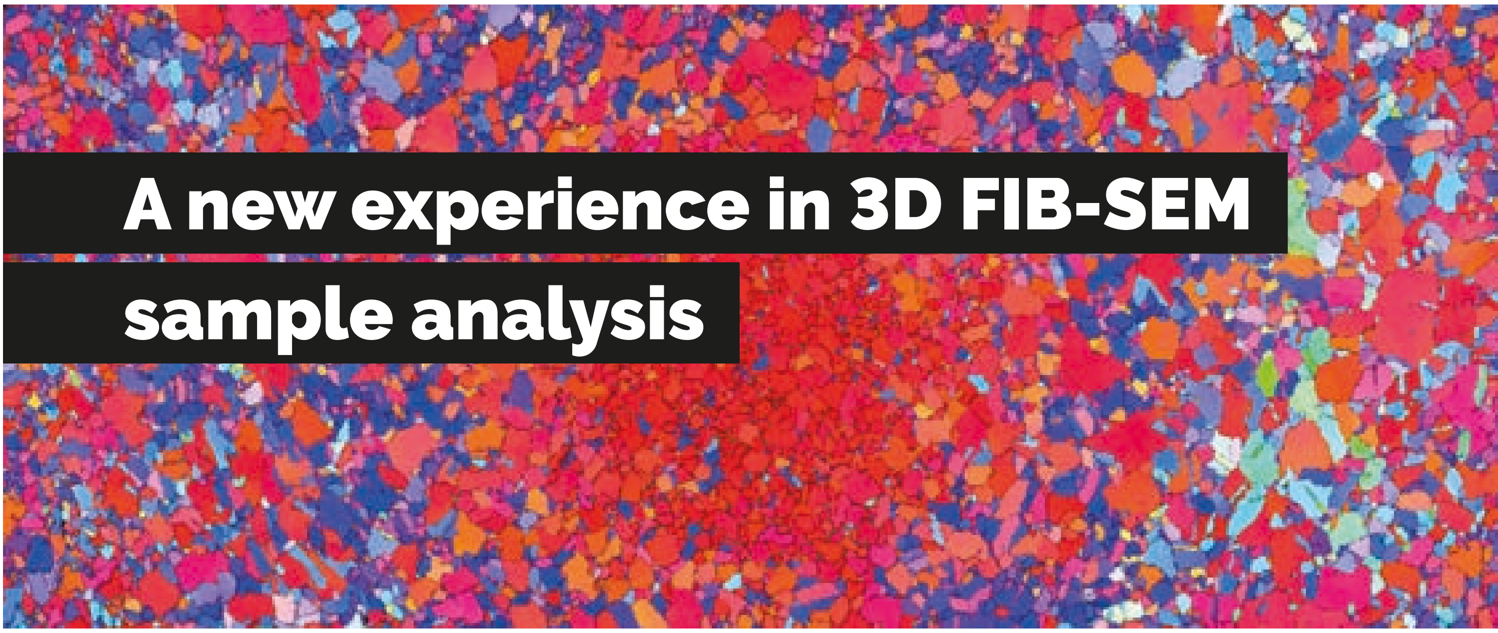

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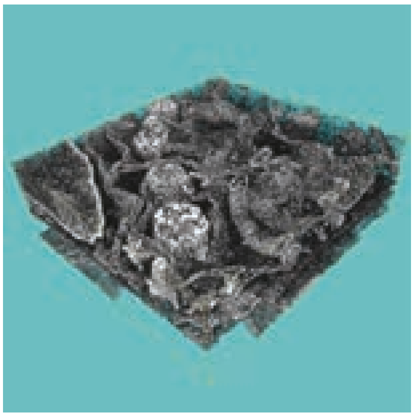
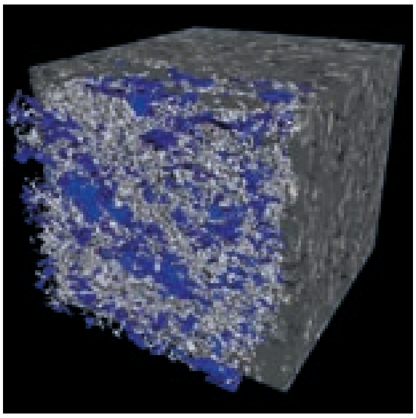
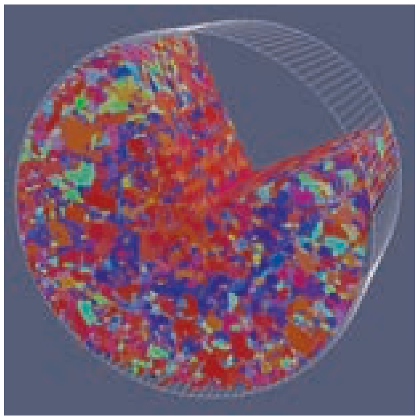


3D ultrastructural reconstruction of mammalian cell. Volume approx. 6 x 8 x 6 μm^3. Sample courtesy of Dr. Xuejun Sun, Dept. of Exp. Oncology, Cross Cancer Institute, University of Alberta, Canada.



3D reconstruction of a SiAlON-graphene sample. Volume approx. 22 x 22 x 67 μm^3. Sample courtesy of Professor Servet Turan from Anadolu University.



3D EBSD visualization of a 90 µm diameter copper wire.

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3rd Workshop of the
European Focused Ion Beam Network

Oral Presentations

June 12th - 14th, 2019
Dresden, Germany

Multigas Plasma FIB (from cell biology to lens manufacturing)

Alex de Marco[1]*

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Inductively coupled plasma ion sources have multiple advantages, among those we can recall high brightness, flexible beam chemistry and long term stability. In my lab we have tested the first prototype of a dual-beam (SEM-FIB) equipped with a plasma source which can be optimized for different ion species. Here, the ability to rapidly switch between different beam chemistries provides the unique chance to optimally work on multiple materials.

I will present the use of oxygen, xenon and argon plasma in cell biology applications such as fast FIB/SEM tomography. Further I will show the advantages of an optimized beam chemistry when milling frozen hydrated samples. Next, I will show the advantages of using plasmas on high precision optical manufacturing of micro-lenses for optical microscopy, and on microfabrication on materials such as lithium niobate and diamond.

in situ TEM Lamella LiftOut for Backside Preparation - LO&Flip

M. Kemmler[1], A. Rummel[1], K. Schock[1] and S. Kleindiek[1*]

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Enhancing the speed of failure analysis workflows can be one of the main drivers for the development of novel techniques. In the case described here, we are exploring a method for preparing lamella for TEM analysis of defects on semiconductor products that are buried several µm beneath the sample surface.

Due to curtaining effects and time constraints, it is desirable to extract a chunk of material from the bulk sample that includes the area of interest and flip it upside down so that the target site is then located close to the (new) top of the sample (Fig. 1). This significantly reduces the time it takes to thin the relevant section of the chunk down to transparency and polish the surface for TEM analysis.

One approach is to use a four axis micromanipulator equipped with a microgripper, the chunk milled from the sample bulk can be extracted, rotated by 180° and attached to a TEM half-grid for further processing (Fig. 2).

Alternatively, a three axis micromanipulator equipped with a microgripper can be used to transfer the sample to grid which is in turn mounted to a horizontal rotation axis. After transfer, the entire TEM grid can be rotated by 180° and the lamella can be processed for TEM analysis. This approach requires a slightly different TEM grid geometry to allow beam access to the sample.

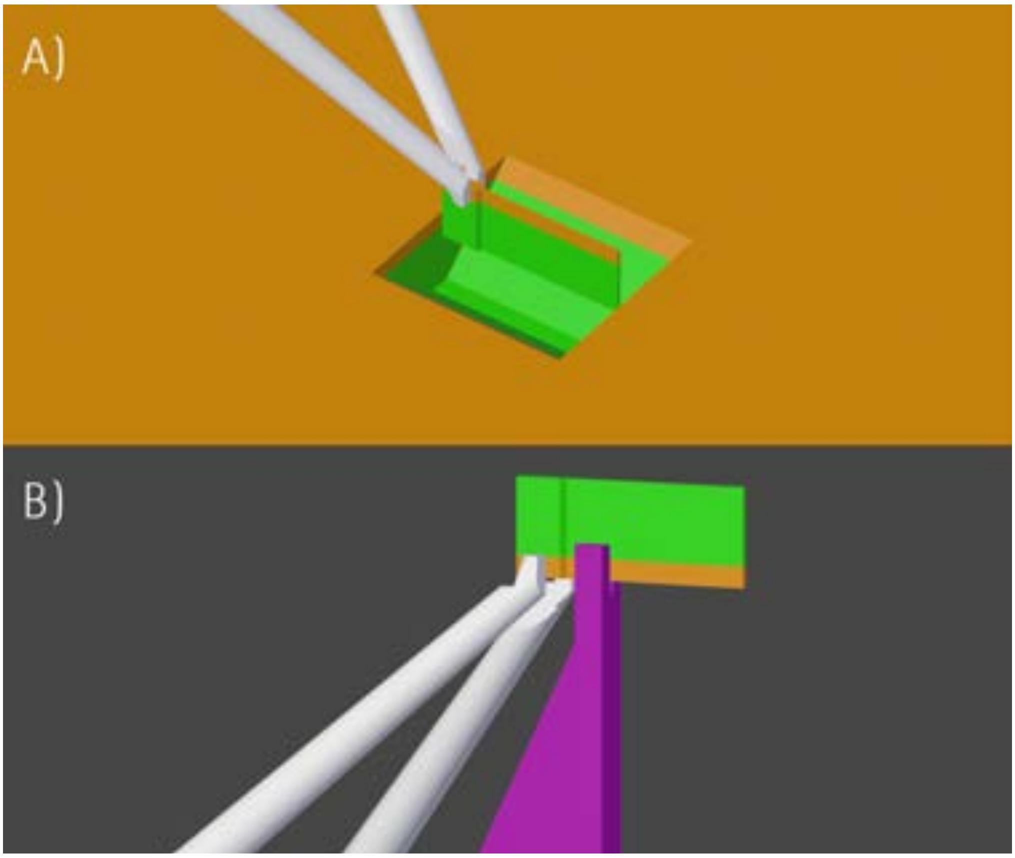

Fig. 1: A) retrieving the lamella.   B) Lamella flipped upside down and attached to TEM grid.

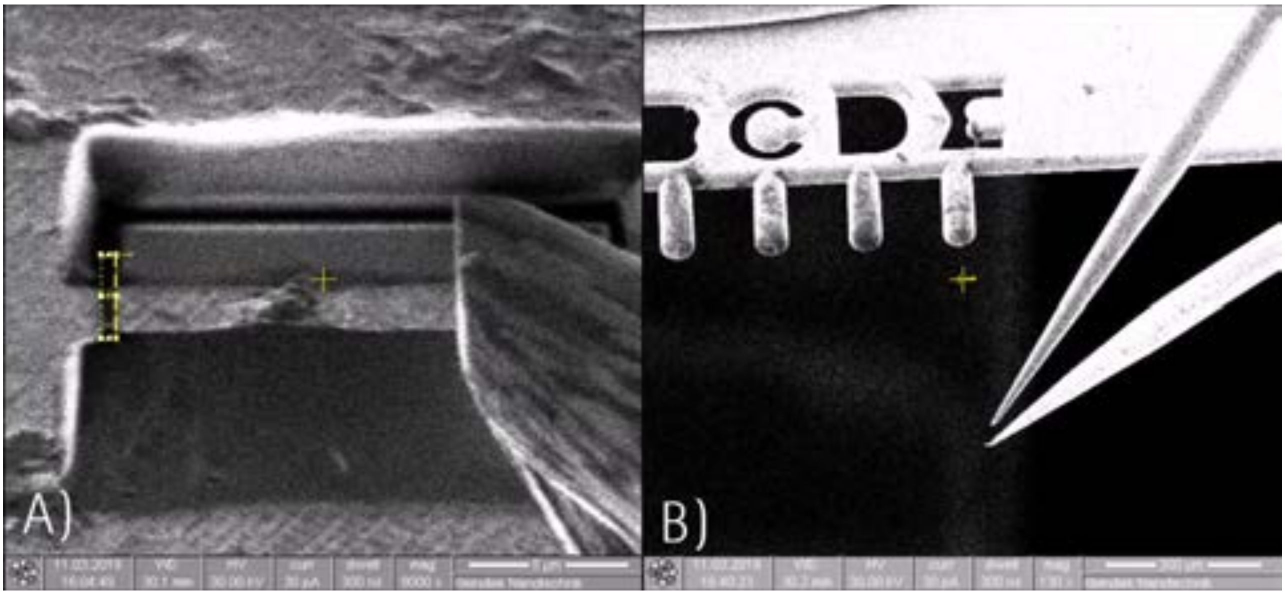

Fig. 2: A) Lamella liftout using a microgripper.   B) Approaching the half-grid after flipping the lamella upside down

FIBing Gallium Arsenide (and others): experiments for understanding and limiting artefacts

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A. Schertel[3], F. Pérez-Willard [3], G. Audoit[1]

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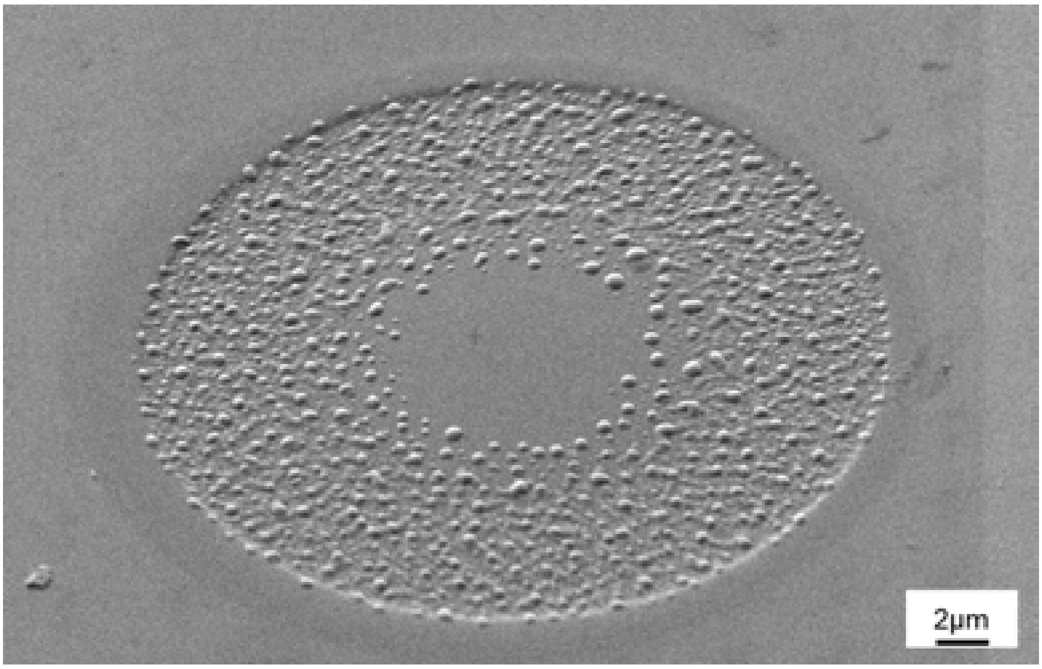
[2] Univ. Grenoble Alpes, CNRS, Grenoble INP, Institut Néel, 38000 Grenoble, France

[3] Carl Zeiss Microscopy GmbH, D-73447 Oberkochen, Germany

III-V and II-VI materials such as GaAs, GaSb and InP are now widely used in semiconductor based devices for microelectronics, optoelectronics or even 3rd generation photovoltaics. Preparing such samples by FIB (TEM lamella, cross section) often results in the formation of artefacts that are sometimes referred to Gallium precipitates or aggregates on the side of the FIB cut. The published literature suggests a local increase of the temperature under the beam, modifying the material composition according to the binary phase diagram. We are presenting experimental results from different approaches to try to understand better this phenomenon and to prevent the formation of this artefact: low kV FIB milling (Xe and Ga), cryo Ga-FIB, Gaz (XeF_2) assisted ion milling and Argon milling.



Artefacts resulting from FIB milling on GaAs

Preparation of TEM samples and their observation by means of an adjustable tilt holder

T. Hrnčíř[1*], P. Doležel[1] and M. Šikula[1]

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This contribution refers to the preparation and observation of transmission electron microscopy (TEM) samples by using the focused ion beam (FIB). All the process is performed in-situ, without breaking the vacuum and without removing the sample out of the chamber. Scanning electron microscopy (SEM) is used for the observation during the preparation process. The common approach starts by cutting out a small portion of the analyzed sample with FIB. This portion is then transferred on the TEM specimen holder by performing in-situ lift-out process. Finally, it is thinned with FIB to the desired final thickness on this holder, making a thin and electron transparent lamella. This lamella can then be observed in-situ by using retractable transmission electron (R-STEM) detector, or transferred for more detailed investigation into a dedicated TEM instrument. However, there are several possibilities how the lamella could be oriented towards the original sample or how the lamella could be treated during or after the lift-out process. Sometimes it is necessary to prepare the lamella perpendicular to the original sample surface (plain lamella), sometimes it should be parallel to the original surface (planar lamella). Moreover, plain lamella should be sometimes rotated to the opposite side, to get FIB access from its bottom side (inverted or back-side lamella). This is a must for front-side semiconductor samples, where the final polishing from the back side (silicon side) gives much better quality of the lamella and allows to prepare much thinner lamellae containing only single transistor rows. Sometimes it is even necessary to prepare another lamella crossing that thin lamella perpendiculary (cross-lamella), to focus on a certain single transistor feature only. For all of these lamella preparation types, the lamella must be rotated in a special way so that it is accessible for FIB thinning from its different sides. To make things even more complex, certain position of the lamella is necessary to perform the final thinning by FIB and to precisely control the end-point, where FIB beam and lamella are almost parallel. Another position is needed to observe the progress of the work e.g. with R-STEM detector, where ideal direction of SEM beam for observation is being perpendicular to the plane of the lamella. Another limitation for backside sample preparation is the necessity of having the nanomanipulator with rotary tip, while the preparation geometry (stage and tip positions) can be quite complex.

We have solved this complex problem by developing a novel TEM sample holder. TEM sample is placed on TEM grid, which is attached to a bracket with adjustable rotation angle. This rotation angle can be varied for different stages of the lamella processing, such as the FIB thinning, the backside thinning or SEM / R-STEM observation. In addition, in the position of the lamella holder for the FIB thinning and for the backside thinning, we can easily check the quality of FIB machining by SEM observation and R-STEM detector or other detectors by changing the main stage tilt. Similarly, by the appropriate change of the angles of the main stage tilt and the rotation of TEM sample holder, preparation and observation of the lamella parallel to the surface of the original sample (planar lamella) can be performed. The great advantage of this solution is the simplicity of TEM sample preparation process, the frontside – backside – planar – SEM / R-STEM observation flexibility, and the fact that there is no need to use a rotary tip nanomanipulator to obtain backside thinning direction.

Channeling Effects in Gold Nanoclusters under He Ion Irradiation: A Molecular Dynamics Study

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[2] Department of Applied Physics, Aalto University School of Science, P.O. Box 11100, 00076 Aalto, Finland

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Ion channeling is a well-known effect in ion irradiation processes, which is a result of particles moving between the rows of atoms. It drastically affects the ion distribution, ion energy-loss and consequently the damage production in the target. Therefore, one could derive the ion-channeling pattern out of the energy-loss behavior of ion-target interaction.

The ion channeling effect is studied for a few pure element crystals and also for some compounds in a systematic way [1]. In this work, we focus on nano-structures which are of major importance, due to their high surface-to-volume ratio, wide spread application and possible toxicological effects. Our results, for different gold cluster sizes and few keV He ion irradiation, show that ion-channeling occurs not only in the principal low-index, but also in other directions in between. The strengths of the different channels are specified, and their correlations with sputtering-yield and damage production is discussed, along with the size-dependence of these properties. The effects of planar defects, such as stacking faults on channeling were also investigated. Finally, we discuss the implications of our results for the analysis of HIM images of metal clusters.

[1] Nordlund, K., and G. Hobler. "Dependence of ion channeling on relative atomic number in compounds." Nuclear Instruments and Methods in Physics Research Section B: Beam Interac- tions with Materials and Atoms (2017).

This work is part of the npSCOPE project and has received funding from the European Union's Horizon 2020 research and innovation program. Grant number: 720964.

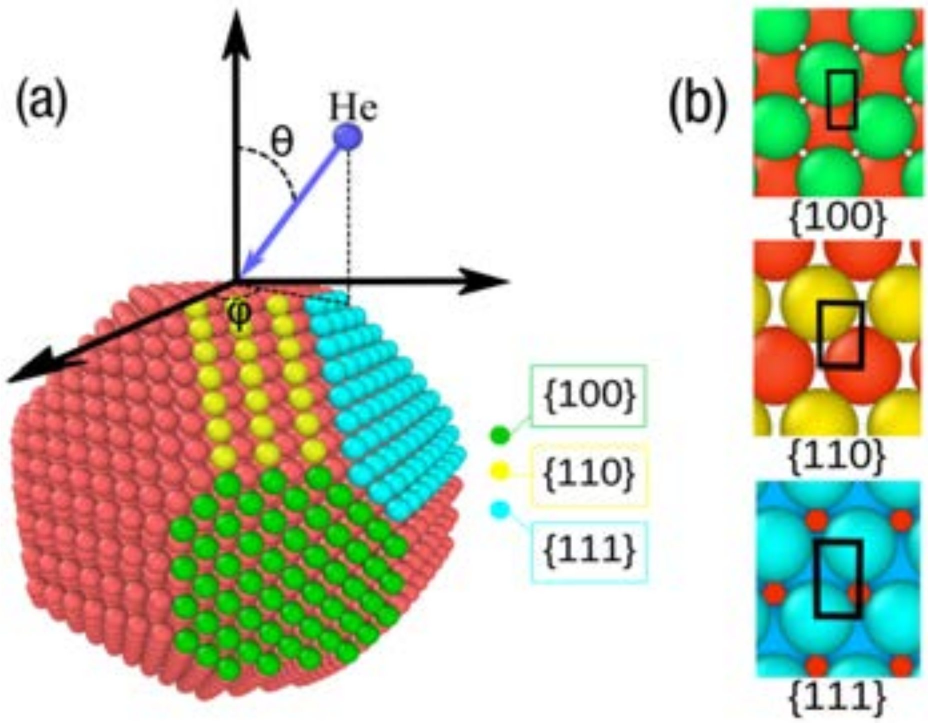

Fig. 1: (a) Atomistic structure of a gold nano-cluster with a diameter of 5 nm.
High symmetry faces and incident ion angles are shown. (b) Irreducible areas used in the ion impact simulations for {100}, {110}, and {111} surfaces.

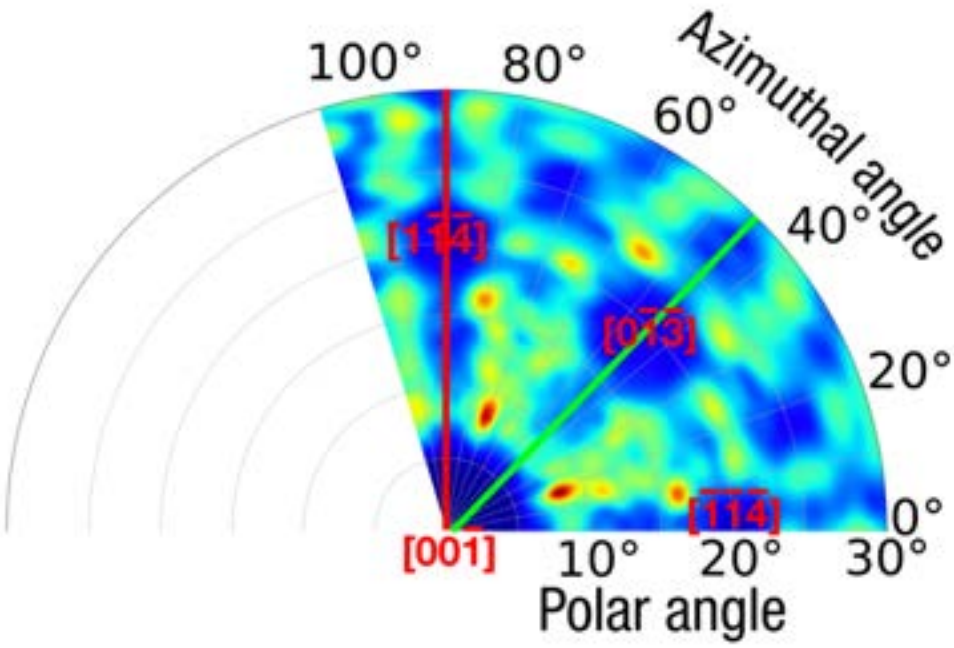

Fig. 2: Channeling map for a gold cluster with a diameter of 10 nm.

