Focused Ion Beam Induced Nanoscale Phase Transitions in Layered Structures

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Specimen preparation for Transmission Electron Microscopy (TEM) using Focused Ion Beam (FIB) is a common method [1]. While it offers many advantages over other methods, like site specific specimen preparation, it also suffers from many disadvantages. Artifacts induced by FIB range from Ion implantation to thermal effects [2,3].

In this work, we investigate thermal effects induced by FIB milling in chalcogenide-based 2D layered Sb_2Te_3 thin films covered by Cu layers. Sb_2Te_3 thin layers are epitaxially grown on p-type Si (111) substrates and polycrystalline samples grown on SiO_2 using pulsed laser deposition [4]. Dependent on beam current used during FIB lamella preparation and Sb_2Te_3 layer thickness, hole formation in the Cu layer, thickness change in the Sb_2Te_3 layer and nanoscale modifications are observed (Fig 1.). The structural changes are confirmed by in situ X-Ray Diffraction heating. The introduction of a separation layer (e.g. Pt) between the Cu and Sb_2Te_3 layers hinders thermal induced structural changes by FIB (Fig 2.). Moreover, Cr - Sb_2Te_3 and a Cu – GeTe layer systems show no modifications during preparation.

[1] T. Ishitani, H. Tsuboi, T. Yaguchi, H. Koike; Transmission electron microscope sample preparation using a focused ion beam; Microscopy (1994), 322.

[2] J. Mayer, L.A. Giannuzzi, T. Kamino, J. Michael; TEM sample preparation and FIB-induced damage; MRS bulletin (2007), 400.

[3] R. Schmied, J. E. Fröch, A. Orthacker, J. Hobisch, G. Trimmel, H. Plank; A combined approach to predict spatial temperature evolution and its consequences during FIB processing of soft matter; Physical Chemistry Chemical Physics (2014), 6153.

[4] H. Bryja, J.W. Gerlach, A. Prager, M. Ehrhardt, B. Rauschenbach, A. Lotnyk; Epitaxial layered Sb2Te3 thin films for memory and neuromorphic applications; 2D Materials (2021), 045027.

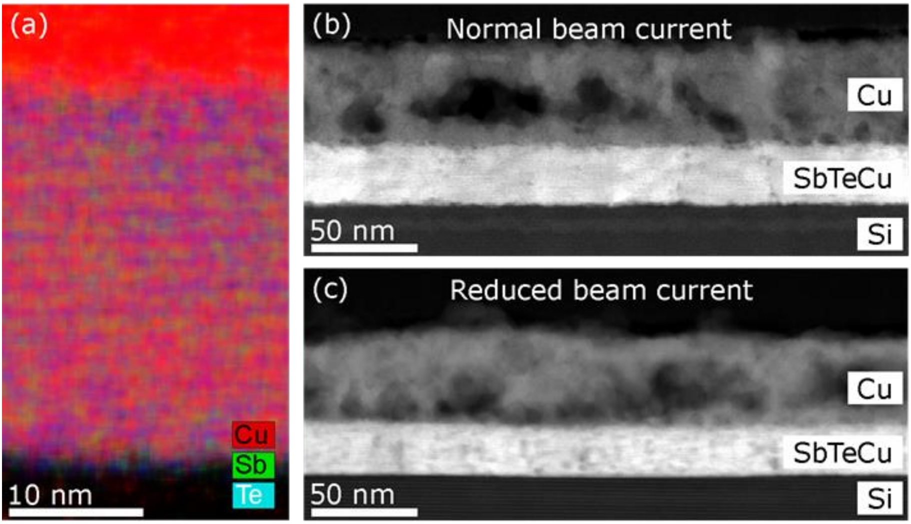

Fig. 1: (a) EDX Map of a lamella prepared with FIB, (b-c) Overview HAADF-STEM images of specimen prepared with normal and reduced FIB beam currents.