Microstructure is the “know-it-all” - classification approaches based on 3D-tomography, data mining and deep learning methods

Frank Mücklich[1,2] and Dominik Britz[2]

[1] Universität des Saarlandes, Saarbrücken, Germany
[2] Material Engineering Center Saarland, Saarbrücken, Germany

The microstructure can be regarded as the multi-scale archive from which we can “read” the quantitative information about the microstructure formation processes and the prediction of the final material properties on each relevant scale. Recent advances in 3D tomography methods on the micro, nano and atomic scale allow to study the differences of microstructures with higher morphological and topological complexity. Classification strategies using Support Vector Machine and Deep Learning will be discussed using morphological and substructure parameters. Images are processed directly in the workflow after an adapted contrasting. The result might be simultaneously segmented and classified.

References

S. M. Azimi, D. Britz, M. Engstler, M. Fritz, and F. Mücklich, Advanced Steel Microstructural Classification by Deep Learning Methods, Scientific Reports (Nature) 8 (2018) 2128.

AFM-in-SEM LiteScope™: Tool for in-situ analysis of FIB modified materials

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[2] Central European Institute of Technology (CEITEC), Brno, Czech Republic
[*] corresponding author email: jan.neuman@nenovision.com

New types of materials and new ways to produce them are desired by many scientific fields. Direct writing of magnetic patterns by FIB irradiation has various advantages in comparison with conventional lithography approaches. It allows rapid prototyping of a large variety of nanostructured samples without the need for further sample processing [2]. The possibility of in-situ analysis of created structures is very useful. Novel AFM instrument LiteScope™ with unique Correlative Probe and Electron microscopy (CPEM) technique is presented. It offers advantages of in-situ analysis of samples after FIB or GIS modification including depth/height profiling, roughness estimation, 3D imaging or local spectroscopy measurements.

The AFM microscope LiteScope™ is designed for easy and fast integration into a wide range of SEMs. It can be mounted directly onto the SEM stage and operated in a tilted position which makes it compatible with FIB etching. This brings an opportunity to investigate the sample right after the FIB surface modification without breaking the vacuum, moving the sample or without the ambient atmosphere influence. Since SEM is used for AFM tip navigation, no marks or other common tricks are needed to localize the area of interest for further analysis, even in nanoscale [1]. As mentioned earlier, LiteScope™ is equipped with CPEM technique for true correlative simultaneous measurement of AFM and SEM images.

This type of analysis will be shown on several examples including metastable $Fe_{78}Ni_{22}$ thin film grown on Cu(100) substrate irradiated by FIB. This material is a great candidate for magnetic patterning since it is paramagnetic (fcc) at room temperature and can be transformed by FIB irradiation to the ferromagnetic material (bcc), see Fig.1 [2]. LiteScope™ has been successfully used for in-situ advanced surface characterization of this material.

[1] NenoVision s.r.o., LiteScope™, NenoVision, 2018, www.nenovision.com/

[2] M. Urbanek, L. Flajsman, V. Krizakova, J. Gloss, M. Horky, M. Schmid, P. Varga; *Research Update: Focused ion beam direct writing of magnetic patterns with controlled structural and magnetic properties;* APL Materials 6 (**2018**).

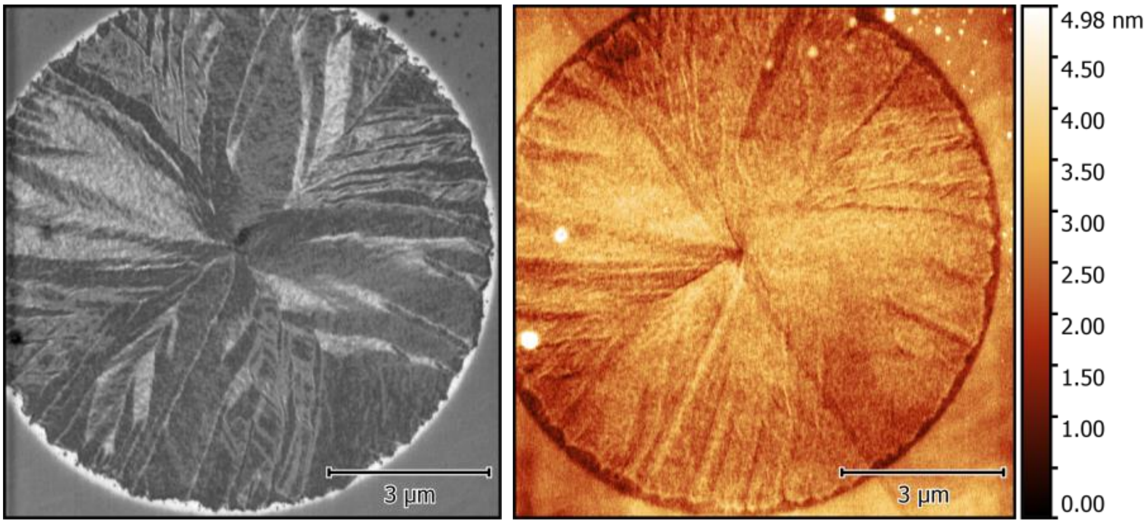

Fig. 1: Simultaneously collected SEM and AFM image of FIB modified $Fe_{78}Ni_{22}$ thin film grown on Cu(100) substrate. The FIB irradiation changes the crystallography of the sample from face-centered cubic (fcc) Fe into body-centered cubic (bcc) Fe.

In-Situ Correlative AFM/SEM/FIB analysis of FIB-treated samples

P. Frank[1], S. Hummel[1], J. Sattelkov[3], R. Winkler[3], S. Andany[2], G. E. Fantner[2], H. Plank[3], C. H. Schwalb[1,*]

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[2] Ecole Polytechnique Fédérale de Lausanne (EPFL), Laboratory for Bio- and Nano-Instrumentation, Institute for Bioengineering, CH-1015 Lausanne, Switzerland
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During the last decade the combination of different microscopic and spectroscopic methods into one instrument gained increasing importance due to the simultaneous acquisition of complementary information. Especially highly localized probing of mechanical, electrical, chemical and crystallographic properties on the nanoscale represents a key success factor for gaining new insights in the micro and nano world.

We present a unique atomic force microscope (AFM) – the AFSEM™ - designed for seamless integration into scanning electron microscopes (SEM) or FIB systems (see Figure 1). Its open design and the use of self-sensing cantilevers with electrical readout allows for simultaneous operation of SEM, FIB and AFM inside the vacuum chamber to perform correlative in-situ AFM/SEM/FIB analysis of ion-beam treated nanostructured materials. We present correlative AFM/EBSD data of a FIB polished ZrO_2 ceramic of phase transformed regions. While EBSD allows for locally identifying areas where the phase transformation has occurred, in-situ AFM can now be utilized to analyze phase-transformation-induced topographic changes with sub-nm resolution. In a further step, we demonstrate how in-situ correlative analysis with the AFSEM™ in an SEM can be extended into the third dimension to measure nanomechanical properties of soft material. To achieve this, FIB slicing and mapping of nanomechanical properties using the AFSEM™ is performed in repetitive steps to build up a 3-dimensional elasticity map (see Figure 2). Finally, we present, for the first time, in-situ correlative AFM results of helium treated surfaces inside the Zeiss ORION Nanofab .

[1] M. Dukic, J. D. Adams and G.E. Fantner, Scientific Reports 5 (**2015**) 16393.

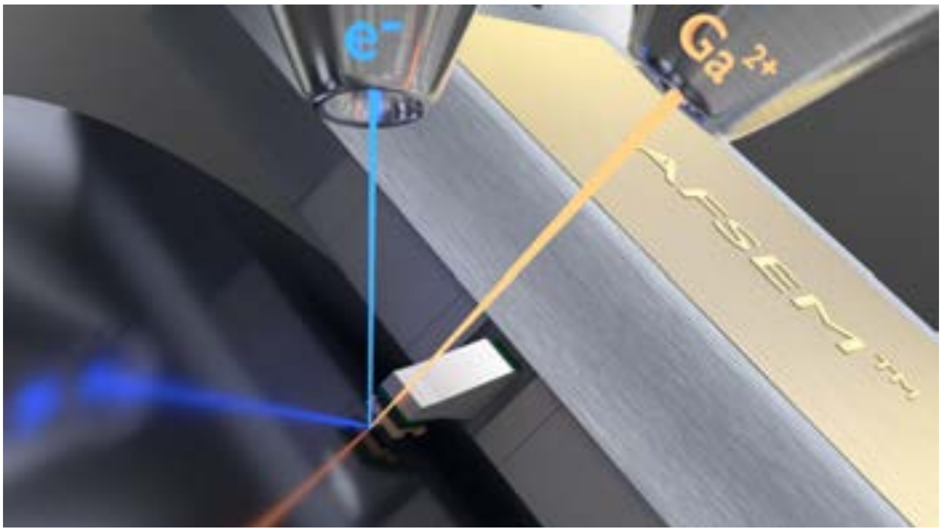

Fig. 1: Schematics of correlative analysis in a dual-beam system using SEM, FIB and AFSEM™ in an interactive experiment.

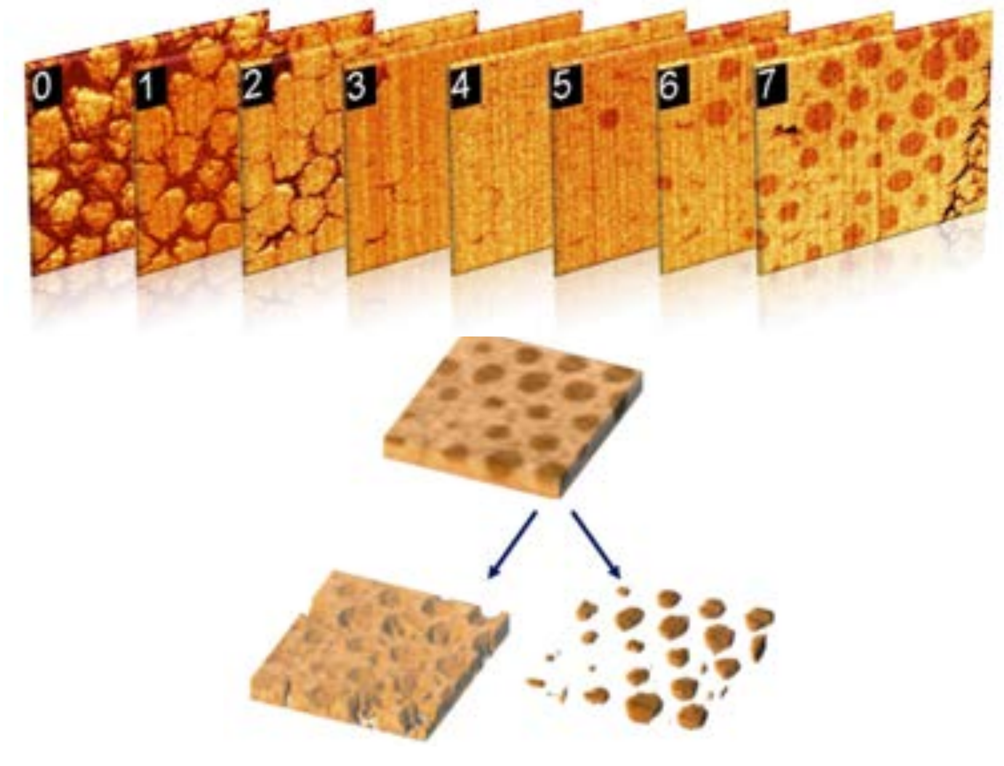

Fig. 2: (Top) Consecutive in-situ AFM elasticity maps of a polymer bead structure after different FIB slicing. (Bottom) 3D reconstruction of nano-mechanical properties of the ion-beam treated polymer bead structure.

Feature Adaptive Sampling for Fast Image Acquisition in FIB and SEM

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Conventional image acquisition in FIB/SEM instruments is realized by uniform line-by-line pixel scanning with a fixed pitch and beam dwell time. Two major problems can arise from this strategy. One is the question of dose in case of beam sensitive samples (e.g. polymers or organic material) where the operator is confronted with the conflict of acceptable signal-to-noise ratio versus dose limitations. The second problem is acquisition time. Techniques like large area automated mapping and FIB/SEM serial sectioning regularly record several hundreds of images. With increasing pixel resolution in modern instruments, the total image acquisition time could easily exceed ten hours and often a compromise is made in terms of actual resolution in order to keep instrument time low.

Here, we present feature adaptive image sampling techniques which greatly reduce image acquisition time and beam dose. In the adaptive dense sampling approach a low dwell time, full frame scan image is acquired from which regions of high contrast (e.g. phase boundaries) are identified. In a second step, a high dwell time, selective scan image is acquired from regions of high contrast as identified in the first step. The final image is composed from both scans. The second approach uses sparse random sampling to acquire a fast, low dose image. Iterative additional scanning is performed in regions of high contrast to obtain the relevant image information without having scanned the full field of view. Techniques like those allow a great reduction of image acquisition time while retaining the relevant details of the image [1].

[1] T. Dahmen, M. Engstler, C. Pauly, P. Trampert, N. De Jonge, F. Mücklich, P. Slusallek, *Feature Adaptive Sampling for Scanning Electron Microscopy*, Sci. Rep. 6 (2016) 25350. doi:10.1038/srep25350.

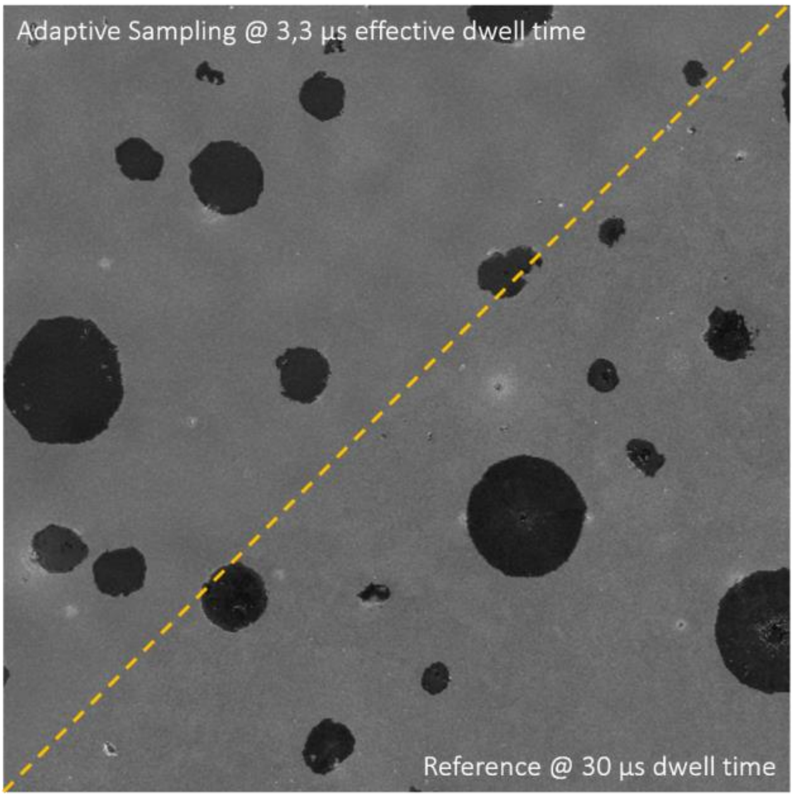

Fig. 1: SEM micrograph of nodular cast iron. Comparison between reference full frame image (lower right hand corner, 30 µs dwell time) and composed adaptive sampling image (upper left hand corner, 3.3 µs average dwell time). Note the equally well-resolved phase boundaries between graphite and matrix. 5 kV, 340 pA, Everhart-Thornley-Detector. Modified from [1].

Removal of Curtaining Effects by a Variational Model from Mathematical Image Processing

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[2]University of Kaiserslautern, Department of Mechanical and Process Engineering, D-67663 Kaiserslautern, Germany
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In the last two decades, focused ion beam systems have been used for sample preparation. For example, the edges of a sample can be polished for analytical measurements or continuous cross-sections can be milled for 3D tomography and reconstruction. One major challenge in both procedures is the so-called curtaining or waterfall effect, i.e., increasing surface roughness in the direction of the milling depth. A software-based solution to remove the curtaining effects after the milling procedure is presented. More precisely, a novel variational model from mathematical image processing is proposed. Besides the curtaining effect, additional artifacts such as discontinuities caused by redeposition of previously removed material or charging effects can be removed. The method splits the input image stack into a clean part and two types of corruptions, namely a striped and a laminar part. Fig. 1 shows exemplary results for one slice of a 3D dataset. The method is applied to the entire 3D dataset, and distortions are reduced by using information of their particular structure and directional dependence. As a result, even highly corrupted images can be used for further analysis.

[1] J. H. Fitschen, et al.; *Removal of curtaining effects by a variational model with directional forward differences*; Computer Vision and Image Understanding (**2017**), 155:24–32.

[2] T. H. Löber, et al.; *Reducing curtaining effects in FIB/SEM applications by a goniometer stage and an image processing method;* Journal of Vacuum Science & Technology B, Nanotechnology and Microelectronics: Materials, Processing, Measurement, and Phenomena (**2017**), 35.

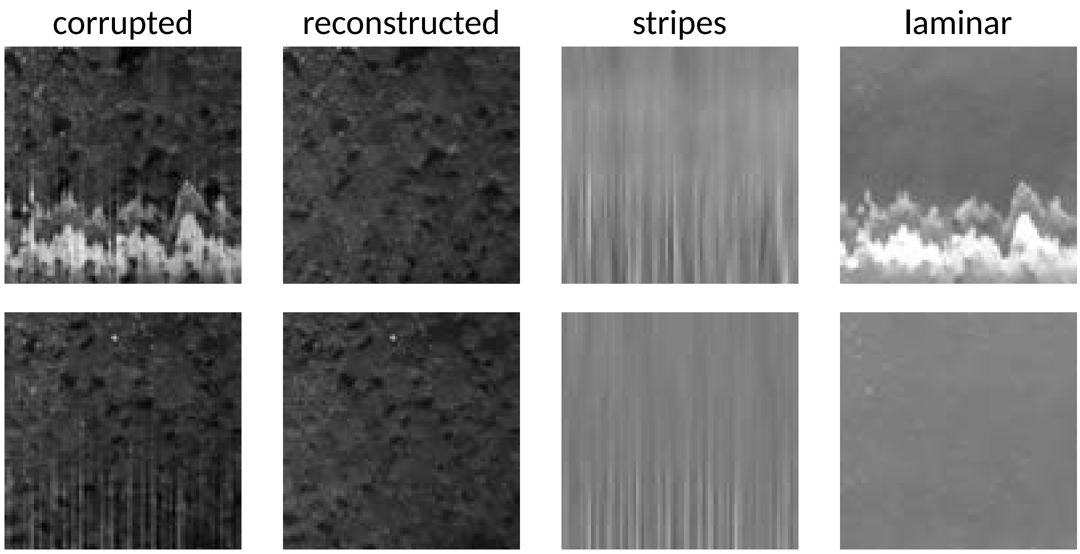

Fig. 1: Results of the method for two exemplary slices of FIB data.

Why FIB is essential for analysis in semiconductor industry

Pascal Limbecker[1], Enrico Vales[1], Moritz-Andreas Meyer[1]

[1] GLOBALFOUNDRIES Dresden Module One Limited Liability Company & Co. KG, Dresden

In today's failure analysis world in the field of semiconductors, the use of Dual Focused Ion Beam (FIB) systems is indispensable, since they combine FIB and Scanning Electron Microscopes (SEM) and therefore open a wide range of applications. Typical applications are SEM cross sectional and TEM sample preparation for process monitoring in manufacturing, physical failure analysis, chip modification (circuit edit) and structural analysis. Conventional preparation methods like manual polishing and optical microscope imaging are not sufficient anymore for analyzing structures of most recent technology nodes down to 7nm. High magnification and resolution imaging of transistors e.g. can only be done by transmission electron microscopy (TEM). Therefore the preparation of ultra-thin TEM lamellas with a thickness <50nm is required and done with FIB. Physical Failure Analysis at technology nodes below 100nm is unthinkable without a FIB/SEM tool, when searching for tiny defects. FIB is used to get cross sections of exactly defined locations on a chip. In leading edge applications FIB is used as a surgical tool to modify or repair integrated circuits to change their electrical behavior. In another application FIB is used to characterize the grain structure and orientation of materials like copper and aluminum by using channeling contrast of the FIB. In my presentation I want to show how we use FIB/SEM tools in our daily work in the central lab of GLOBALFOUNDRIES, special applications and challenges.

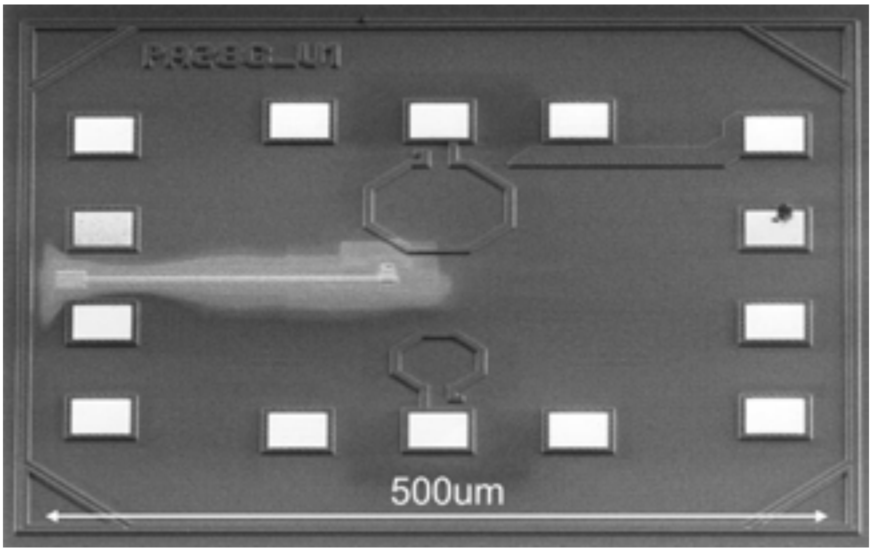

Fig. 1: Chip modification: probe pad added with FIB assisted platinum deposition

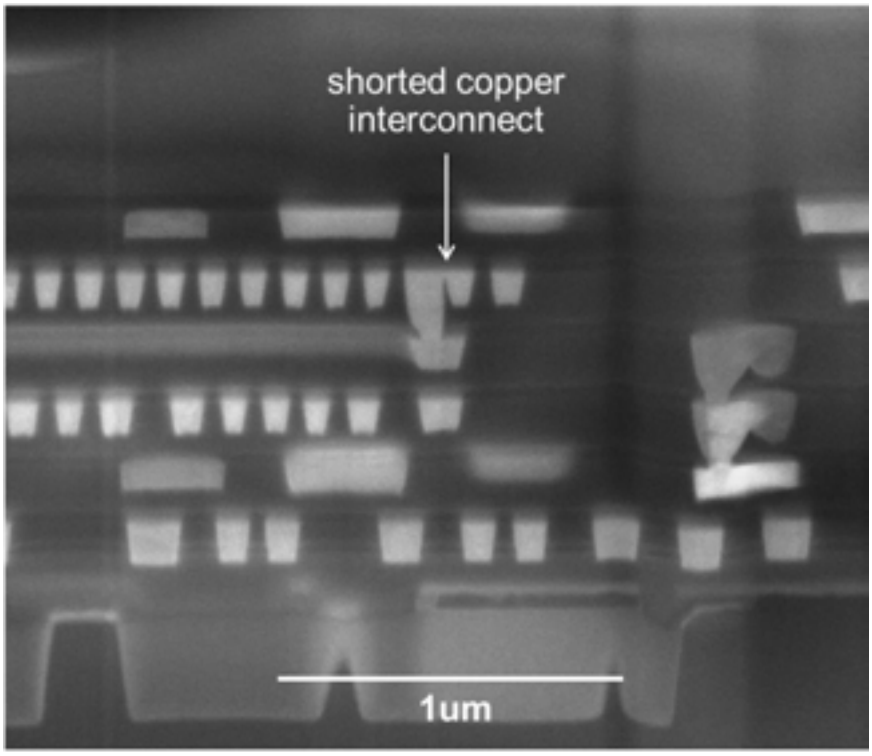

Fig. 2: FIB/SEM cross section: PFA with slice & view

Latest advances of JEOL`s JIB-4700F Multi Beam System in focused ion-beam lithography

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[1] JEOL (Germany) GmbH, Gute Änger 30, 85356 Freising, Germany
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JEOL is a world leading manufacturer of electron- and ion-beam systems. The compa-ny's portfolio includes lithography tools using both electron and ion beams as well as characterization systems like Scanning Electron Microscopes (SEM) and Transmission Electron Microscopes (TEM).

Advances in materials and life sciences necessitate an increasing performance of the imaging, analytical and preparatory capabilities of modern focused ion beam (FIB) sys-tems. The new JEOL JIB-4700F multi beam system is a highly stable yet flexible plat-form to encounter a variety of applications ranging from morphological and chemical studies to the crystallographic analysis of a wide variety of materials. In addition to the standard load-lock system for high sample throughput, the high-performance electron and ion emitters ensure a fast and easy sample characterization.

The JIB-4700F features a hybrid conical objective lens, GENTLEBEAMTM (GB) mode and an in-lens detector system to deliver a guaranteed resolution of 1.6 nm at a low ac-celeration voltage of 1 kV. Using an "in-lens Schottky-emission electron gun" that pro-duces an electron beam with a maximum probe current of 300 nA, this newly-developed instrument allows for high-resolution observation and fast analyses. For the FIB column, a high current density Ga ion beam of up to 90 nA maximum probe current is employed for fast ion milling and processing of samples. Concurrent with high-speed cross-section processing by FIB, high resolution SEM observations and fast analyses can be conduct-ed utilizing energy dispersive X-ray spectroscopy (EDS) and electron backscatter dif-fraction (EBSD). Besides the standard feature of a three-dimensional analysis function that automatically captures SEM images at certain intervals in cross-section processing, the JIB-4700F also provides "STEMPLING", an optional automatic TEM specimen preparation function. Owing to this function, it is now much easier to fully automate a reliable preparation of high quality TEM lamellae and significantly improve throughput.

Defect production in supported two-dimensional materials under ion irradiation from atomistic simulations: the substrate is crucial

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The response of two-dimensional (2D) materials to ion irradiation is interesting from a fundamental point of view - understanding the damage mechanism in a system with reduced dimensionality - as well as for potential applications e.g. device engineering using focused ion beams such as helium ion microscope (HIM). Here, we use molecular dynamic simulations combined with a Monte Carlo method to investigate effects of ion irradiation on single layer MoS_2 supported by SiO_2 substrate [1]. Depending on the ion energy and ion type the main damage mechanisms can be characterized as direct sputtering by the impacting ion or indirect sputtering, which originates from backscattered ions and sputtered substrate atoms. Specifically, for typical HIM energies, we show that indirect sputtering dominates the direct sputtering, so that the model of free-standing material cannot be used.

[1] S. Kretschmer, et. al. *Supported Two-Dimensional Materials under Ion Irradiation: The Substrate Governs Defect Production*; ACS Appl. Mater. Interfaces 10 (2018), pp 30827–30836

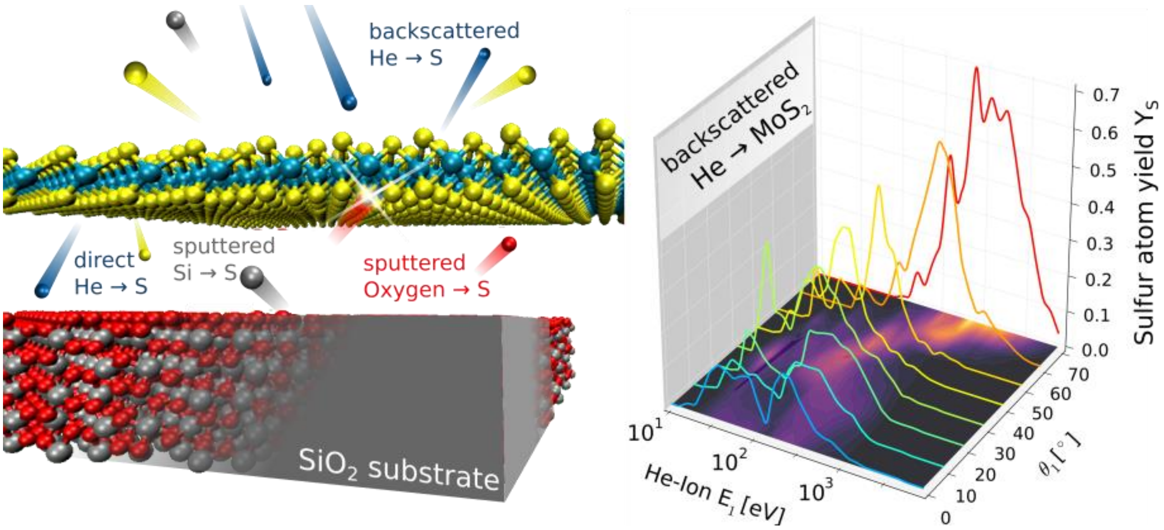

Fig. 1: Visualization of defect production channels for 2D MoS_2 supported by SiO_2 substrate.

Advances in Multiple Ion Species Plasma FIB Technology

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Commercially available ion source technologies for FIB-SEM systems continue to develop, with Ga^+ liquid metal ion source (LMIS) being available already for a while and the more recent Xe^+ inductively couple plasma (ICP) source. LMIS Ga^+ FIB technology offers a ion beam that provides high resolution capability for nanoprototyping and offers enough current for S/TEM sample preparation of most samples in less than an hour as well as FIB serial sectioning tomography (SST). On the other hand, Xe^+ PFIB technology has a much larger beam current capability allowing researchers to investigate significantly larger volumes. Additionally, xenon ions offer the user the ability to prepare gallium-free S/TEM samples or cross-sections.

In this presentation the latest generation of Thermo Fisher's plasma FIB instruments, which is now supporting multiple ion species as a primary ion beam, will be introduced. Helios Hydra PFIB provides researchers with 3 additional ion species in addition to xenon – argon, oxygen and nitrogen. A single ion source can deliver all 4 ion species independently with a patented, automated, fast and easy switching capability. Access to multiple ion species "on-the-fly" will significantly expand the FIB's application space in addition to providing advantages to various use cases. For example, final low energy Ar^+ polishing of S/TEM samples to improve contrast in HR-STEM.

An Improved Experimental Setup for the Preparation of Back Side and Planar View TEM samples

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Over the last two decades manufacturers have continuously improved their FIB-SEM instruments. Tasks that in the past required a high operator skill level are routine today. This is the case for the preparation of high-quality site-specific TEM samples in the so-called front side geometry. Other workflows for the fabrication of back side or planar view lamellas are more complex and remain by far less common although they offer important advantages for some applications. In this contribution we will review these “exotic” workflows and present a simple experimental setup to streamline them.

All workflows above have four steps in common: First, the navigation and localization of the site of interest. Second, the preparation of an at least 1µm-thick lamella, the chunk. Third, the in-situ lift-out. And finally, the thinning of the chunk to electron transparency. Depending on the orientation of the chunk relative to the bulk sample and the grid, we distinguish between front side (or standard cross section), planar view and back side preparation (see Fig. 1).

The planar view preparation aims at fabricating a lamella originally parallel to the bulk sample surface.

The back side workflow targets a cross section lamella mounted upside down on the grid, so that thinning can happen from the bottom. This can be useful to avoid curtaining, bring the region of interest closer to the lamella top or avoid thinning through soft layers.

Planar view and back side recipes require flipping the grid between upright and horizontal positions. Our new experimental setup allows users to accomplish this in a fast and simple way.

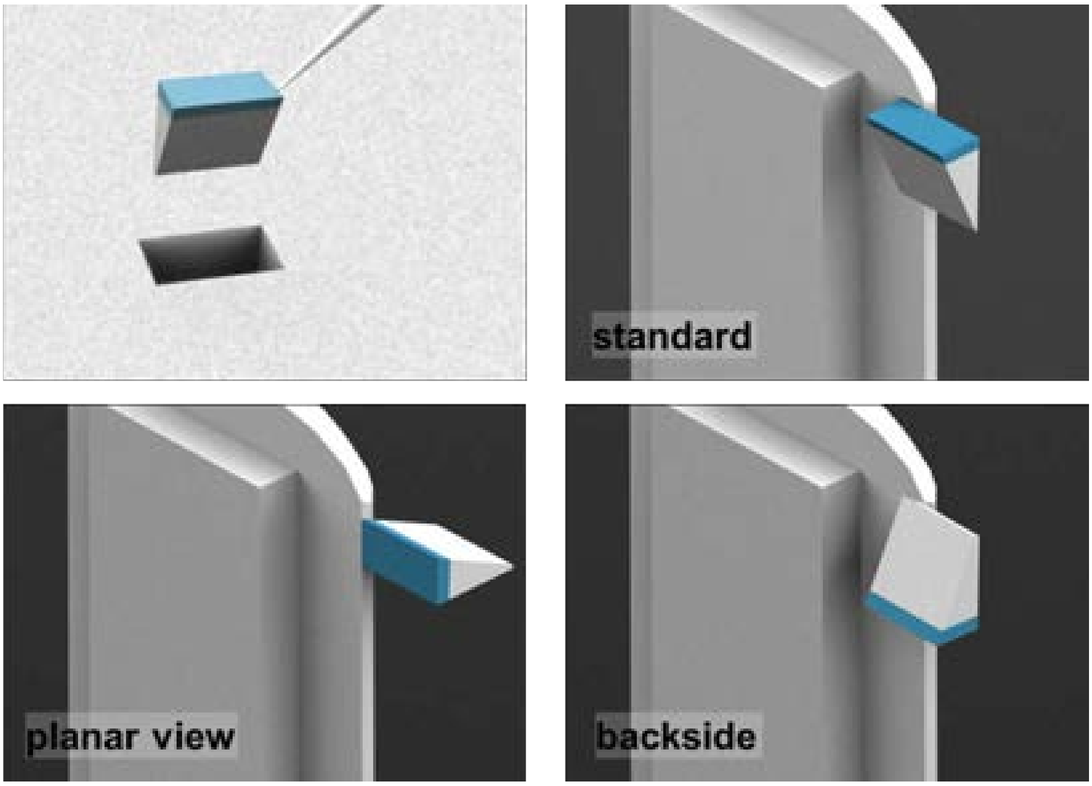

Fig. 1: Chunk and different lamella geometries (schematics courtesy of Oxford Instruments).

Site-specific Atom Probe Tomography Sample Preparation Method by Orthogonal FIB-SEM Column Layout

Yoshihisa Orai[1], Xin Man[2], Jamil J. Clarke[3] , and Tsuyoshi Onishi[1*]

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Recently, advanced semiconductor devices are composed of very small 3D structures, requiring very high sensitivity 3D elemental analysis for device quality evaluation and failure analysis. Atom Probe Tomography (APT) is known as a suitable analytical method corresponding to these needs.

The procedure for APT preparation requires a combined Focused Ion Beam and Scanning Electron Microscope (FIB-SEM) system for lift out and pillar shaping to process semiconductor devices to a thickness of less than 100nm in diameter, thereafter the specimen is introduced into the APT system. Key requirements here are to position the analytical target of interest at the topmost area of the pillar accurately, shape the pillar top uniformly, with the added factor as to not leave any FIB deposited metalized protection material on the specimen surface. This specimen preparation process is very challenging by traditional FIB-SEM systems as the success rate for process quality and accuracy is not entirely repeatable which often tends to rely on operator skill.

This presentation will demonstrate an effective preparation method to produce high quality APT specimens with very high yield utilizing 2 different tilt axes STEM images for monitoring the internal specimen structure for precise FIB milling on by an orthogonally configured FIB-SEM system.

Fig. 1 shows the case this method was applied to a 22nm node SRAM device. Monitoring the internal specimen structure by tilting the specimen by +/-45 deg. enabled precise FIB milling to retain the analytical target at the pillar center successfully. The processed specimen was then isolated further by removing the protection layer and neighbouring gate structures. Fig.2 shows the analytical result from the APT system. Elemental distribution around the FIN area is mapped out very clearly.

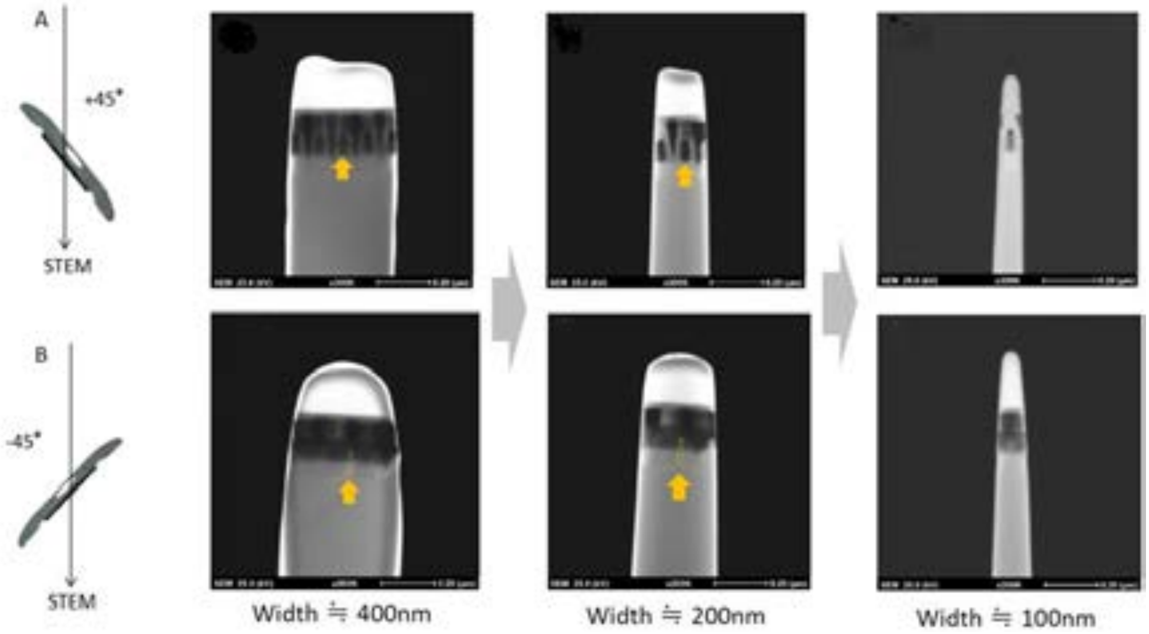

Fig.1 FIB milling using STEM images from 2 different tilt angles.

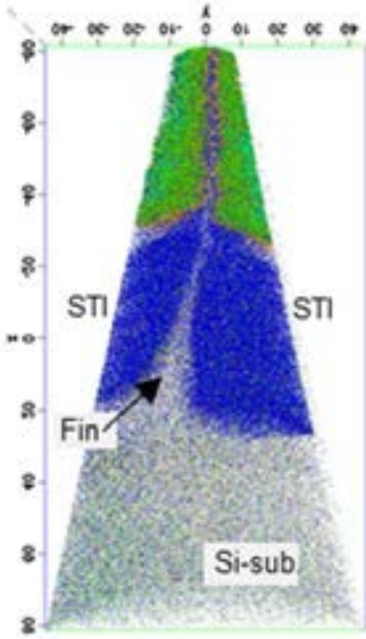

Fig.2 APT Atomic Map of p-type FinFET

Ion Sources for Focused Ion Beams – Present Status and Prospective Developments

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Focused Ion Beam (FIB) processing has been developed into a well-established, irreplaceable and still promising technique in nearly all fields of nano-technology in particular for direct patterning and proto-typing on the µm scale and well below as well as sample preparation for further investigations, using SEM or TEM. At the moment nearly exclusively gallium Liquid Metal Ion Sources (LMIS) are used for ion beam generation.

Therefore, the Liquid Metal Alloy Ion Sources (LMAIS) represent a promising new alternative research area to expand the global FIB application fields. Here, especially, IBL (Ion Beam Lithography) - a direct, resistless and three-dimensional patterning - enables a simultaneous in-situ process control by cross-sectioning and inspection. Thanks to this, nearly half of the elements of the periodic table are made available in the FIB technology as a result of continuous research in this area during the last forty years [1]. Key features of a LMAIS are long life-time, high brightness and stable ion current. Recent developments could make these sources as an alternative technology feasible for nano patterning challenges e.g. to tune electrical, optical, magnetic or mechanic properties.

In this contribution the operation principle, the preparation and testing technology as well as prospective domains for modern FIB applications will be presented. As an example we will introduce a $Ga_{35}Bi_{60}Li_{5}$ LMAIS in detail. It enables high resolution imaging with light Li ions, shown in Fig.1 obtained with a VELION FIB/SEM system (Raith GmbH), as well as heavy Bi ions or polyatomic clusters, all coming from one ion source [2].

Additionally, also new ion source developments based on gas field emission (GFIS), on ionic liquids (ILIS), on magneto-optical traps (MOTIS) or on ICP or ECR high current sources for Xe-FIB are presented. Combined with an optimized FIB optics design they can open a bright field of new employments. These alternative ion sources will be introduced and briefly described.

[1] L. Bischoff, P. Mazarov, L. Bruchhaus, and J. Gierak, *Liquid Metal Alloy Ion Sources - An Alternative for Focused Ion Beam Technology,* Appl. Phys. Rev. 3 (**2016**) 021101.

[2] W. Pilz, N. Klingner, L. Bischoff, P. Mazarov, and S. Bauerdick, *Lithium Ion Beams from Liquid Metal Alloy Ion Sources,* J. Vac. Sci. Technol. B 37 (**2019**) 021802-1.

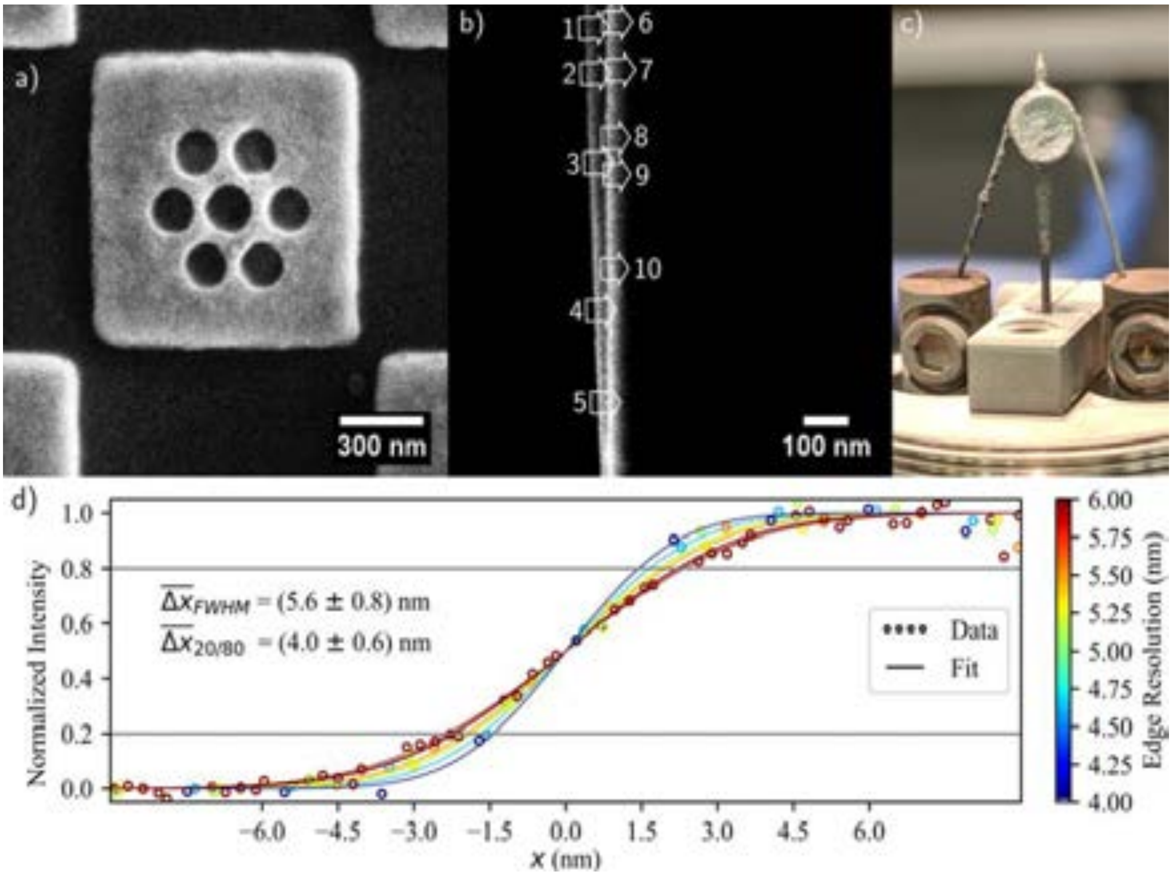

Fig. 1: a) FIB-SE image of 100 nm thick gold structures using a 35 keV ^{7}Li beam. 7 milled circles with a diameter of 125 nm. b) Sharp edge used for resolution measurements. c) Photography of the emitter module after 1000 µAh operation. d) 10 edge profiles have been extracted from the areas indicated by the arrows in image (b). Each of these edge profiles is averaged over 36 consecutive lines respectively 50 nm in width, normalized and fitted by an error function. For better visualization the edge profiles have been aligned to the fitted center of the error function. Data points as well as fit functions are colored according to their edge resolution (FWHM). The mean edge resolution evaluates to (4.0 ± 0.6) nm, determined from a 20% to 80% raise of the intensity [2].

New Applications in advanced FIB-SEM Nanofabrication with a FIB-centric Lithography System

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With properties such as direct, resistless and three-dimensional patterning, FIB based nanofabrication can be seen as complementary to electron beam lithography (EBL) providing simplification by reducing the number of processing steps necessary. However, while high resolution patterns can easily be achieved in resist by EBL, requirements for sophisticated FIB based nanofabrication are specifically demanding in terms of ion beam characteristics and its reduction of beam tails. In addition, for large area nanostructures stability of the beam current becomes increasingly important. There is always a trade-off between resolution (at low current for small beam size) and acceptable process time (usually at high current for large volume removal). With a FIB-centric setup where the ion beam is always perpendicular to the sample plane and the use of a laser interferometer stage at nm accuracy more sophisticated applications are possible. The tilted, additional SEM column allows for in-situ inspection and live process control.

We report on latest applications that display the full potential of fabricating complex high resolution, large area nanostructures exceeding the size of common write fields utilizing a FIB-centric nanofabrication system. A highly automated workflow, milling strategies adapted to the pattern geometry, long-term stability and stitch-free continuous writing modes open the door to a new world of applications. The choice of various ion species further expands the range of options enabling Ga-free milling and the selection of appropriate ion species.

SIMPLE – A FIB for Deterministic Single Ion Implantation

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[1]Surrey Ion Beam Centre, University of Surrey, Guildford, GU27XH, UK
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[3] The Photon Science Institute, University of Manchester, Oxford Road, Manchester M139PL, UK
[4]Advanced Technology Institute, University of Surrey, Guildford, GU2 7XH, UK

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Single isolated dopant atoms implanted into solid state devices have been shown to be a viable architecture for quantum technologies. Ion implantation provides many advantages as a manufacturing method for such devices, such as speed and scalability, but there is currently no way to completely control the number of implanted ions.

The SIMPLE (Single Ion Multispecies Positioning at Low Energy) tool, is a new focused ion beam tool in operation designed for the manufacture of quantum technologies. The tool has a 25kV LMIG set up for femtoAmp sample currents, with ultra-fast beam blanking, neutral blocking and a highly efficient secondary electron detection system. Deterministic ion implantation is achieved through extraction of single ions through fast beam blanking with low currents, ion implant detection through collection of secondary electron (SE) signal from the target, and high spatial precision in ion placement. Currently the tool has achieved an 80% probability of implanting a single Bi^+ ion into bulk silicon without error, with a 20nm beam spotsize determining dopant placement precision. A lot of work has gone into maximizing the detection efficiency for secondary electrons and investigating the factors which affect the SE yield.

Currently the system is running with Bi source, and there are In sources available. Alongside the development of the instrument there is also research into developing a series of liquid-metal ion sources for elements with optical and quantum applications including P, Te, Se and Cd.

A second SIMPLE tool has also been installed at the Ion Beam Centre, which operates with a 20kV duoplasmatron arc source, capable of 50nm spotsizes. SIMPLE #2 will initially operate with nitrogen source for the fabrication of NV centers in diamond.

3D EBSD and EDS Developments

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The integration of energy dispersive spectrometry (EDS) and electron backscattered diffraction (EBSD) on a scanning electron microscope (SEM) are routine methods of material analysis. EDS offers chemical quantification and element spatial distribution in the form of quantitative analysis and X-ray mapping. EBSD enables microstructural characterization studying phase relationships, local misorientations and grain properties such as size, morphology and boundary characteristics. The integration of these two techniques on a single platform with simultaneous acquisition enables full material characterization and data correlation within a single user interface.

Development in automating these analysis techniques with focused ion beam (FIB) technology has extended this powerful material characterization into the third dimension. This is invaluable in applications where a true 3D understanding of the sample is required [1]. This analysis follows an iterative process, whereby the ion beam removes a slice of material before an electron image, EDS and EBSD map are acquired. By repeating this flow, a stack of data can be processed to reconstruct a representation of the volume of interest in 3D.

The benefits from the development of next generation CMOS based EBSD detectors and large area EDS detectors, which can collect high quality data at high acquisition speeds can also be applied to the collection of 3D data. The result is that data collection in 3D is now becoming far more achievable on a greater range of samples, examples are shown on Fig. 1 and Fig. 2. [2]

[1] S. Zaefferer and S. Wright in "EBSD in Material Science", ed. A. Schwartz, M. Kumar, B Adams, D. Field (Springer) p.109.

[2] Parts of the presented results were achieved in collaboration with the University of Plymouth. This work has been supported by the European Regional Development Fund.

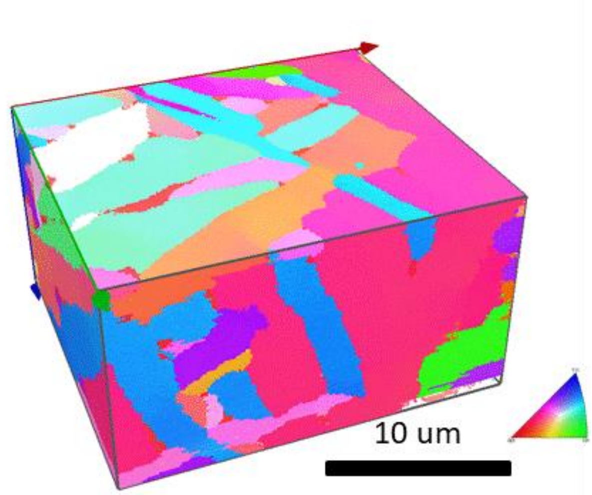

Fig. 1: 3D EBSD analysis of a Ti64 alloy, data shown is an orientation map (IPF Y) and is used to study retained beta grains and grain boundaries. Acknowledgement: Natasha Stephen & Plymouth Electron Microscopy Centre, University of Plymouth.

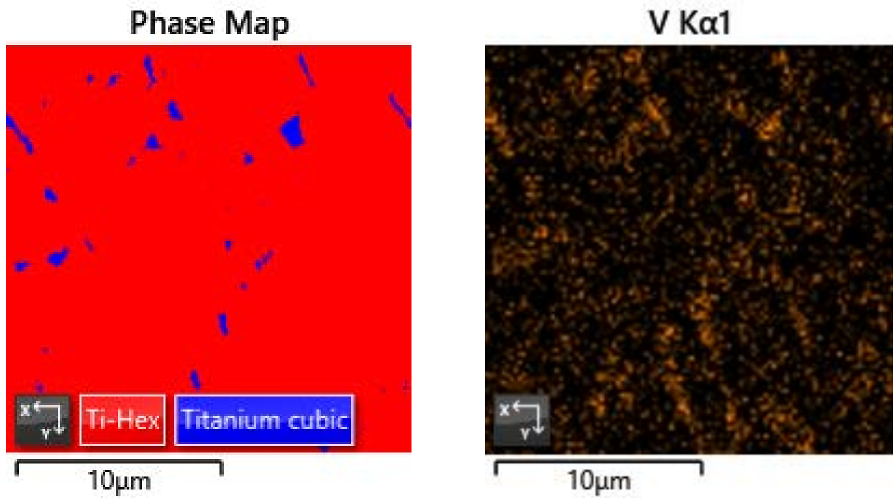

Fig. 2: 2D EBSD phase map and Vanadium element map extracted from the 3D volume shown in Fig. 1. Acquiring EDS and EBSD simultaneously allows correlation between the data, in this case it shows the Vanadium is concentrated into the beta-Ti phase.

Combined laser and FIB preparation for TEM planar analysis of flash memory cells

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Integrated circuits (IC) used in automotive applications have been produced with zero ppm failure rates. To achieve this quality goal single electrically failed ICs from qualification or field application has to be traced down to its physical failure root cause to derive further improvements of IC processing and reliability testing. Electrical defects can be localized by emission microscopy or laser beam based techniques and then have to be analyzed by scanning or transmission electron microscopy (TEM). Often specific step by step preparation strategies has to be applied to identify and analyze the related structural defects.

In this work we demonstrate a complex preparation workflow based on precise laser milling followed by plasma-FIB trimming, including large area planar TEM preparation of the active Si substrate and final lamella cross sectioning at the defect site to visualize dislocation bundles as root cause for local electrical shorts. In a first step the IC sample has been delayered until contact level. The electrical defect within the defective flash memory cell was verified by SEM in-situ nanoprobing investigation. Then MicroPrep™ laser processing by XL-Chunk™ technique, fig. 1, was applied to cut out the defective IC structure. The chunk was separated, tilted by 90° and glued to a Cu half ring suitable for a TEM sample holder, fig. 2 left. Further laser milling was processed to remove the Si substrate underneath the defect site. As next step large area plasma-FIB milling and final Ga-FIB polishing down to electron transparency was performed to prepare an approx. 30µmx20µm and about 1.5µm thick TEM lamella of the active Si substrate, fig. 2 right. Afterwards the planar lamella was investigated by STEM. At both localized defect areas a cluster of three dislocations were detected, fig.3 left. Both defects show almost identical length, shape and position relative to the IC layout. Additional a single dislocation was observed at a 3rd defect position, which is of same length as the other dislocations.

To clarify the vertical positon of these dislocations related to the doped source and drain areas further cross section TEM-lamellae were prepared at both affected transistors, fig. 3 right. The final TEM investigation showed that the dislocations start and end at the Si surface and slope of the field oxide. By combining the planar and vertical TEM cross section analysis the 3D shape of the dislocations causing the leakage within the flash memory cell could be verified.

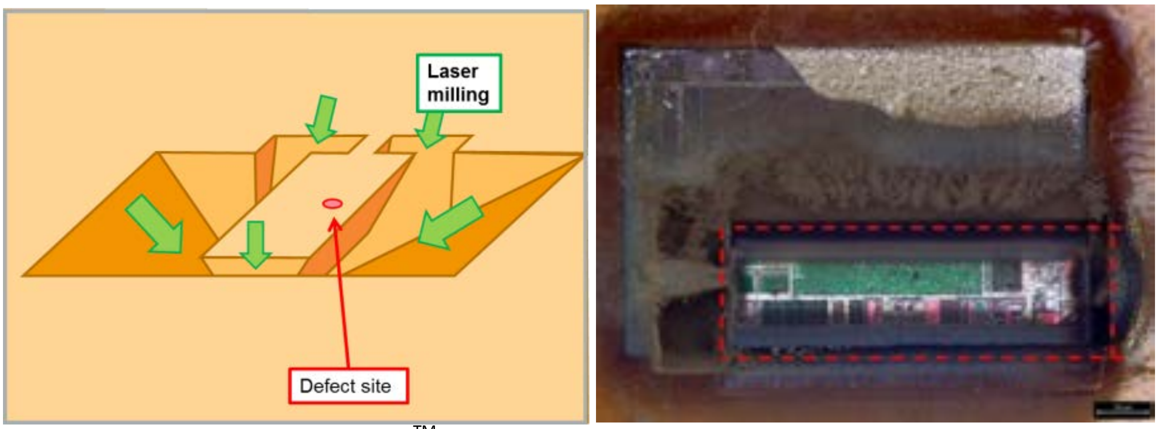

Fig. 1: Laser processing by XL-Chunk™ technique to extract the IC structure from the die

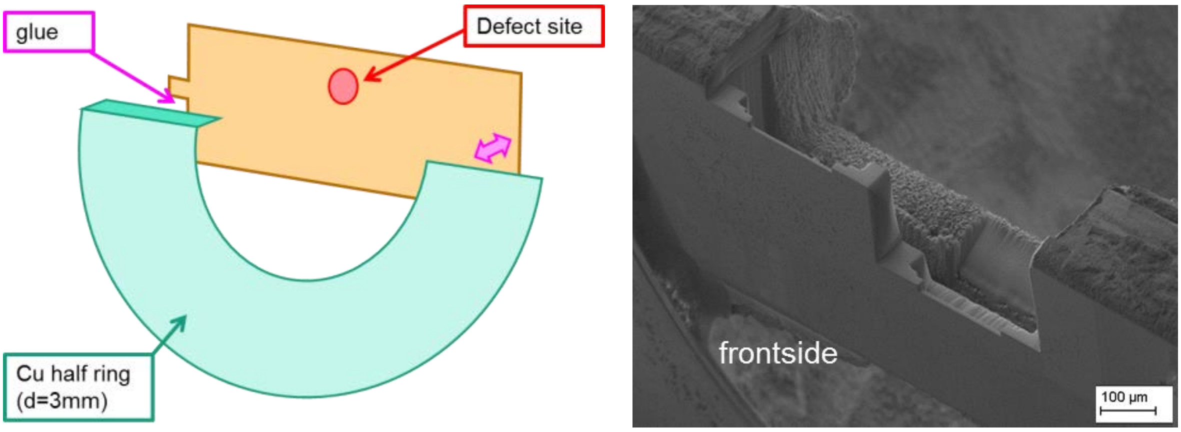

Fig.2: Adaption of the IC Chunk to a half Cu ring (left) and further laser, Plasma-FIB and Ga FIB thinning (right)

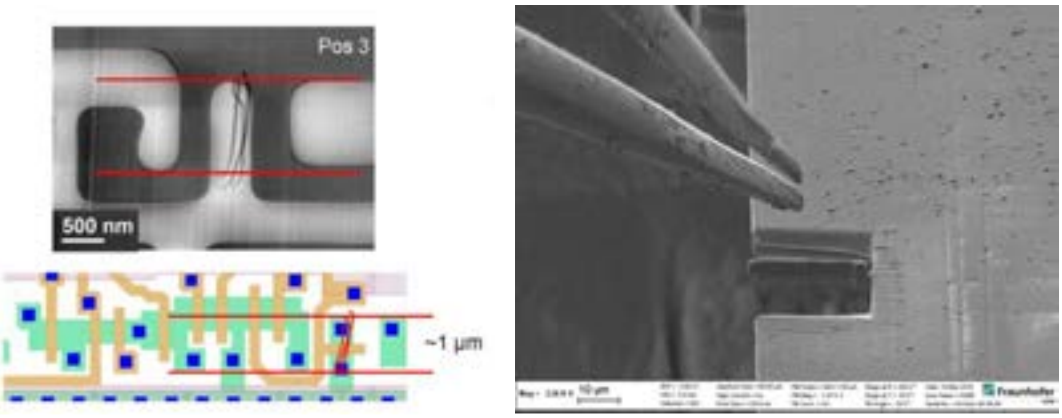

Fig.3: Dislocation cluster between source and drain (left) and further TEM prep for vertical TEM analysis (right)

How FIB induced artefacts influence *in situ* characterization in the TEM

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Recent developments of *in situ* TEM holders have allowed to access unique and groundbreaking results by facilitating the implementation of TEM experiment in gas, in liquid, at high temperature and under electrical biasing. This was made possible by replacing conventional TEM sample support by MEMS Environmental chips (E-chips) which have the power to apply precise and controlled stimuli on a sample during TEM observation. However, behind every successful TEM, and *in situ* TEM experiment, there is a high-quality sample preparation necessary.

FIB induced artefacts have been extensively studied, and techniques have been developed to minimize them and produce high quality samples. From these known phenomenon, new challenges emerge during *in situ* applications. Understanding how these known artefacts can impact the sample quality, or the application of a stimuli is paramount for successful *in situ* TEM experiments [1]. To illustrate this, a TEM foil composed of a ferroelectric material is prepared using the FIB and electrically connected to the Fusion™ holder. The sample was successfully connected without short circuit and ferroelectric domain walls could be moved and tracked at atomic resolution during *in situ* electrical characterization as shown in Fig.1.

Then, we will discuss how problematics caused by FIB induced artefact can apply differently depending on the targeted *in situ* application. In this example, a GaP//Ga(N,As,P)//GaP stack is prepared on a MEMS cell for the Atmosphere™ system using the FIB, and observed high temperature in a gas environment [2]. High resolution image can be obtained with this method even at high temperature and at a 1,000 Pa pressure as shown in Fig.2.

Acknowledgments:

We would like to thank Goran Drazic from the Slovenian National Institute of Chemistry and Gregor Kapun from the Jožef Stefan Institute in Ljubljana, Slovenia, for sharing the successful experiment on the ferroelectric ceramic sample. We also thank the ERC Holoview from David Cooper for funding the work done on FIB induced artefact for *in situ* biasing, which was performed at CEA-Leti [1].

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[2] Rainer Straubinger, Microsc. Microanal. 22, 515–519, **2016**

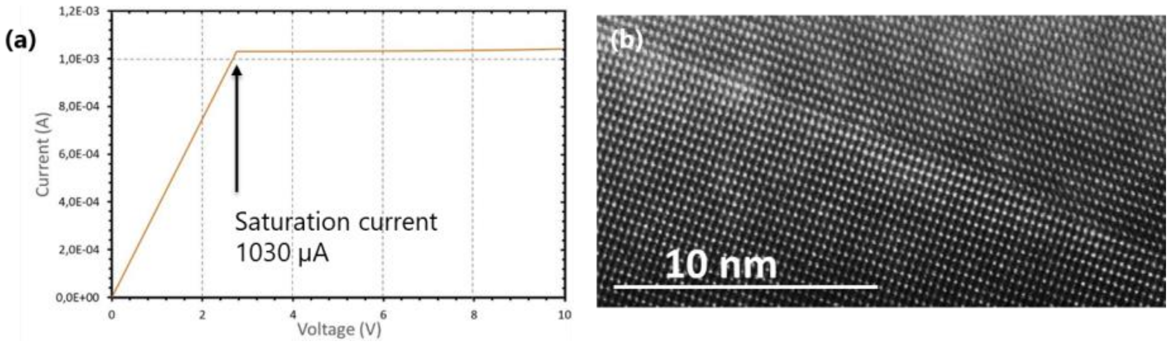

Figure 1. (a) I(V) profile obtained during electrical operation of the FIB prepared ferroelectric ceramic using the Fusion system (b) STEM bright field image showing a single domain wall at atomic resolution, appearing brighter than the rest of the lattice.

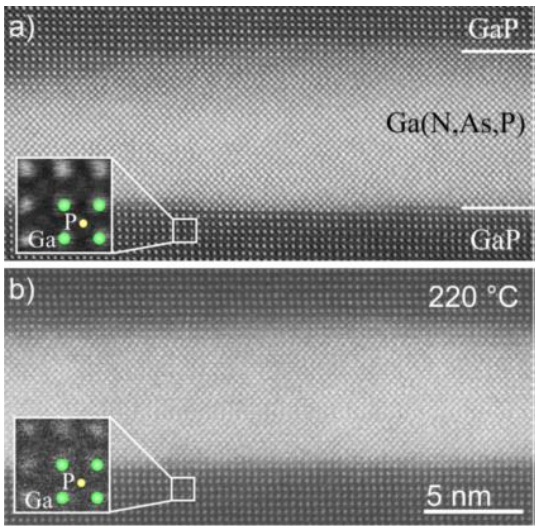

Figure 2. (a) STEM image of a FIB lamella before depositing the lamella in the environmental cell. An exemplary unit cell is shown enlarged as an inset. (b) STEM image of the same lamella in the cell at 220°C and 1,000 Pa of N_2 environment [2].

Development of a new integrated instrument for accurate and reproducible physico-chemical characterisation of nanoparticles

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Nowadays many consumer products contain nanoparticles in order for them to have certain desired properties. However, with the addition of nanoparticles these products can have potentially unknown health risks to humans, animal and plant species, and to the environment in general. The current approach for nanomaterial risk identification involves their physico-chemical characterisation employing a variety of techniques and separate instruments. This makes the characterisation an expensive and time-consuming process.

In the framework of the H2020 project npSCOPE, we are developing a new integrated instrument for the characterisation of nanoparticles. The aim is to improve the efficiency of the nanomaterial characterisation workflow by integrating several techniques in one single instrument. The npSCOPE instrument couples the ultra-high resolution of the Gas Field Ionisation Source (GFIS) technology [1] with detectors for secondary electron imaging, a secondary ion mass spectrometer (SIMS) for chemical analysis [2] and a transmission ion detector for 3D visualisation. The instrument will allow the characterisation of nanoparticles in their raw form and embedded in complex matrices (e.g. biological tissue, liquid, composite, etc.). A further key feature of the instrument are cryo-capabilities in order to perform analyses of the interactions between nanoparticles and biological systems close to the native state.

Currently the instrument is in the assembly/initial testing phase and the corresponding work progress will be reported here. Furthermore, some preliminary results on HIM-SIMS investigations of biological samples containing nanoparticles will be presented.

(For further information please visit www.npscope.eu)

This project has received funding from the European Union’s Horizon 2020 Research and Innovation Programme under grant agreement No. 720964.

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TOF-SIMS with highest lateral resolution by pulsing the Ne-GFIS in a HIM

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The helium ion microscope (HIM), well known for its high-resolution imaging and nanofabrication performance, suffered from the lack of a well integrated analytic method that can enrich the highly detailed morphological images with materials contrast. Recently, a magnetic sector and a time-of-flight secondary ion mass spectrometer (TOF-SIMS) have been developed that can be retrofitted to existing microscopes [1,2].

We report on our time-of-flight setup using a straight secondary ion extraction optics that has been designed and optimized for highest transmission. The high efficiency is the most crucial parameter to collect enough signal from nanoparticles prior to their complete removal by ion sputtering. As a major advantage the time-of-flight approach inherently can measure all masses in parallel and thus provides the complete picture of the sample composition. The TOF-SIMS is a versatile add-on that helps the user to get previously unknown details about his samples and is therefore beneficial for many applications. At the end we will also give an outlook on future developments.

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Combination of the FIB-TOF-SIMS technique with GIS – increasing the ionization probability and sputtering rates of thin films

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Incorporation of a Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) detector to commercial dual beam systems combining Focused Ion Beam (FIB) with Scanning Electron Microscope (SEM) has become popular in recent years. This solution allows a 3D elemental structure of a sample to be represented, the topology of the sample surface to be imaged insitu before and after a TOF-SIMS measurement and the sputtering process to be monitored online using FIB secondary electrons. In this work we went one step further by combing FIB-SEM with TOF-SIMS and Gas Injection System (GIS). By simultaneously co-injecting supplementary gases (water vapor or fluorine gas) in the proximity of a sample surface during a Ga^+ primary beam bombardment we explored the GIS potential for enhancing element ionization probabilities and modifying the sputtering rates. The experiments were conducted on four 100 nm thick metallic thin films (Cu, Zr, Ag and W) fabricated with the Physical Vapor Deposition (PVD) process. The highest useful yield enhancements were obtained in the case of Cu – by a factor of 10 due to the presence of water vapor and by a factor of 510 when exposing the sample to the fluorine gas. Moreover, in most cases fluorine has increased sputtering rates twice.

Ultrastructural changes accompanying the intracellular mineral formation in alga E. huxleyi': a cryo-FIB/SEM study

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Focused ion beam milling and serial block face imaging in cryogenic conditions (cryo-FIB/SEM) is a novel technique with great potential for revealing the structural organization of cells in 3D at nanometric resolution. Such information enable precise quantification of cells organelles' morphological features and spatial relationships.

Here, we use cryo-FIB/SEM to characterize one of the most intriguing biomineralization systems, i.e. single-celled algae known as coccolithophores. These algae decorate their cell surface with scales made of calcite (coccoliths) that are produced intracellularly in a specialized compartment called coccolith vesicle.

We investigated the ultrastructural framework underlying mineral formation in the most abundant coccolithophore Emiliania huxleyi by visualizing cells in different stages of coccolith formation. Using cryo-FIB/SEM we describe the evolution of the cells ultrastructure as the mineralization proceeds.

Our results show that coccoliths at early formation stage exhibit a labyrinth-like structure termed reticular body adjacent to the coccolith vesicle. The reticular body was not observed for more advanced stages of coccolith formation. Instead, in cells with almost mature coccoliths, large vesicles with bright contrast, appearing to originate from the Golgi apparatus, were observed close to the surface of the growing coccolith. Furthermore, 3D visualization revealed an extended membrane system surrounding the late-stage coccolith. Finally, we find that all the cells, at any stage of coccolith formation, exhibit a second, membrane bound, pool of calcium, which is also rich in phosphorous1. This compartment has been hypothesized to act as a calcium reservoir for coccolith calcite formation2.

For the first time, using cryo-FIB/SEM, we could describe the 3D ultrastructural changes proceeding with coccolith maturation in E. huxleyi and discover new players in the mineralization pathway.

1 Nature Communications, 7, 11228, (2016)

2 Advanced Science, 4(10), 1700088, (2017)

Avoiding amorphization during ion beam irradiation and critical dimension reduction of nanostructures

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Ion beam induced collateral damage is becoming an issue in FIB processing, as it limits the application of ion beams for nanostructure fabrication. This is of special importance for the application of focused ion beams for nanostructure fabrication.
Here, we present an approach to mitigate the ion beam induced damage inflicted on semiconductor nanostructures during ion beam irradiation. Nanopillars (with a diameter of 35 nm and a height of 70 nm) have been irradiated with both, a 50 keV Si^+ broad beam and a 25 keV focused Ne^+ beam from a helium ion microscope (HIM). Upon irradiation of the nanopillars at room temperature with a medium fluence ($2x10^{16}$ ions/cm²), strong plastic deformation has been observed which hinders further device integration. The shape and crystallinity has been studied by HIM and TEM. This differs from predictions made by Monte-Carlo based simulations using the TRI3DYN. However, irradiation at elevated temperatures with the same fluence not only preserves the shape of the nanopillars but allows for controlled diameter reduction by as much as 50 % without significant change in pillar height.
It is well known that above a critical temperature amorphization of silicon is prevented independent of the applied fluence. At high enough temperatures and for not too high flux this prevents the ion beam hammering and viscous flow of the nanostructures. These two effects are responsible for the shape change observed at low temperature. We find that irradiation above 650 K preserves the crystalline nature of the pillars and prevents viscous flow. In addition, a steady thinning process of the nanopillars to a diameter of 10 nm without a significant change in height is observed for higher fluencies at elevated temperatures. As the original pillar diameter is smaller than the size of the collision cascade, enhanced forward sputtering through the sidewalls of the pillar is responsible for this pillar-thinning effect. Results for various ion beam energies, fluencies, fluxes and temperatures will be presented and compared to TRI3DYN simulations. Such a reliable and CMOS-compatible process could serve as a potential down scaling technique for large-scale fabrication of nanostructure based electronics and many other FIB based milling applications.

Ultra-fast growth of W-C metal nanostructures by Focused Ion Beam Induced Deposition under cryogenic conditions (cryo-FIBID)

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We report on an ultra-fast method to locally grow metal layers, nanowires and contacts. It relies on a Focused Ion Beam (FIB) and a suitable condensed layer formed on the substrate under cryogenic conditions. The technique implies cooling the substrate below the condensation temperature of the precursor material, subsequent ion irradiation with the shape of the desired pattern, and posterior substrate heating above the condensation temperature. Here, using the $W(CO)_6$ precursor material, a Ga^+ FIB, and a -100°C substrate temperature, W-C metal layers and nanowires with a resolution down to 38 nm have been grown using cryogenic focused-ion-beam-induced deposition (cryo-FIBID). The most important advantages of cryo-FIBID have been found to be the fast growth rate (up to three orders of magnitude higher than conventional FIBID with the precursor material in gas phase) and the low ion irradiation dose required (< 100 $\mu C/cm^2$), which gives rise to very low Ga concentrations in the grown material and the substrate, below the detection limit of energy-dispersive x-ray spectroscopy. Thus, the typical problems of the FIB technique are avoided given the minimal influence of ion implantation, ion milling, ion-induced creation of defects and amorphization. Electrical measurements indicate that W-C layers and nanowires grown by cryo-FIBID exhibit metal resistivities with values around 400 $\mu\Omega$cm, which, in combination with the ultra-fast growth and minimal ion irradiation, paves the way for their use in circuit editing and photomask repair in semiconductor industry and, more generally, for the local growth of metal layers and nanowires and establishment of metal contacts in various applications relevant in nanotechnology [1, 2]. Figure 1 illustrates the cryo-FIBID process.

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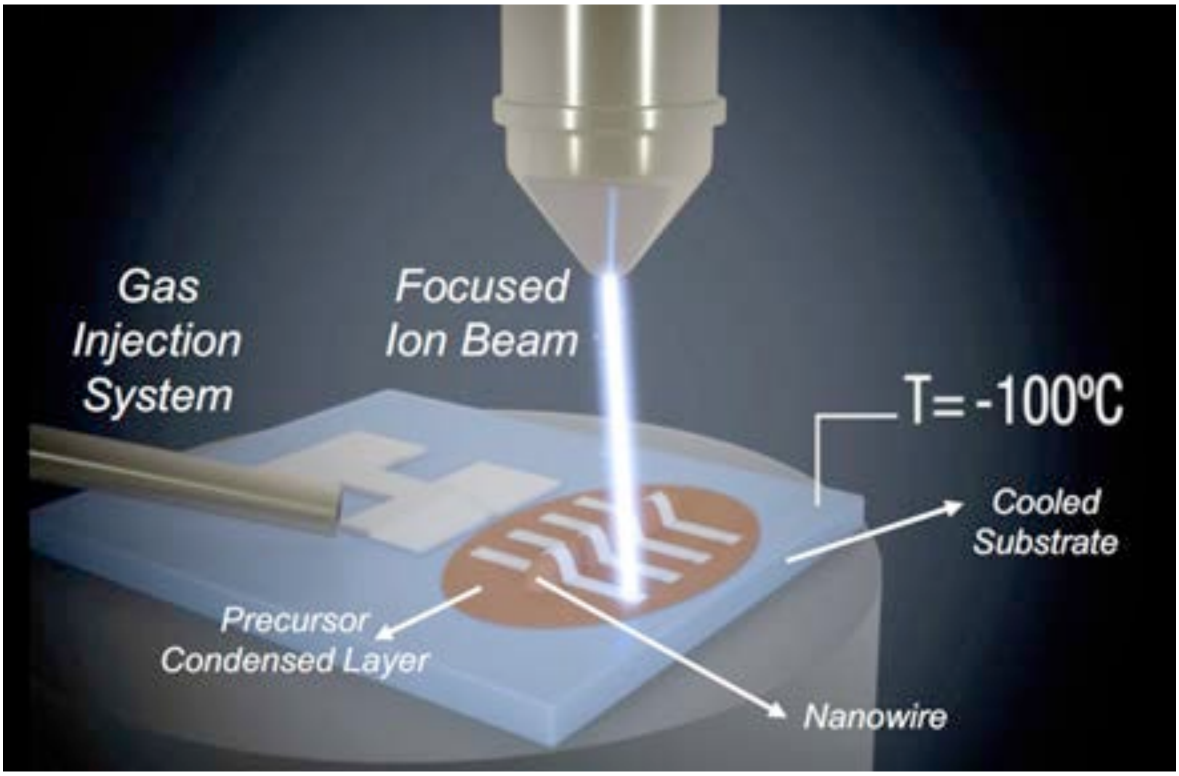

Fig. 1: Representation of the cryo-FIBID technique, applied in this case to the growth of electrical contacts onto a nanowire. The cryo-FIBID technique implies the use of a condensed precursor layer, FIB irradiation and subsequent heating to remove the unexposed precursor areas.

Using FIB as a broad ion source for nanofabrication on AIII-BV(InSb) semiconductors

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Nowadays there is big interest in production of low-dimensional high aspect ratio nanostructures on AIII-BV semiconductor surfaces for various electronic and photonic applications. One way to achieve this goal is to use ion beam induced self-assembling on AIII-BV semiconductor surfaces which is not sophisticated and cheap bottom-up process. This, however, requires usage of broad ion beams (beam flux of ~10^{13} -10^{14} ions/cm^2 s.) which is required to achieve the linear sputtering regime to be able to control the nanostructures formation process involving non stoichiometric sputtering and surface diffusion of adatoms.
We show that one can successfully use normal incidence gallium Focused Ion Beam (FIB) in Dual Beam SEM/FIB Quanta 3D FEG (FEI) microscope for the controlled nanostructures formation in form of standing high aspect ratio nanopillars [1]. The FIB irradiation conditions were selected to mimic behavior of broad ion beam (spot ~100-200nm and overlap ~98%) which gives average beam flux 5.4×10^{14} ions/cm^2 s. In the experiments we irradiated with FIB gallium ions with energy in the range 3-30keV InSb single crystals to the final ion fluence of 2.8×10^{17} ions/cm^2. The usage of FIB is also beneficial since simultaneously to the FIB ion bombardment the sample surface could be imaged by SEM, which gives direct access to the nanostructures time (ion fluence) evolution dynamics which we studied [1]. The FIB/SEM experiments allow us also to tract not only collective system behavior (averaged over hundreds of nanostructures) but also single nanostructure growth dynamics could be observed [2]. The performed FIB experiments lead us to the conclusion that in addition to the sputtering and surface diffusion of adatoms also redeposition of sputtered material plays a key role. The formed nanostructures were examined by TEM together with EDX and nano beam diffraction to reveal chemical composition and atomic structure [1,2].

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Creating mesoscale ballistic transport devices from ultra-pure quantum materials

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Experiments on quantum materials are often limited by the size and form of the available crystallites, especially in newly discovered compounds for which the synthesis route has not yet been optimized to yield the largest growth results. This issue can be addressed by utilizing focused ion beam technology, which is ideally suited to take small crystals and tailor their shape into the specific requirements of a given experiment. Electrical transport experiments, for instance, require current paths running along specific directions of a crystals and well-defined voltage contacts in order to accurately determine the conductivity of a device. An example where these demands were met to create a functional, micromachined device from an oven-grown crystal is displayed in Fig. 1.

A key benefit of microstructured transport devices is their mesoscopic length scale, which provides the opportunity to study transport phenomena in which the shape and size of the sample itself influences the electronic properties of the material. This was recently demonstrated [1] in the metallic oxide $PdCoO_2$, a compound which can be synthesized at remarkably high purity. As a result, the low-temperature electronic mean-free-path exceeds 20µm. Using the FIB to reduce the sample size to below this length scale enables us to study the highly unusual ballistic properties in $PdCoO_2$.

We have found, that the ballistic electrons in this material do not propagate isotropically, but are directed along 3 principle orientations, governed but the underlying electronic structure. We demonstrate this experimentally by investigating the flow of electrons through narrow channels, c.f. Fig. 1 as well as by studying the cyclotron motion of the electrons in a magnetic field, by adapting the technique of transverse electron focusing for microstructured, three-dimensional samples. This FIB-based approach showcases the generic route towards the investigation of ballistic behavior in quantum materials in which lithography based methods are inhibited due to the crystal size or chemical composition.

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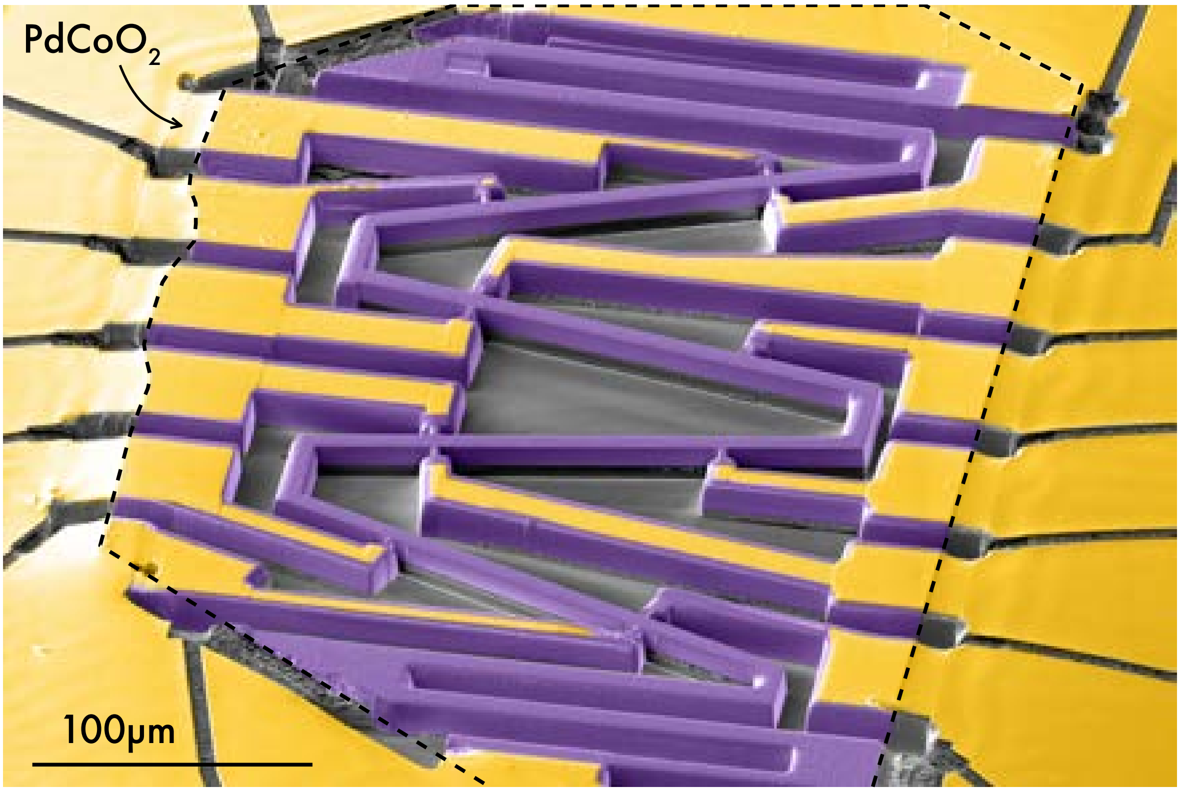

Fig. 1: A crystalline microdevice fabricated from the ultra-pure metal $PdCoO_2$. The 400 micrometer wide, oven-grown crystal has been patterned into a multi-contact crystalline circuit with the aim of investigating the in-plane anisotropy of the conductivity.

Out-of-plane Transport in ZrSiS, ZrSiSe, and HfSiS Microstructures

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A recent class of topological nodal-line semimetals with the general formula XSiY (X = Zr, Hf and Y = S, Se, Te) have attracted much experimental and theoretical interest due to their properties, particularly their large magnetoresistances and high carrier mobilities [1]. The platelet-like nature of the XSiY crystals and their extremely low residual resistivities result in measurement of the resistivity along the [001] direction which are extremely challenging. To accomplish such measurements, microstructures of single crystals were prepared using Focused Ion Beam techniques (Fig. 1). Microstructures prepared in this manner have very well-defined geometries and maintain their high crystal quality, verified by the quantum oscillations we observed. We will present magnetoresistance and quantum oscillation data for currents applied along both [001] and [100] in ZrSiS, ZrSiSe, and HfSiS and discuss the role microstructuring can play in the study of these materials. Additionally, we will discuss our ability to make these microstructures free-standing and the implications for future experiments.

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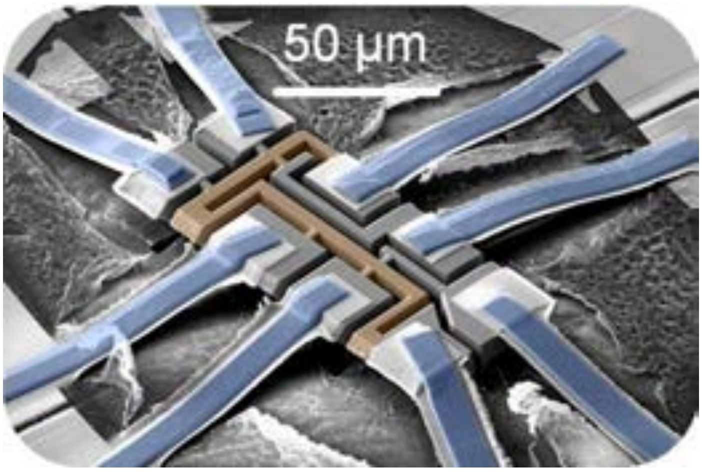
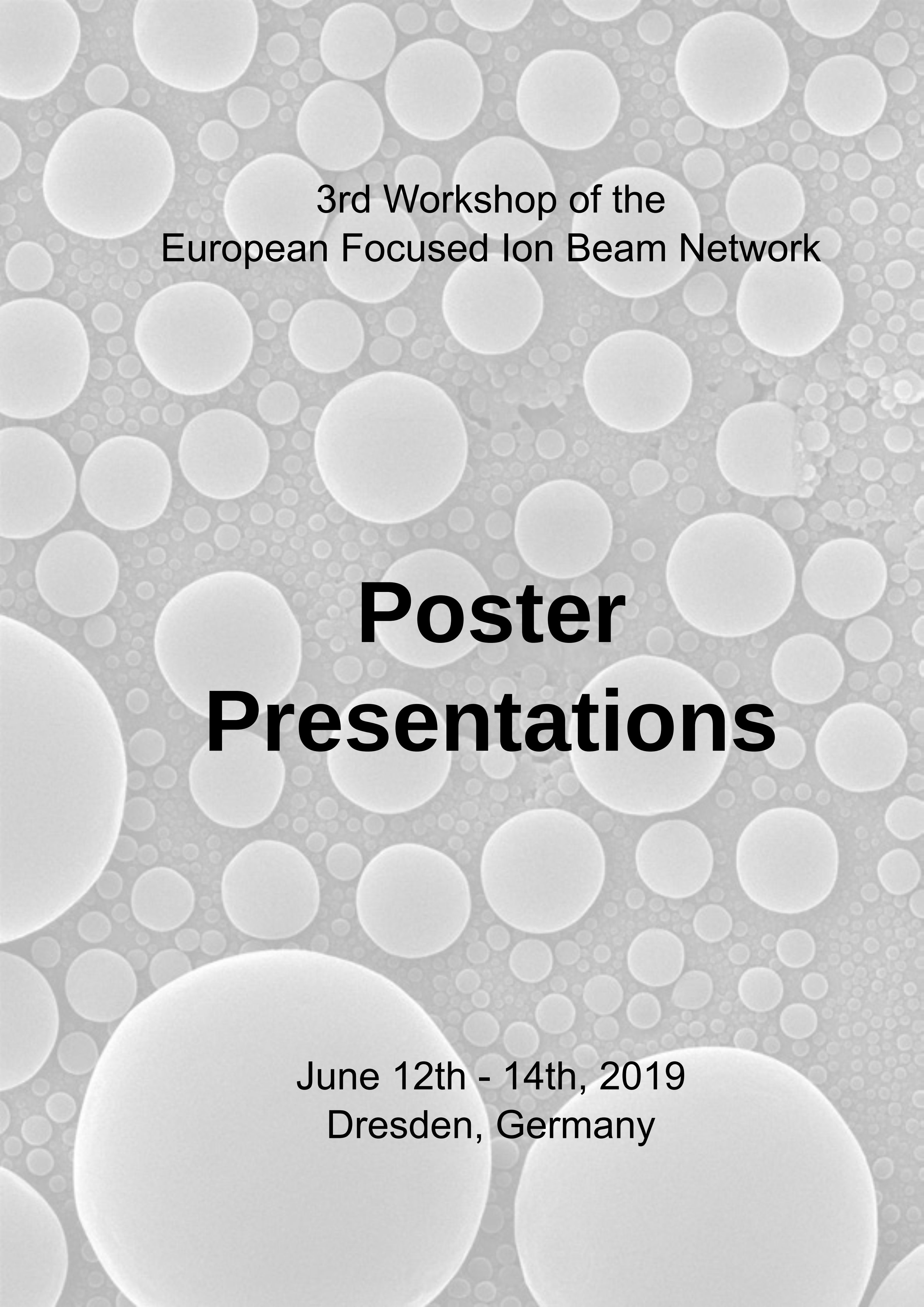


Fig. 1: A free-standing microstructure of HfSiS. The device is designed so that two four-point resistance bars, in series, can be measured simultaneously. One bar is along the [100] direction while the other is along [001].



3rd Workshop of the
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Poster Presentations

June 12th - 14th, 2019
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Microstructure fabrication for the precise study of electric anisotropy

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Electric anisotropy is an important aspect in the search of novel materials. In the case of topological semimetals, the study of electric anisotropy can provide strong indication of anomalous transport properties, such as the chiral anomaly effect[1]. However, it is of great difficulty to study the electric anisotropy of a bulk material with high precision. Besides the unavoidable misalignment, it is also challenging to apply the electric current along arbitrary crystalline directions.

In order to precisely study the electric anisotropy, a microcircuit is patterned using focus ion beam technique. As displayed in Fig. 1, the current is applied throughout this semi-circle, while there are multiple channels for the voltage measurements. With this particular setup, not only the contact geometry and sample dimensions are well defined to reduce misalignment and current jetting effect, but also the current can be applied along any crystalline directions, which makes a comprehensive study of the electric anisotropy possible. Moreover, with the rotation of this microcircuit under the presence of external magnetic field, one can systematically study the angular dependence of magnetoresistance with current applied along any crystalline directions. These measurements can provide crucial information for not only the electric anisotropy, but also the underlying electronic symmetry of the material.

[1] N. P. Armitage, E. J. Mele, and A. Vishwanath; Weyl and Dirac semimetals in three-dimensional solids; Rev. Mod. Phys. 90 **(2018)**, 015001

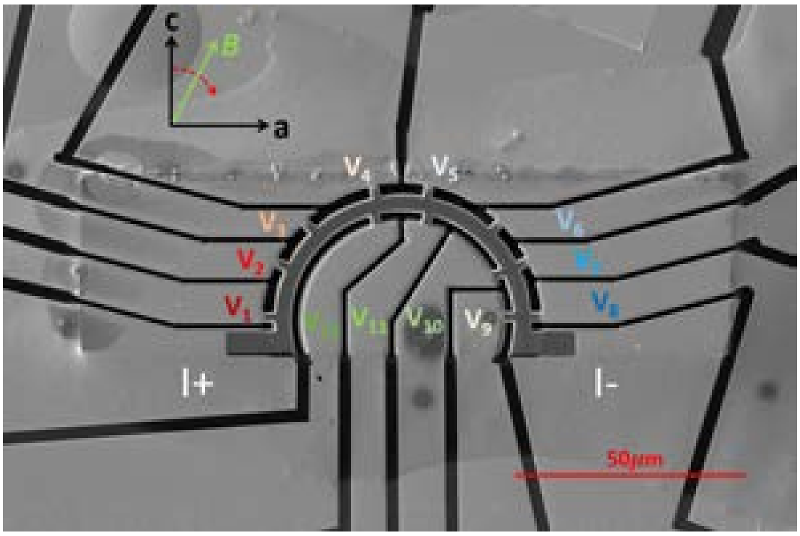

Fig. 1: SEM image of the semi-circle microstructure. The crystallographic directions are highlighted, as well as the current and voltage contacts. With this device, one can systematically study the electric anisotropy of the sample.

Micromachining of Cantilevers to Achieve Large Strain Gradients

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The application of strain is one of the ways in which we can explore the novel physics exhibited by new materials. For example, in materials like Cd_3As_2, strain could make the electrons behave as if they were under the influence of a magnetic field [1]. In many cases, the substantial strain gradients needed are virtually impossible to achieve on macroscopic samples, where dislocations or domains might lead to their breaking. With the Focused Ion Beam (FIB) we can overcome this difficulty by designing 3D microstructures specifically tailored to probe and withstand large strain gradients. In this work, we fabricate crystalline cantilevers to investigate the effect of elastic strain on the transport properties of the material Cd_3As_2.

We propose an experimental set-up where microstructures of Cd_3As_2, prepared from *single crystals* by FIB machining, are mechanically bent by moving a positioner with sub-micro-meter precision. The machined sample is shown in Fig. 1. It consists of a cantilever cut parallel to the *c* axis of the crystal. The long and thicker "paddle" section is pushed upwards by the positioner, consequently straining the thinner legs. The two outermost legs act as current and voltage leads, while the central branch is the part being measured. The thinning down of these legs is done not only to ease the bending but also to increase their resistance, thus guaranteeing that most of the measured signal comes from this part of the sample. The lamella is platinum welded to the edge of a sapphire chip, where we perform transport measurements as we increase the radius of curvature. As shown in Fig.2, with our set-up we can elastically bend the crystal to levels that would be impossible to achieve at macroscopic scales. We hope to carry further experiments and, thanks to the possibilities offered by the FIB, explore additional sample designs to obtain large strain gradients.

[1] Adolfo G. Grushin, Jörn W. F. Venderbos, Ashvin Vishwanath and Roni Ilan. Phys. Rev. X 6, 041046 (2016).

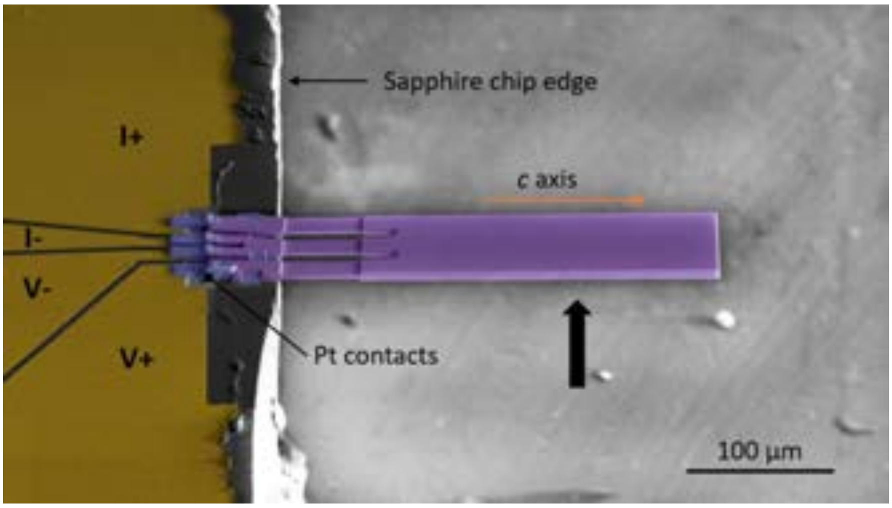

Fig. 1: Cd_3As_2 microstructure for probing strain effects by transport measurements.

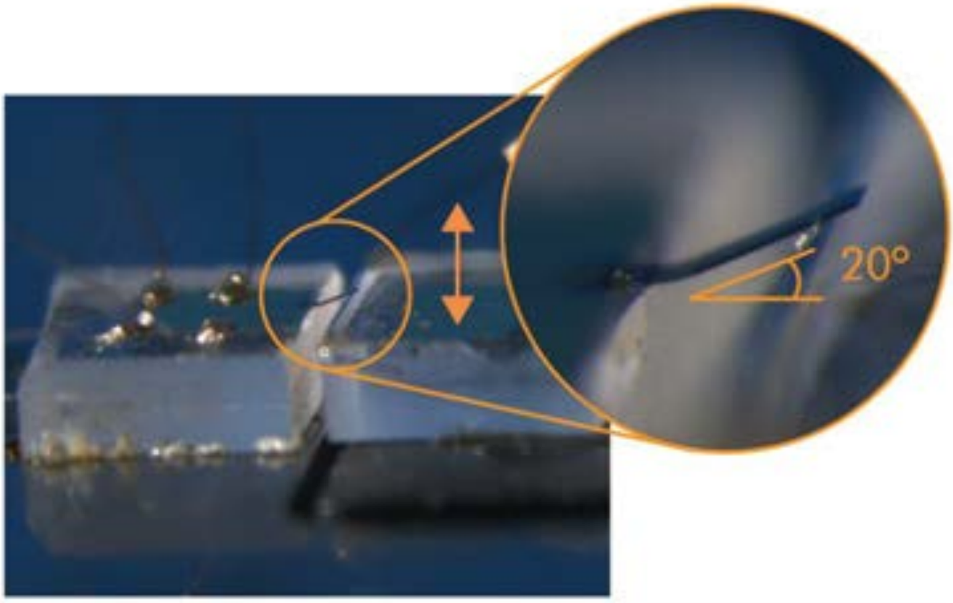

Fig. 2: experimental set-up. Combining the capabilities of FIB design with a mechanical positioner we can reach bending angles of 20° without plastically deforming the sample.

3D Micro Structuring of Quantum Materials

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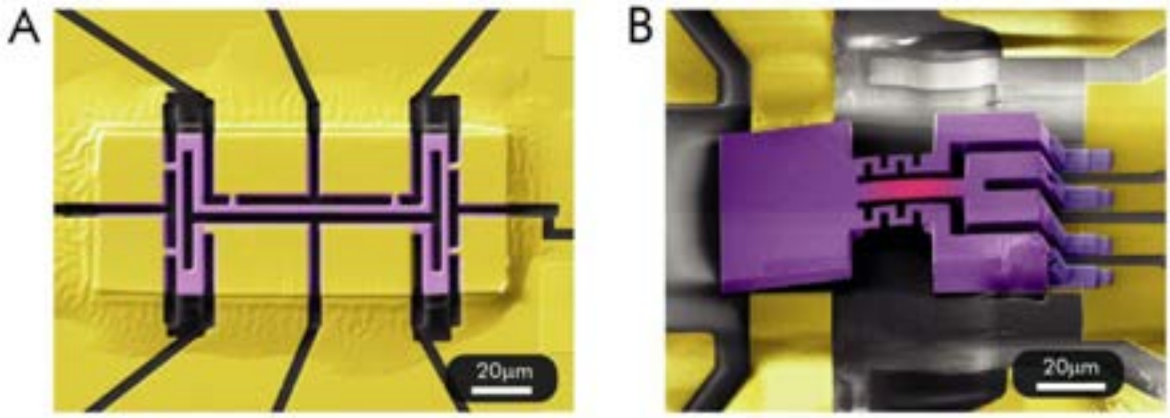
Using focused ion beam micro structuring has allowed us to investigate the electronic properties in a wide range of quantum materials in recent years. For this a macro lamella process is used to produce thin platelets from which we cut quasi two-dimensional micro structures (fig.1A). Here I will present recent advances in micro fabrication of quantum materials that enable us to extend our samples into more complex three-dimensional objects (fig.1B). This has led to devices where twist and bending strains are produced on the microscopic length scale with a strength inaccessible in macroscopic crystals. While strong bends and twists lead to the propagation of cracks in macroscopic single crystals and hence to a high failure rate we were able to obtain twist angles up to 6°/μm in micro structures, thereby enabling us to test quantum materials where chiral symmetries have been proposed.

The excitation via capacitive forces used in micro cantilever made from quantum materials might also hold possibilities beyond the application of strain and open new routes to investigate electronic correlations in dynamically tuned system.

Fig1: A) Quasi 2D micro structure of $PdCoO_2$. B) 3D capacitive micro cantilever made from Cd_3As_2.



3D Localization of Spinel and Sodium Contamination in Alumina by TOF-SIMS

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Phase and chemical compositions are crucial for properties of advanced ceramic materials. Study of the phase and chemical composition is nowadays limited to localized 2-dimensional methods and its sensitivity to local changes. Alumina as the most used ceramic materials is often doped by MgO to prevent abnormal grain growth to allow annihilation of pores pinned at grain boundaries. The phase equilibria of Al_2O_3-MgO has been widely studied and discussed. However, chemical composition in three dimensions of spinel ($MgAl_2O_4$) has never been described. 2D & 3D TOF-SIMS analysis of the spinel in an alumina matrix and its chemical composition will be presented.

We successfully measured the formation of the spinel in the alumina for different concentration of MgO using 3D TOF-SIMS imaging. The presented analytical method allowed characterization of advanced ceramic materials in volume and allowed the study of grain formation and contamination. Green body analysis by TOF-SIMS revealed high homogeneity of MgO distribution in the green body after ball milling and no MgO rich areas were observed. After sintering at 1700 °C for 30 min, majority of MgO was present in form of $MgAl_2O_4$ (spinel), which was confirmed by XRD analysis and by the Mg^+/Al^+ ratio in observed grains (Fig. 1). Majority of spinel grains have a volume below 1 μm^3, however, also the grains with a volume of tens of μm^3 are presented (Fig. 2). Sodium impurities presented in the starting powder were concentrated nearby or in the spinel grains.

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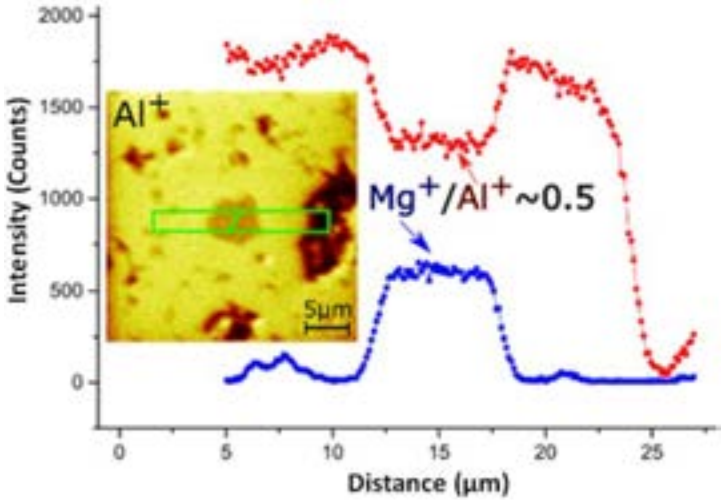

Fig. 1: TOF-SIMS image and lateral profile of Mg and Al through a spinel grain.

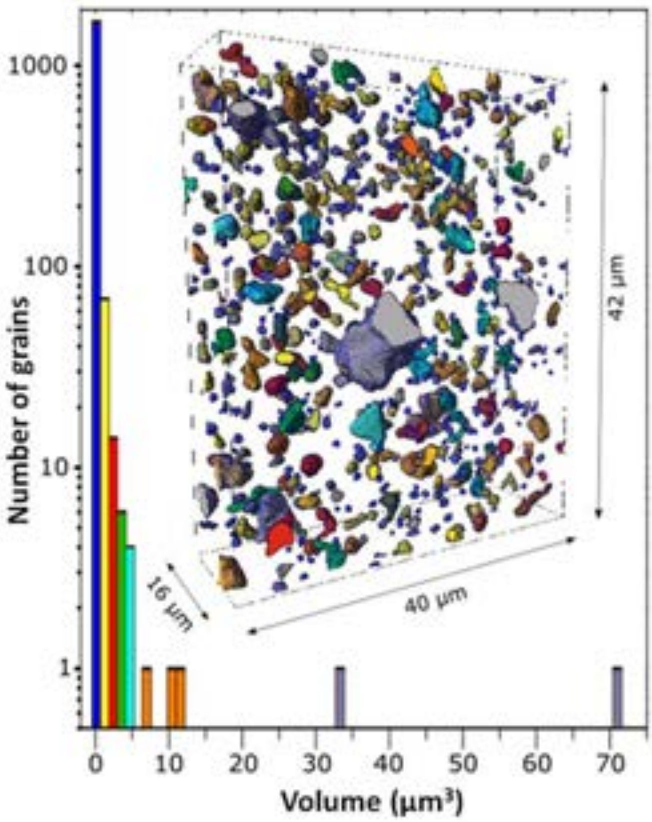

Fig. 2: Spinel grain size distribution in sintered alumina measured by TOF-SIMS. 3D render was colorized according to the volume of grains.

TEM Characterization of Periodic Arrays of ZnO Nanorods

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Zinc oxide is a wide band gap semiconductor, which has been recently deeply investigated for its beneficial properties such as piezoelectricity, absorption and emission in the UV region, optical transparency in the visible region or surface sensitivity. The applications include electronic devices, light emitters, chemical or biological sensors and other devices in the field of photovoltaics or medicine.

Highly ordered arrays of uniform nanorods can be fabricated with the help of electron or ion beam lithography (EBL or FIBL). The focused ion (electron) beam is used to define the positions of nucleation centers on the substrate (GaN or ZnO seed layer) covered by a resist layer. Chemical bath deposition is then used to grow the nanorods from the nucleation centers. Fundamental mechanisms of nucleation and growth of the ZnO nanorods are further studied by several techniques.

TEM characterization of ZnO nanorods prepared by FIBL or EBL can provide an insight into the growth process, particularly when the interface of the nanorod and the substrate is investigated. Preparation of a TEM specimen from as-grown standing nanorods is a challenging task. There are several issues related to the preparation of the TEM specimen: non-homogenous deposition of a protection layer, precision of selection of one row of nanorods, preferential sputtering, shadowing and curtaining during the polishing process, etc. We show how these obstacles in the fabrication process of the TEM lamella can be overcome.

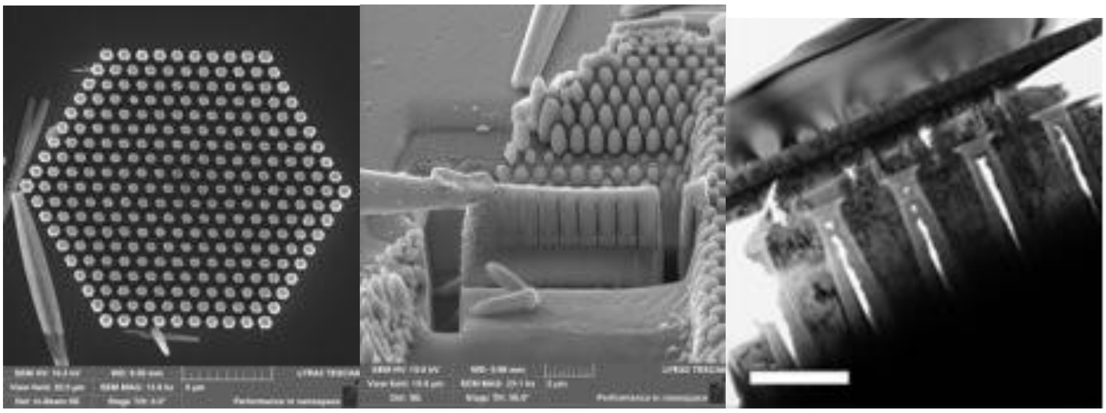

Fig. 1: A top view of a periodic array of ZnO nanorods prepared by FIBL (left), the lift-up of the TEM lamella from the standing row of nanorods (middle), the TEM overview of the final sample (right).

Investigation of the semi-conducting properties of oxides in the vicinity of the metal-oxide interface throughout lifetime of zirconium based fuel cladding

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Zirconium alloys are used as nuclear materials, especially as fuel cladding, for most Light water reactors as well as CANDU's. One of the "in-service" degradation phenomena for these materials is oxidation and hydrogen ingress into the base metal. The latter will cause hydride precipitation and thus embrittlement of fuel cladding reducing component lifetime. The ingress of hydrogen is thought to be strongly affected by the semi conducting properties of the oxide formed during component lifetime [1]. Therefore an interest to measure these properties directly exists in the research community.

FIB was used in a novel way during this study to create samples of real fuel cladding with varying thickness of oxide. The samples were cut in a wedge shape with platinum electrodes deposited at different thickness of oxide, a schematic diagram is shown in Fig 1. and SEM images in Fig. 2. Whilst inside the FIB/SEM micromanipulator arms could be used to contact different electrodes and using a circuit connected through the SEM/FIB and attached to a multimeter the resistance could then be directly measured. Examples from during the measurements can be seen in Fig 3. The final step of cutting under the electrodes with FIB allowed the accurate measurement of oxide thickness to be used in resistivity calculations. Some details of the method in ref [2].

Samples of Zircaloy-2 LK3 after 3 cycles and 9 cycles in the reactor have been compared. So far results suggest the semi-conducting region of oxide decreases drastically from 2000nm to 700-800nm between 3 and 9 cycles. And resistivity for both alloys plateaus, within the distance measured, at resistivity values in the magnitude of 1×10^4 Ohms.m. These results can be seen in graph 1. [3]

[1] A. Couet, A. Motta, R. Comstock, A. Ambard; "Oxide Electronic Conductivity and Hydrogen Pickup Fraction in Zr alloys" Corrosion science (2016)

[2] R. Vanta, S. Abolhassani, S. Kumar, M. Dadras, A. Baris, G. Boetsch, H. Brandenberger, A. Rummel; "Examination of semiconducting properties of oxides in the vicinity of metal-oxide interfaces for selected alloys"; The 16th European Microscopy Congress (2016)

[3] S. Abolhassani et al. "Towards an improved understanding of the mechanisms involved in the increased hydrogen uptake and corrosion at high burnups in zirconium based claddings" 19th ASTM International Symposium on Zirconium in the Nuclear Industry (2019)

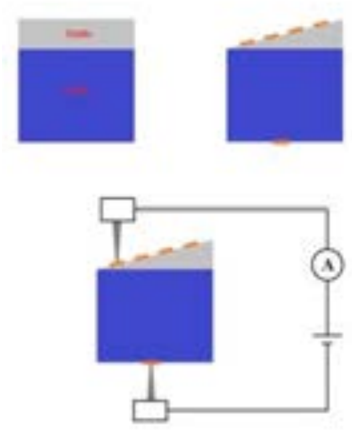


Fig. 1: Schematic representation of the set up for measuring conductivity of different oxide thicnkesses

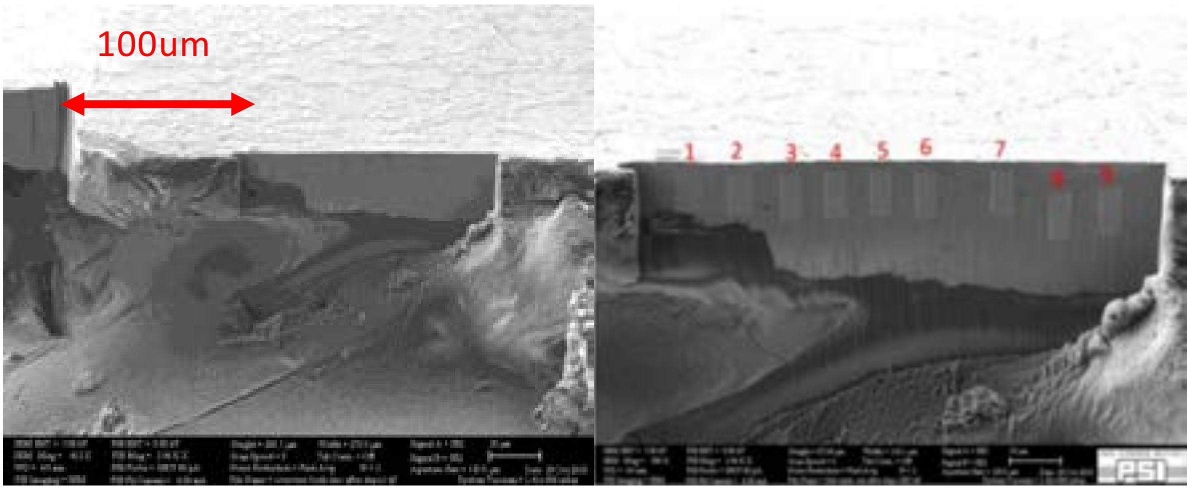


Fig. 2: SEM images showing the wedge cut and position of Pt electrodes.

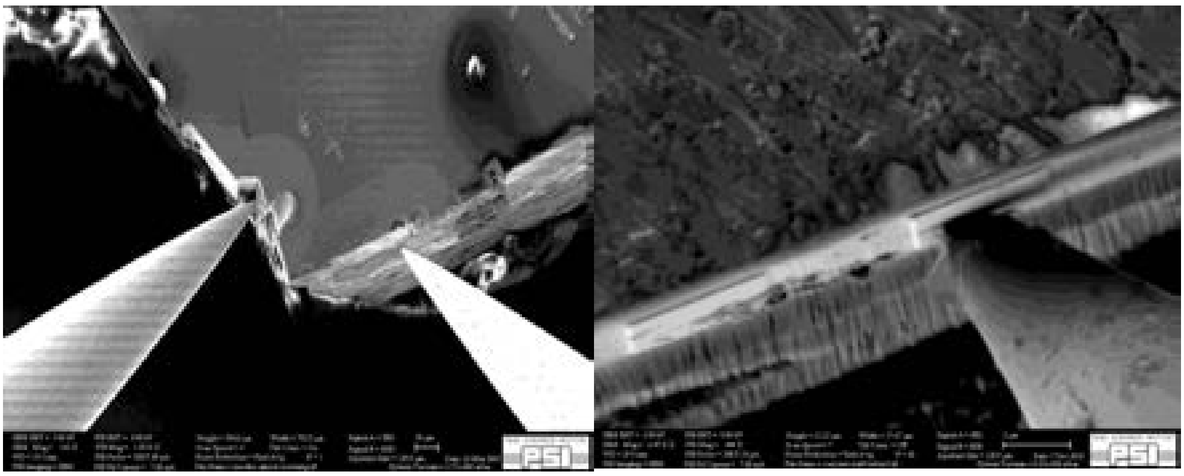
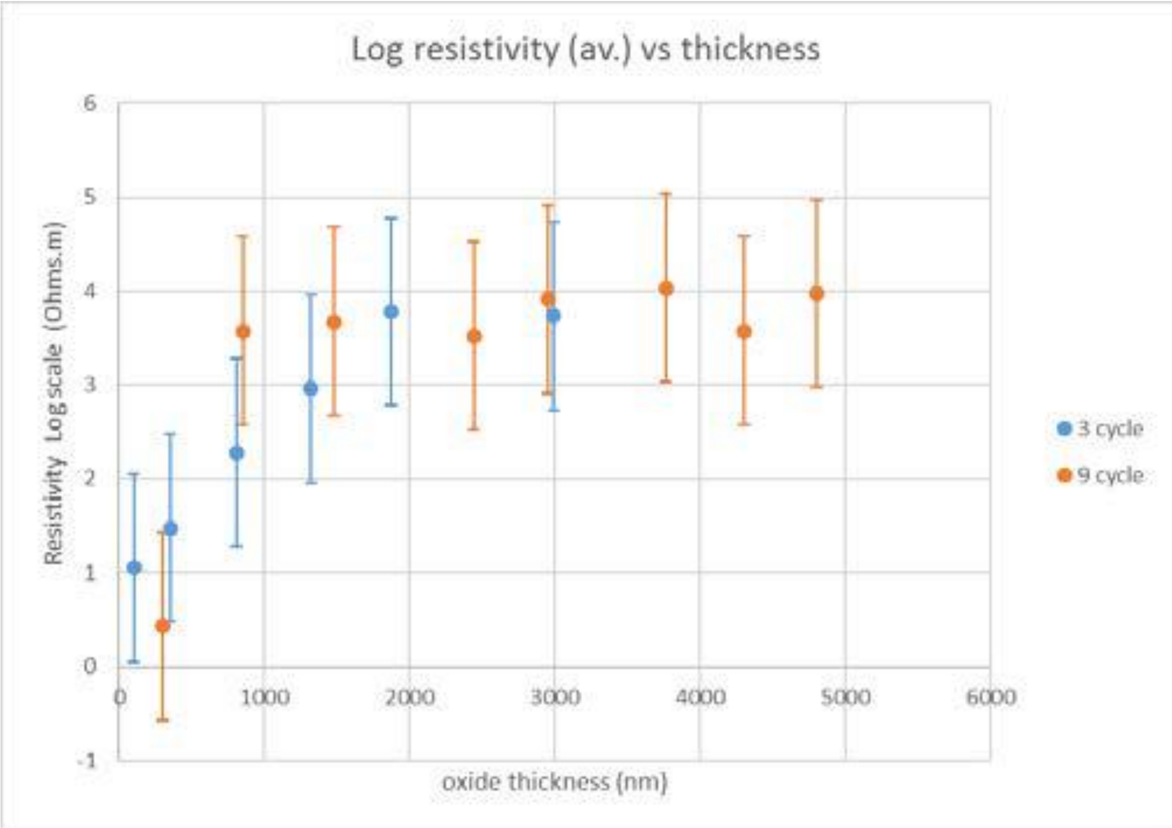


Fig. 3: SEM images showing micromanipulators contacting Pt electrodes.



Graph 1: The results from 3 and 9 cycles Zircaloy-2 LK3 comparing oxide thickness (from metal oxide interface) x axis and oxide resistvity.

Possible ways of FIB polishing quality improvement

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Focused Ion Beam (FIB) and Scanning Electron Microscopy (SEM) are essential techniques for detailed sample analysis. FIB is used for cross-sectioning the sample by ion milling or, when accompanied by a Gas Injection System (GIS), for creating protection layers by FIB induced deposition (FIBID). SEM is used for high-resolution imaging of the resulting cross sections, for charge compensation, or as a source of electrons for other analytical techniques. Two parameters of the cross-sectioning process are crucial – rapid milling rates and the high quality of the surface, with no damage or artefacts obstructing the analysis process.

The width and depth of the cross section, milled using Ga FIB, are usually ~10 μm. For larger cross sections with dimensions exceeding 100 μm, a Xe plasma FIB is much more convenient as the FIB milling rate increases approximately by a factor of 50 compared to Ga FIB [1]. E.g. in semiconductor failure analysis, this allows expanding FIB milling from thin IC layers to thick silicon structures, packaging materials and 3D devices where it is necessary to remove much larger volumes at a specific location. However, the surface milled by Xe plasma FIB is often not as smooth as the surface milled by Ga FIB. The cross-section quality is usually improved by polishing it from several directions to mitigate the curtaining effect, and has been applied on large Through Silicon Vias (TSV) samples [2] to mitigate the curtaining effect. Unfortunately, this method makes cross-sectioning more difficult and less accurate. To overcome this drawback, an improved method has been developed by using a multi-tilt sample stage (called a Rocking stage). It allows forward tilt compensation of the so-called taper angle and additional sequential tilting also in the plane of the cross section which enables simultaneous SEM observation of the curtaining mitigation process [3]. While this method delivers significantly reduced curtaining effect with most samples, there are some materials which exhibit quite poor cross section quality due to FIB-induced ripples [4, 5]. These ripples emerge during polishing while forming terraces, stairs and pillars and are more pronounced on plasma FIB instruments due to their larger spot sizes. However, we have also observed them on Ga FIB when using beam currents exceeding 10 nA. Therefore, we have proposed a method for mitigation of such artifacts, called TRUE X-sectioning and utilizing a silicon mask placed above the sample surface [6]. As a newest contribution to this research, we are including a study of influence of stage rocking sequences and angles on FIB polishing process, by using a compact and fully eucentric Anti Curtaining Table (ACT) from Kleindiek Nanotechnik, see Fig. 1.

[1] T. Hrnčíř *et al.*; *Novel plasma FIB/SEM for high speed failure analysis and real time imaging of large volume removal*, ISTFA 2012: Conference Proceedings from the 38th International Symposium for Testing and Failure Analysis (**2012**), 26.

[2] F. Altmann *et al.*; *Cross Section Analysis of Cu Filled TSVs Based on High Throughput Plasma- FIB Milling*, ISTFA 2012: Conference Proceedings from the 38th International Symposium for Testing and Failure Analysis (**2012**), 39.

[3] T. Hrnčíř *et al.*; *Advances in FIB-SEM Analysis of TSV and Solder Bumps - Approaching Higher Precision, Throughput and Comprehensiveness*, ISTFA 2014: Conference Proceedings from the 40th International Symposium for Testing and Failure Analysis (**2014**), 136.

[4] MoberlyChan *et al.*; *Fundamentals of focused ion beam nanostructural processing: Below, at, and above the Surface.*, MRS Bulletin Vol. 32 (**2007**), 424.

[5] R. M. Bradley, J. M. E. Harper; *Theory of ripple topography induced by ion bombardment*, J. Vac. Sci. Technol. A 6 (**1988**), 2390.

[6] T. Hrnčíř *et al.*; *How to achieve artefact-free FIB milling on polyimide packages,* ISTFA 2016: Conference Proceedings from the 42th International Symposium for Testing and Failure Analysis (**2016**), 642.

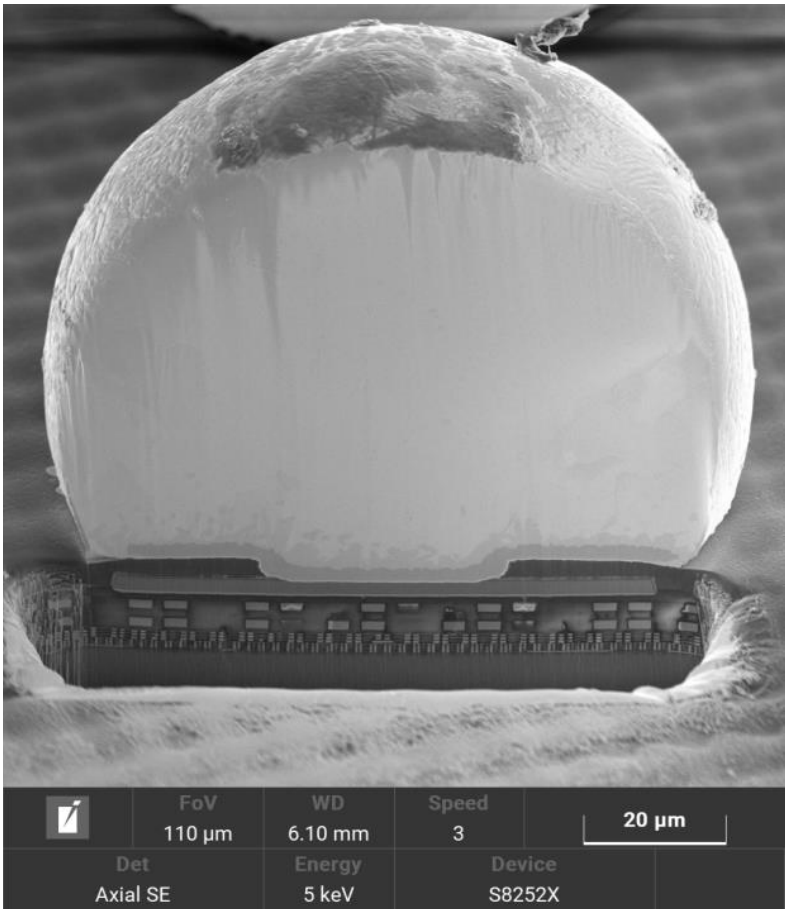

Fig. 1: An example of rapid high-quality polishing of 100 µm solder bump by using ACT stage continuously tilting ±10 degrees.

Optimisation of FIB Lift-Out with Analytics-Part 2

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A generic FIB process for preparing a lift-out sample (e.g. for TEM lamella samples or 3D) has been documented [1], and is shown in Fig. 1. This paper further develops this concept. It describes the integration of analytical tools and sample manipulation, on FIB-SEM, which both facilitates the identification of areas of interest and optimises the milling workflow.

EDS offers chemical analysis and spatial distribution in the form of quantitative analysis and X-ray mapping. EBSD delivers microstructural characterisation, studying phase relationships, local mis-orientations and grain properties. These techniques can be applied both to bulk samples and TEM lamella.

Recent innovation in these analytical tools makes this more powerful. For example, regions with a distinct chemical signature can be easily isolated through real time X-ray mapping. In addition, combining this chemical data with the crystallographic EBSD data, gives a full microstructural characterisation from which to identify specific areas of interest. Importantly, recent improvement in the speed to data acquisition, both of EDS and EBSD, means that the collection of analytical data can form part of this workflow, without prohibitive time constraints.

This sample characterisation can be used to optimise the milling process for lamella preparation. By understanding the nature of the sample the orientation of the lift-out specimen is optimised through rotation on the nanomanipulator. This is combined with an EDS technique which quantifies lamella thickness, up to 2 μm [2]. This technique also assists in identifying both ion beam damage not visible by imaging, illustrated in Fig. 2.

[1] J Lindsay, J. Sagar, J. Goulden Optimisation of the FIB Lift-Out Process through the Addition of Analytic Tools, EUFN · 2018

[2] C. Lang, M. Hiscock, M. Dawson C. Hartfield. Local thickness and composition analysis of TEM lamellae in the FIB, Microelectronics Reliability 54 · September 2014

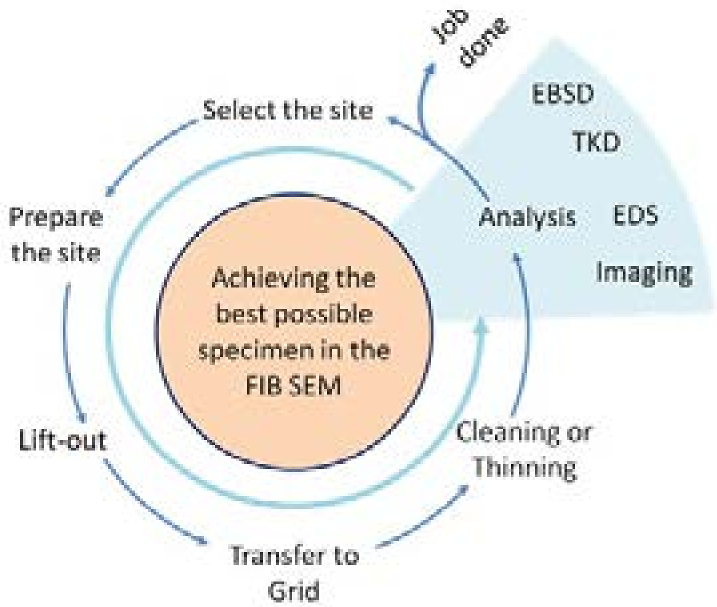

Fig. 1 Generic schematic of a FIB lift-out process, traditionally analytics tools are only used at the start or end of this process. The application of analytical tools to optimise this process will be demonstrated.

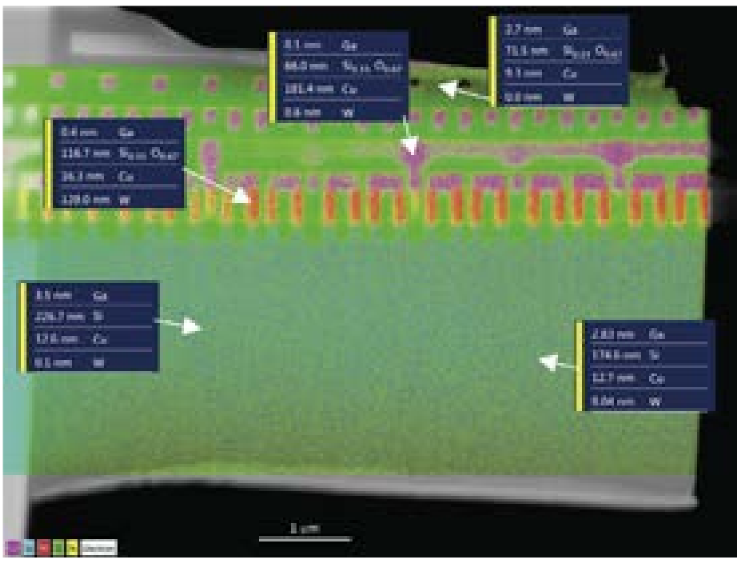

Fig. 2 EDS map from a thick, aggressively milled TEM lamella. EDS layer thickness measurements indicate the sample is too thick for optimal TEM measurements and significant Ga implantation and Cu redeposition.

3D reconstruction of organic matter in the Black Shale, South Africa

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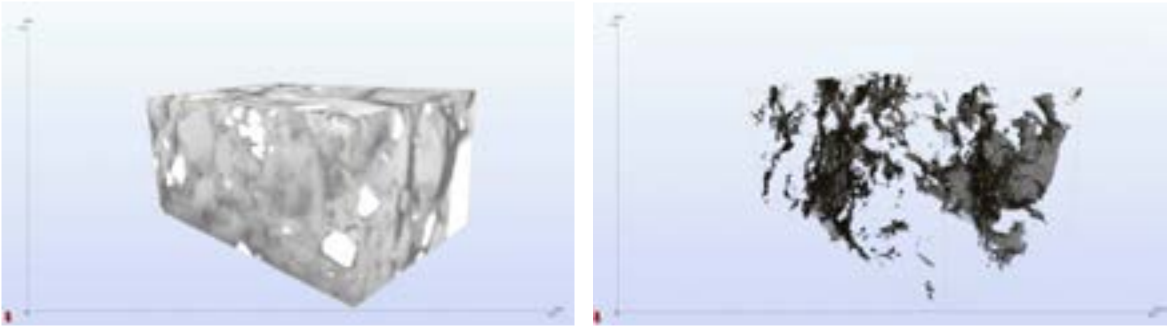
The primary target for shale gas in the South Africa is the Lower Ecca Group (Karoo Supergroup). While most of the black shale in the southern Lower Ecca Group is over mature and consists of high Total Organic Carbon contents (up to 14% locally). In unconventional hydrocarbon reservoirs, gas is present in very small pores and adsorbed by the organic matter (e.g., Chere 2015; Chukwuma et al., 2018). Thus, the composition, texture and porosity of the gas-bearing shales are important not only for resource estimation, but also for successful drilling campaigns. However, the organic matter distribution and estimation in the black shale is difficult. Here, the porosity and permeability of the black shale's were visualized using the FIB-SEM tomography technique and found to be the porosity confined mostly to the organic matter. Further, we produced the 3D reconstruction model of organic matter in black shale to determine the extent of its connectivity using Avizo and other advanced image processing softwares (Fig. 1).

[1] Chere, N., 2015. Sedimentological and geochemical investigations on borehole cores of the Lower Ecca Group black shales, for their gas potential: Karoo basim, South Africa. MSc Dissertation. Nelson Mandela University, Port Elizabeth, South Africa.

[2] Chukwuma, K., Bordy, E.M., Coetzer, A. 2018. Evolution of porosity and pore geometry in the Permian Whitehill Formation of South Africa – A FE-SEM image analysis study. Marine and Petroleum Geology 91: 262-278

Fig. 1: a) 3D reconstruction of lower Ecca Group Black Shale material (b) and organic matter including porosity in the lower Ecca Group Black Shale.

a) b)



Elastic Moduli of Single Crystals Probed by FIB-Fabricated Micro-Resonators

Amelia Estry[1,2,] Toni Helm[1], Maja D. Bachmann[1], Carsten Putzke[1,2], K. R. Shirer[1], K. A. Modic[1], X. Huang[1,2], Tino Zimmerling[1], J. J. Diaz[1,2], Eric D. Bauer[3], Filip Ronning[3], Marcus Schmidt[1], Philip J. W. Moll[1,2]*

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[2]École polytechnique fédérale de Lausanne (EPFL), L'Institut des Matériaux, CH-1015 Lausanne, Switzerland
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The elastic properties of a metal contain valuable information about its properties and thus are instrumental to understand newly discovered materials. We develop a new measurement technique based on resonant measurements of elastic micro-resonators from exotic materials discovered in inorganic chemistry. Measurements of the elastic properties in the plastic regime have been very successful in the realm of FIB-fabricated micropillars. Here I take a related but strikingly simple alternate approach: The resonance frequency of a mechanical oscillator directly encodes the materials stiffness as the effective spring constant. I carve micro-cantilevers with precisely known orientations and geometries along selected crystal directions from small single crystal samples. With the shape precision allowed by the FIB, we can create thin cantilevers in the micron scale to explore single domains and domain walls – a capability unique to this technique. Critically, by studying the mechanical resonance we remain in the linear regime, setting this approach apart from micropillars. Supporting finite-element based simulations will allow me to determine the elastic constants from the measured resonance frequencies of the cantilevers. Additionally, the cantilevers can be designed such that either the twisting or the shear modes become favorable – allowing us to tune the experiment to explore interesting elastic deformations of the material. These cantilevers are excited by a piezo-electric transducer and the resonance frequencies are detected optically, such that the elastic moduli can be directly calculated from the eigenfrequencies. This novel technique will provide new insights into quantum matter, particularly in the cases of newly discovered materials where no large-scale crystalline samples are yet available.

FEBID technology for high sensitive and high resolution investigations performed using MEMS/NEMS devices

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Focused electron beam (FIB) and scanning electron microscopy (SEM) enable fabrication of three dimensional (3D) structures at the locations defined with single nanometer resolution. To the technologies which are based on FIB and SEM methodology belongs focused electron beam induced deposition (FEBID) making it possible to integrate various conductive and magnetic structures with micro- and nanoelectromechanical systems (MEMS and NEMS). During the presentation we will present a method of fabrication and characterization of FEBID nanogranular resistors (NGRs) fabricated basing on the platinum and cobalt precursors, which can be used as the deflection detector of silicon nitride microbridges. In our experiments we applied the thin film double clamped microstructures with platinum metallization as stable structures of resonance frequency reaching MHz. The deflection sensitivity of the NGR strain sensor was calibrated in the contact atomic force microscopy (AFM) technology. We will also report on measurement technology enabling electrical detection of resonance microbridge behavior. Moreover characterization of magnetic cobalt FEBID magnetic nanostructures applied for excitation of the so called small AFM cantilevers (of the length of 20 microns) will be presented as well. It is extremely important to be able to excite the structure vibration in the reliable way taking into account that that the cantilevers dimension rule out integration of the "on board" deflection actuator. Precise tuning of the amplitude and frequency of the structure vibration is possible when the entire structure is immersed in the magnetic field excited by an external coil. Further structure functionality is added when on the standard AFM tip a FEBID nanotip is fabricated, which opens completely new surface measurement possibilities as the investigation throughput and resolution are significantly increased.

References

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[2] J. Longenecker, E. Moore, & J. Marohn, *Journal of Vacuum Science & Technology B: Microelectronics and Nanometer Structures*, *29*(3), (2011) 032001.

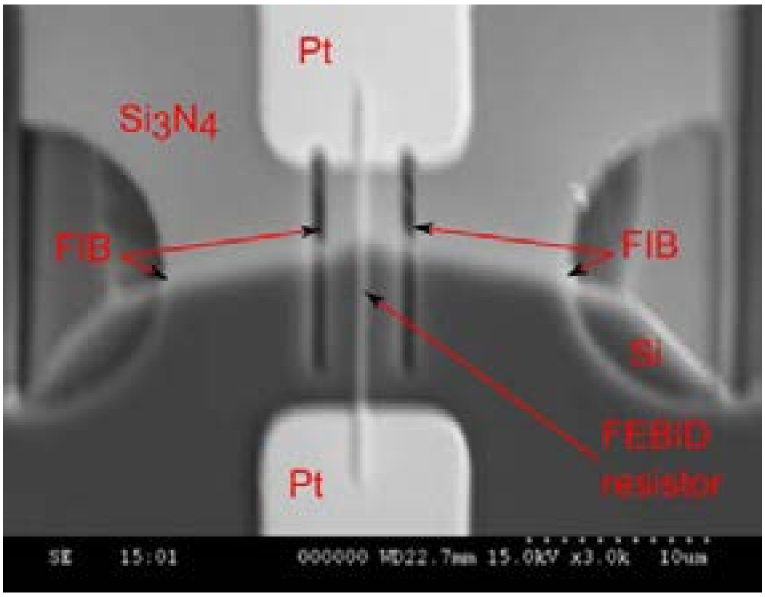

Fig. 1: Silicon nitride microbridge with platinum thin film metallization before FEBID NGR deposition.

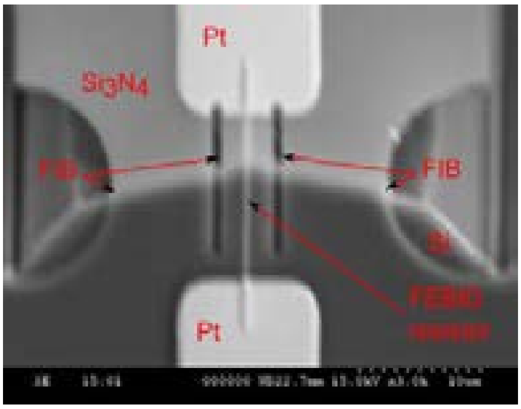

Fig. 2: NEMS bridge after the FIB modification with deposited FEBID high sensitive piezoresistive deflection detectors

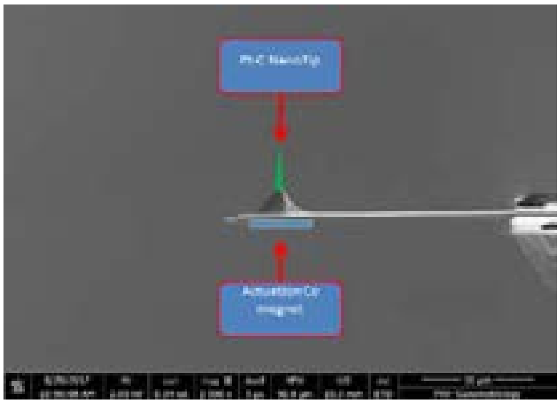

Fig. 3: Small AFM cantilever with FEBID actuation structure and FEBID nanotip

Using a grid after electrical failure localization to highlight the position for the Focused Ion Beam cross section

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Focused Ion Beam (FIB) microscopes can be used for various modifications.
A special application is the deposition of grids, which are used for localizing electrical failure on the back or front side of an integrated circuit (IC) chip.
There are several different methods for electrical failure localization, which are chosen depending on the number of different layers and the doping levels of the chip substrate.
It is primarily necessary to thin the backside of the chip, as the high doping level of the substrate would absorb the emitted light from the hotspot. The thinning of the backside, the removal of the silicon substrate, can be done mechanically with a parallel polishing tool. A different approach to thin is to use the VION Plasma FIB from Thermo Fisher. In order to save time or reduce damage to the chip, it can be used to thin the substrate partially on the expected position of the failure.

To create a front side grid, oxide deposition may be used, as it displays a good contrast of a dark oxide grid. Grids are deposited with a single beam Xenon Plasma FIB VION from Thermo Fisher which uses Xenon ions. One benefit of using the Xenon Plasma FIB is the non-existing Ga contamination of the sample. If gallium contamination is no problem it is nevertheless possible to create a front side grid with a dual beam Focused Ion Beam tool, belongs to the configuration of the tool. One of the case studies showed good performance for new substrates like GaN with a deposited carbon grid created with the dual beam FIB Strata 400 from Thermo Fisher.
A different possibility of creating a grid on the backside is to cut trenches into the backside with the Focused Ion Beam.
After creating a suiting grid the electrical failure analyses is performed again and the position of the failure is clearly visible on the deposited grid. One trick is to use Image Overlay to isolate the deviation of the failure position. With this method the consequent FIB cut can be positioned very well at the position of the hot spot. Finally the failure can be visualized and the root cause for the failure analysis can be detected.

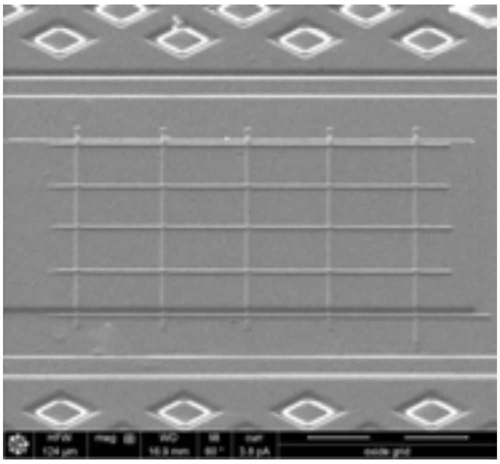

Fig. 1: oxide grid, front side

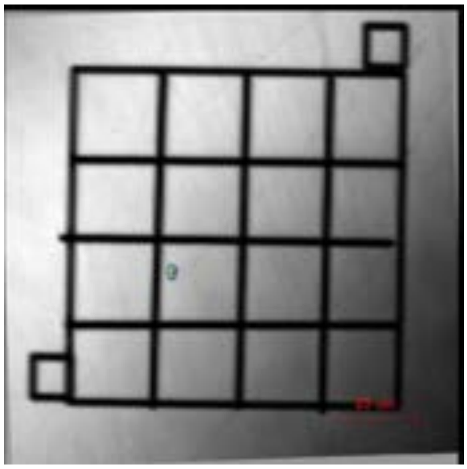

Fig. 2: back side grid with electrical spot
(Photo Emissions Microscopy)

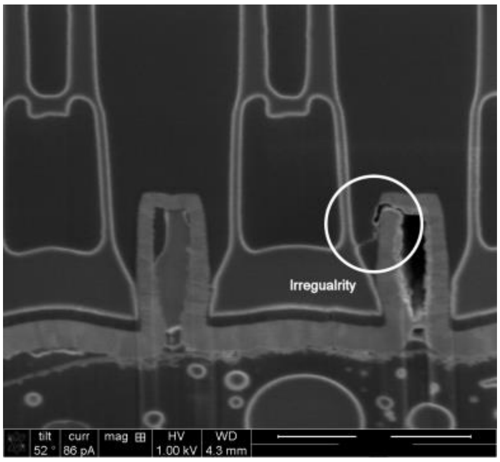

Fig. 3: Irregularity / FIB cross section

Electron 'lensing' in micro-structured semimetal Cd_3As_2 fabricated by Focused Ion Beam (FIB)

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When a magnetic field B is applied to a high-mobility material, the conductivity differences between different directions induced by the magnetic field can be huge. This enormous anisotropy can strongly impact the current distribution [1]. For example, in semimetal Cd_3As_2, with a mobility ~40000 $cm^2/V{\cdot}s$ at 2K, magnetic field induced anisotropy leads to a beam-like current flow along the magnetic field direction. This electron 'lensing' effect can be used to get different voltages simultaneously at voltage contacts far away from current contacts in a multi-channel device.

Focused Ion Beam(FIB) is an ideal tool to micro-machine such a 3D device out of a crystalline sample. A typical false-color Scanning Electron Microscopy (SEM) image of a micro-structured Cd_3As_2(purple) sample with Au(yellow) contacts electron 'lensing' device fabricated by FIB is showed in Fig.1a. A current is injected from contact a to b. the bottom voltage V_{cd}, V_{de} and V_{ef} are measured between the contact pairs c-d, d-e and e-f. At a temperature of 2K and a magnetic field of 14T, the magnetic field is rotated clockwise in the plane. When the magnetic field is parallel to the blue solid line, the angle is 0°. The angular dependence of the three bottom voltages is shown in Fig.1b. Because of the electron 'lensing' effect as illustrated in Fig.1a. When the magnetic field is parallel to the red dash line, V_{cd} gets the maximum, V_{de} gets the minima and V_{ef} is around zero. Similarly, when the magnetic field is applied along the blue dash line and black dash line. The three bottom voltages are all different.

[1] Pippard A.B. *Magneto-resistance in metals*; Cambridge University Press (1989), 43–46.

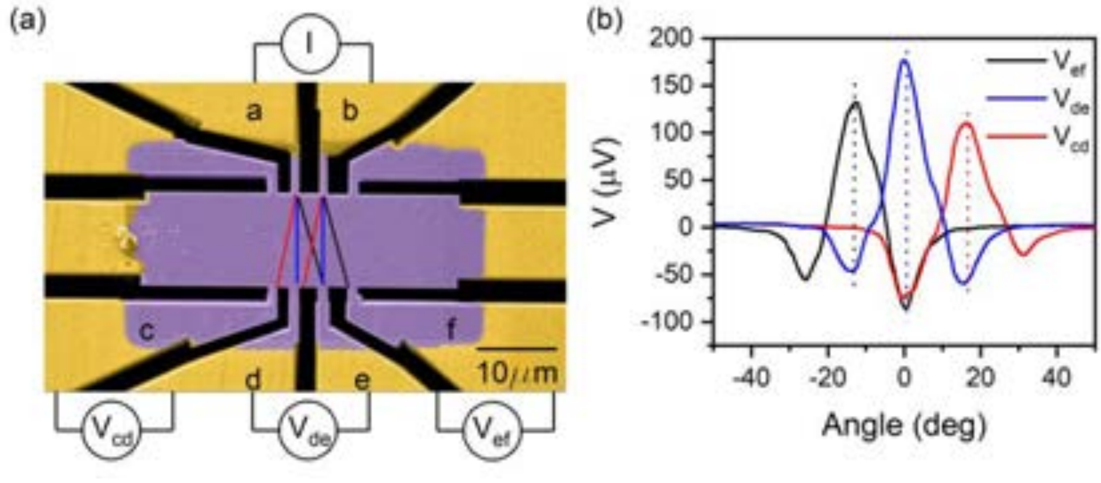

Fig1: (a): False color scanning electron microscopy(SEM) image of a Cd_3As_2(purple) multi-channel device with Au(yellow) contacts fabricated by FIB. At a temperature of 2K and a magnetic field of 14T, the magnetic field is clockwise rotated in the plane. A current is injected from contact a to b, and the bottom voltages V_{cd}, V_{de} and V_{ef} are measured with the angle between the magnetic field and the blue solid line. The colored solid lines are the schematic illustration of the electron beams when the magnetic field is along different angles shown by the dash lines in (b). The experimental data of bottom voltage V_{cd}, V_{de} and V_{ef} with the angle when source current I=10μA from contact a to b are shown in (b).

Fabrication of aperture probes for SNOM using focused ion beam

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The use of focused ion beams for the formation and modification of probes for scanning probe microscopy is now one of the promising ways to create SPM probes with unique parameters [1]. At the same time, aperture probe formation technologies for scanning near-field optical microscopy (SNOM) are highly demanded. Currently, aperture cantilevers for SNOM are produced using technologies in which the key operation is focused-beam milling of the tip for the formation of desired aperture. However, this method is rather complicated and does not allow changing the parameters of the tips in a wide range [2].

This paper describes a new technology for the manufacture of aperture cantilever tips, based on a focused ion beam-induced deposition of carbon. The key feature of the proposed technology is the formation of a hollow conical tip of the probe on a beam of a standard AFM cantilever sequentially by deposition of an array of structures in the form of concentric circles with decreasing diameter as shown at Fig.1. This procedure can be made based on a standard cantilever with a worn or broken tip. To do this, an entrance aperture with a diameter of 5 to 15 microns is first formed in the cantilever beam by ion beam milling (depending on the desired probe parameters). After that, a pattern consisting of a set of concentric circles with gradually decreasing diameter is developed. The number of circles, as well as the values of their diameter and height determine the final parameters of the tip of the probe, including the diameter of the exit aperture. If necessary, the formed tip can be covered with a thin layer of gold to improve the parameters of the probe. After such an operation, it may be necessary to restore the exit aperture of the probe, which is produced by the FIB local milling.

The developed technology allows the manufacture of aperture cantilevers for SNOM with specified tip parameters based on standard probes.

This work was supported by the Russian Science Foundation Grant No. 18-79-00175.

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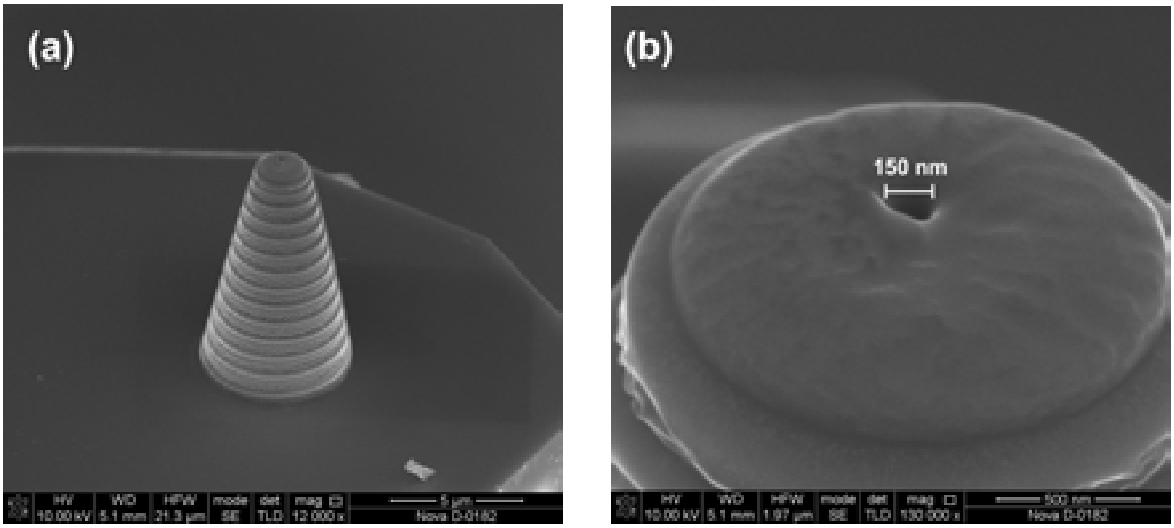

Fig. 1: SEM images of FIB-fabricated SNOM probe (a) with an aperture of 150 nm (b)

From FIB to advanced FIB Nanofabrication: True 3D, Multi Ion Species and Large Area Nanopatterning

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Raith's FIB/SEM multi-technique nanofabrication instrumentation routinely offers nanopatterning resolution with sub 10nm feature sizes. It is well suited for research on the nm-scale with its excellent beam properties as well as beam control. Advanced hardware, patterning control and patterning strategies for this tool come into operation in order to fully unlock its true nanofabrication potential to secure stable, reproducible and highest precision for efficient operation.

In order to meet the requirements for advanced FIB nanofabrication we have integrated our latest generation of FIB-SEM technology into a true lithography platform that is optimized for nanometer scale patterning over large areas and extended periods of time. 2D-and 3D-applications in quantum technologies, optics, plasmonics, nanofluidics and nanobiotechnology, such as X-ray zone plates [1], large area gratings [2], plasmonic arrays, and wafer-scale nanopore devices have successfully been demonstrated. Innovative stitching error free patterning modes enabled by a high precise laserinterferometer controlled stage provide access from nm-scale patterning to the cm-regime.

We present latest applications that demonstrate the capabilities and the full potential of fabricating complex high resolution, large area nanostructures exceeding the size of common write fields utilizing a FIB-centric nanofabrication system. A highly automated workflow, milling strategies adapted to the pattern geometry, long-term stability and stitch-free continuous writing modes open the door to a new world of applications. The choice of various ion species further expands the range of options enabling Ga-free milling and the selection of appropriate ion species.

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[1] A. Nadzeyka, L. Peto, S. Bauerdick, M. Mayer, K. Keskinbora, C. Grévent, M. Weigand, M. Hirscher, G. Schütz, Microelectr. Engineering 98 (2012), 198-201. http://dx.doi.org/10.1016/j.mee.2012.07.036

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Crossing borders of material science – a new approach of aerogel preparation for electron microscopy

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Aerogels are innovative materials exhibting a porosity of up to 99.98% demonstrating a wide range of possible applications. To get a better understanding of the morphology of these materials, electron microscopy proves to be an important analytical method. Since classic metallographic embedding techniques are not suited for organic aerogels, new approaches are necessary to enable more efficiency and possibilities of data and image acquisition. With regard to electron microscopy, organic aerogels show a similar behavior to biological samples. Therefore, a possible approach is to adept biological preparation techniques to material science samples where conventional techniques find their limits to image and measure the aerogels' pore sizes, interconnection and morphology. Resins used for biological samples are comparable to organic aerogels constituents in regard of mechanical properties, don't shrink during hardening and infiltrate the sample very effectively. These properties are also beneficial for the investigation of highly porous aerogels. The preparation starts with the embedding of the aerogel in the appropriate resin. Subsequently, the resin is trimmed very precisely with an ultramicrotome to bring the aerogel to the surface (Fig. 1). Now the sample is well suited for further processing in a dual beam system combining SEM and FIB. By milling cross-sections into the embedded sample, planar sections can be analyzed to give information about the particle structure and size within the aerogel (Fig. 2). By creating a slice and viewing tomogram, it is even possible to analyze the structure of a whole volume and getting the data of up to 1500 SEM images in one measurement, thus investigating the pore -size, -morphology as well as structure and volume of the aerogel. Additionally, a 3-dimensional reconstruction of the images can be achieved. To get further information about nano-pores and their morphology, lamella for TEM investigations can be prepared out of the embedded sample, preventing an overlapping of particles as observed in the classic approach (Fig. 3).

[1] Lorenz Ratke; Aerogels – A Practical Workshop; 5. International Summer School on Aerogels (2018)

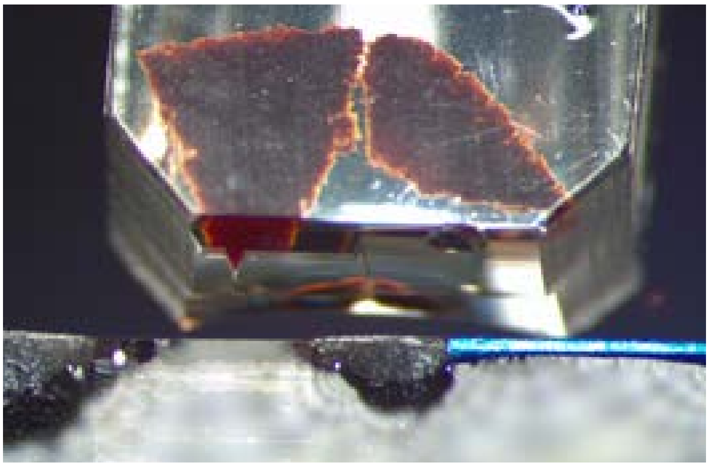

Fig. 1: Organic aerogel embedded in Embed-812, an epoxy resin used for biological EM sample preparation trimmed with an ultramicrotome.

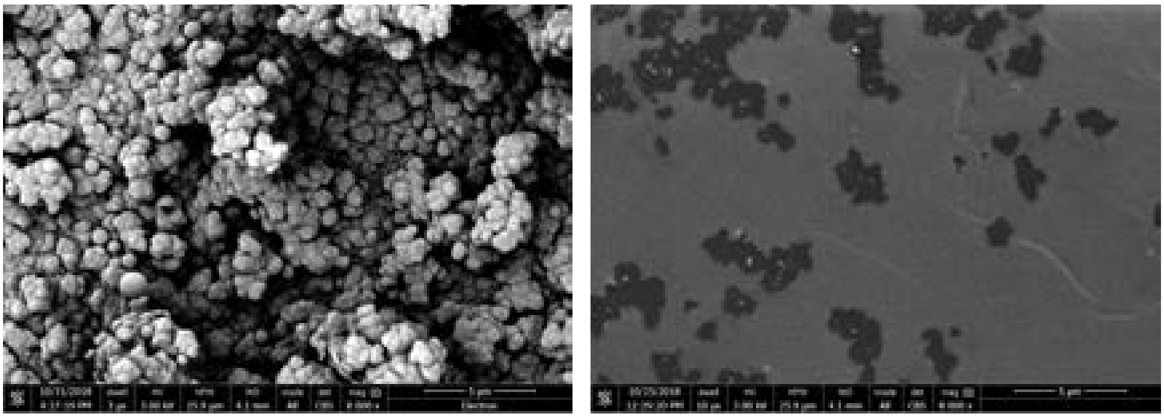

Fig. 1: SEM images of an organic aerogel prepared by the classic approach (left) without embedding and with embedding (right).

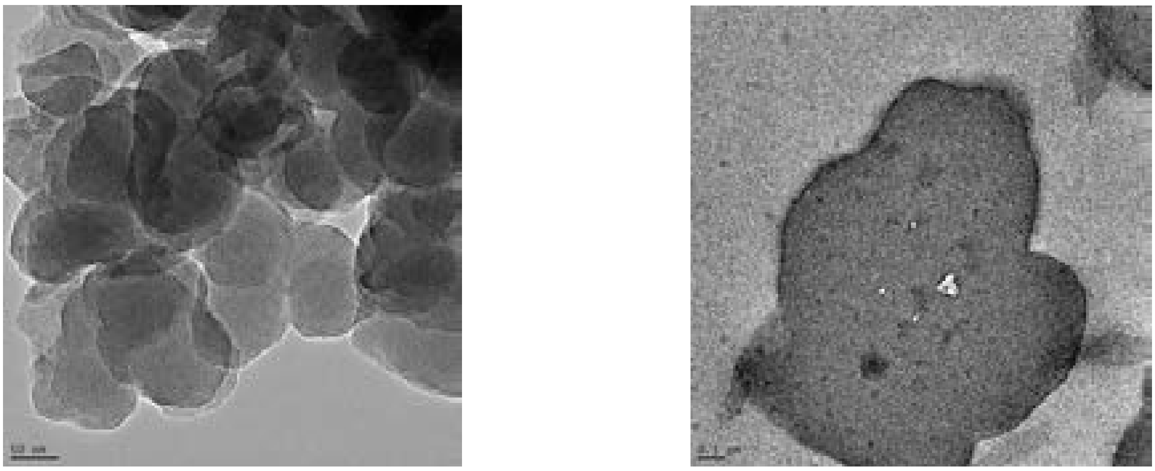

Fig. 2: TEM images of an organic aerogel. The left image shows an aerogel on a TEM grid prepared by mortar grinding, the right one an ultramicrotome slice of an embedded sample.

In-situ analysis of FIB milled structures using AFM-in-SEM LiteScope™

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In-situ analysis of structures prepared by focused ion beam (FIB) is an application useful in a wide range of scientific fields. Novel AFM instrument LiteScope™ with unique Correlative Probe and Electron microscopy (CPEM) technique is presented. It offers advantages of in-situ analysis of samples after FIB or GIS modification including depth/height profiling, roughness estimation, 3D imaging or local spectroscopy measurements.

The AFM microscope LiteScope™ is designed for easy and fast integration into a wide range of SEMs. It can be mounted directly onto the SEM stage and operated in a tilted position which makes it compatible with FIB etching. This brings an opportunity to investigate the sample right after the FIB surface modification without breaking the vacuum, moving the sample or without the ambient atmosphere influence. It shortens the time needed optimization of the milling procedure and sputtering rate. Since SEM is used for AFM tip navigation, no marks or other common tricks are needed to localize the area of interest for further analysis, even in nanoscale [1]. As mentioned earlier, LiteScope™ is equipped with CPEM technique for true correlative simultaneous measurement of AFM and SEM images.

This type of analysis will be shown on several examples including sputtering rate estimation on a sensitive sample of CdTe (see Fig.1) or investigation of integrated circuit layer by layer. LiteScope™ has been successfully used for in-situ advanced surface characterization of various materials.

[1] NenoVision s.r.o., LiteScope™, NenoVision, 2018, www.nenovision.com/

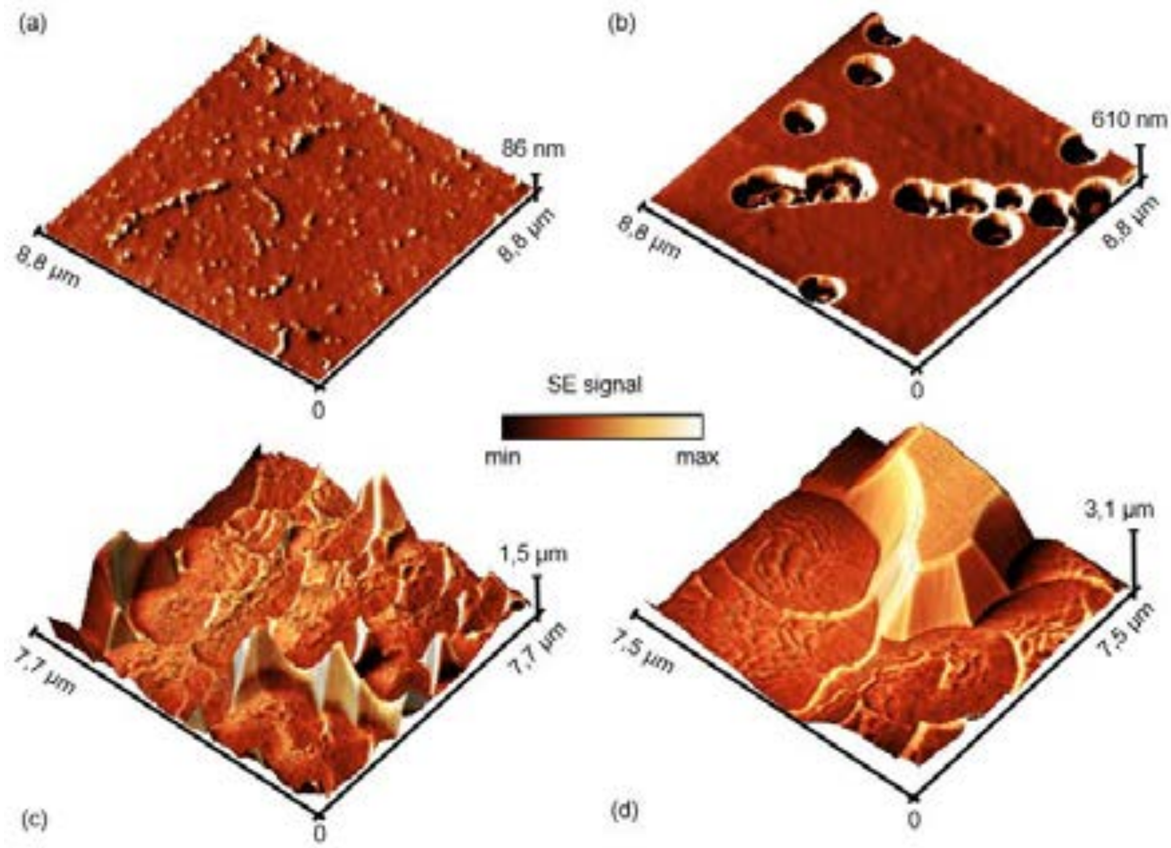

Fig. 1: 3D CPEM view of simultaneously measured SEM and AFM images of CdTe. FIB depth of (a) 0.1 µm (b) 0.6 µm (c) 1.5 µm (d) 2.5 µm .

Time	Status	Titel	Presenter	Affiliation
	EuFN Workshop Day 1 (June 12th)			
12:30	Get Together, Registration			
13:00	Official Opening		S. Facsko	Head of IBC, HZDR
13:20	Invited	Multigas Plasma FIB (from cell biology to lens manufacturing)	de Marco, A.	Monash University Clayton
13:55	Exhibitor	in situ TEM Lamella LiftOut for Backside Preparation - LO&Flip	Smith, A.	Kleindiek
14:15	Contributed	FIBing Gallium Arsenide (and others): experiments for understanding and limiting artefacts	Audoit, G.	Leti Grenoble
14:35	Exhibitor	Preparation of TEM samples and their observation by means of an adjustable tilt holder	Hrncir, T.	Tescan
14:55	Contributed	Channeling Effects in Gold Nanoclusters under He Ion Irradiation: A Molecular Dynamics Study	Ghaderzadeh, S.	HZDR
15:15	Coffee Break			
16:15	Invited	Microstructure is the “know-it-all” - classification approaches based on 3D-tomography, data mining and deep learning methods	Mücklich, F.	Uni Saarbrücken
16:50	Exhibitor	AFM-in-SEM LiteScopeTM: Tool for in-situ analysis of FIB modified materials	Novotna, V.	NenoVision
17:10	Exhibitor	In-Situ Correlative AFM/SEM/FIB analysis of FIB-treated samples	Schwalb, C. H.	GETec
17:30	Contributed	Feature Adaptive Sampling for Fast Image Acquisition in FIB and SEM	Pauly, C.	Uni Saarbrücken
17:50	Contributed	Removal of Curtaining Eects by a Variaonal Model from Mathemacal Image Processing	Fitschen, J. H.	Kaiserlautern
	EuFN Workshop Day 2 (June 13th)			
9:00	Get Together			
9:15	Invited	Why FIB is essential for analysis in semiconductor industry	Limbecker, P.	Globalfoundaries
9:50	Exhibitor	Latest advances of JEOL`s JIB-4700F Multi Beam System in focused ion-beam lithography	Harzer, T.	JOEL
10:10	Contributed	Defect production in supported two-dimensional materials under ion irradiation from atomistic simulations: the substrate is crucial	Kretschmer, S.	HZDR
10:30	Coffee Break			
11:00	Exhibitor	Advances in Multiple Ion Species Plasma FIB Technology	Prokhodtseva,A.	Thermo Fisher Scientific
11:20	Exhibitor	An Improved Experimental Setup for the Preparation of Back Side and Planar View TEM samples	Perez Willard, F.	Zeiss
11:40	Exhibitor	Site-specific Atom Probe Tomography Sample Preparation Method by Orthogonal FIB-SEM Column Layout	Onishi, T.	Hitachi
12:00	Lunch			
13:30	Invited	Ion Sources for Focused Ion Beams – Present Status and Prospective Developments	Bischoff, L.	HZDR
14:05	Exhibitor	New Applications in advanced FIB-SEM Nanofabrication with a FIB-centric Lithography System	Stodolka, J.	Raith
14:25	Contributed	SIMPLE – A FIB for Deterministic Single Ion Implantation	Cassidy, N.	University of Surrey
14:45	Exhibitor	3D EBSD and EDS Developments	Larsen, K.	Oxford Instruments
15:05	Contributed	Combined laser and FIB preparation for TEM planar analysis of flash memory cells	Simon-Najasek, M.	FhG IMWS
15:25	Coffee Break			
16:20	Contributed	How FIB induced artefacts influence in situ characterization in the TEM	Berthier, M.	Protochips EMEA
16:40	Contributed	Development of a new integrated instrument for accurate and reproducible physico-chemical characterisation of nanoparticles	De Castro, O.	LIST
17:00	Contributed	TOF-SIMS with highest lateral resolution by pulsing the Ne-GFIS in a HIM	Klingner, N.	HZDR
17:20	Contributed	Combination of the FIB-TOF-SIMS technique with GIS – increasing the ionization probability and sputtering rates of thin films	Priebe, A.	EMPA
	Conference Dinner			
	EuFN Workshop Day 3 (June 14th)			
9:00	Get Together			
9:15	Invited	Ultrastructural changes accompanying the intracellular mineral formation in alga E. huxleyi': a cryo-FIB/SEM study	Bertinetti, L.	MPIKG Potsdam
9:50	Contributed	Avoiding amorphization during ion beam irradiation and critical dimension reduction of nanostructures	Hlawacek, G.	HZDR
10:10	Contributed	Ultra-fast growth of W-C metal nanostructures by Focused Ion Beam Induced Deposition under cryogenic conditions (cryo-FIBID)	De Teresa, J. M.	University of Zaragoza
10:30	Coffee Break			
11:00	Contributed	Using FIB as a broad ion source for nanofabricaton on AIII-BV(InSb) semiconductors	Jany, B. R.	Jagiellonian University
11:20	Contributed	Creating mesoscale ballistic transport devices from ultra-pure quantum materials	Bachmann, M. D.	MPI-CPFS Dresden
11:40	Contributed	Out-of-plane Transport in ZrSiS, ZrSiSe, and HfSiS Microstructures	Shirer, K. R.	MPI-CPFS Dresden
	Concluding Remarks			
	Lunch			