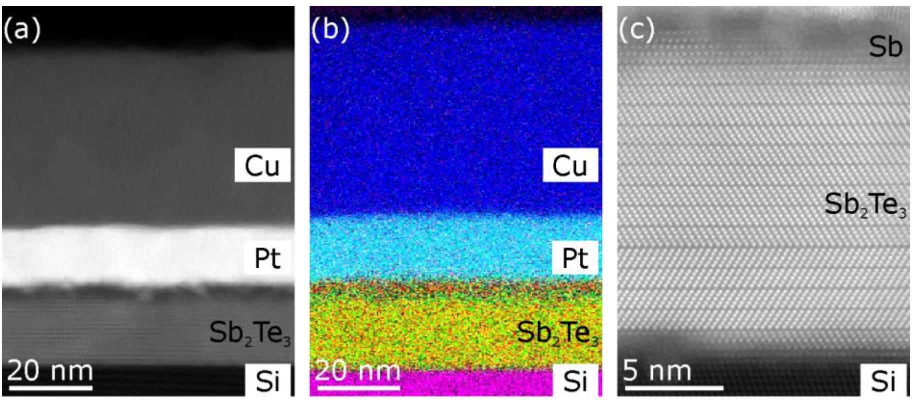

Fig. 2: Cu/Pt/Sb2Te3 layer stack. (a) Overview HAADF-STEM image. (b) Overview EDX elemental map. (c) Atomic-resolution HAADF-STEM image, showing initial Sb_2Te_3 structure and no redeposition of Cu.

The Manufacture of van der Waals Heterostructures Using He Ion Beam Patterning

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Polaritons in two-dimensional materials exhibit enhanced light-matter interactions, which makes them interesting for low-loss, highly confined light transport. A polariton is a quasiparticle that combines a photon with a dipole-carrying excitation in matter and is strongly dependent on the type and geometry of the material. The hybridization of polaritonic modes in different 2D materials may provide strong localization of plasmonic excitations with long propagation distances of phonon modes [1]. By modifying the geometry of van der Waals (vdW) heterostructures at the nanoscale, we tune hybrid polaritonic modes.

We fabricate and patterning of heterostructures based on single crystalline gold or silver flakes, graphene, and hexagonal boron nitride (hBN). For dry transfer we used polydimethylsiloxane (PDMS) and poly(propylene) carbonate (PPC) films due to their strong adhesion to 2D materials at room temperature. Therewith, single-layer to few-layer 2D materials were successfully transferred onto thin electron transparent membranes of silicon nitride.

To modify the geometry of the heterostructures at the nanoscale, a Zeiss Orion Nanofab microscope is then used for patterning by He and/or Ne ion beam milling (cf. fig. 1). As polaritonic modes are not only strongly influenced by geometry, but also by material quality, an important step in the study is therefore to investigate different currents, acceleration voltages, and ion types to determine what damaging effects they have on the crystalline lattice and the corresponding m material response. The optimization of the patterning routines is carried out with the help of FIB-o-Mat, which provides complete control over the beam path [2].

In the following step, monochromated, low-loss scanning transmission electron microscopy (STEM), electron energy-loss spectroscopy (EELS) [3] is used to map the optical properties of the fabricated heterostructures, and the results are compared to near-field optical methods.

[1] A. Woessner, M. B. Lundeberg, Y. Gao, A. Principi, P. Alonso-gonzález, M. Carrega, K. Watanabe, T. Taniguchi, G. Vignale, M. Polini, J. Hone, R. Hillenbrand, and F. H. L. Koppens; Nature Materials 14 (**2015**), 421.

[2] V. Deinhart, L. Kern, J. N. Kirchhof, S. Juergensen, J. Sturm, E. Krauss, T. Feichtner, S. Kovalchuk, M. Schneider, D. Engel, B. Pfau, B. Hecht, K. I. Bolotin, S. Reich, and K. Höflich; Beilstein J. Nanotechnol. 12 (**2021**), 304.

[3] O. L. Krivanek, N. Dellby, J. A. Hachtel, J. Idrobo, M. T. Hotz, N. J. Bacon, A. L. Bleloch, G. J. Corbin, M. V. Hoffman, C. E. Meyer, T. C. Lovejoy; Progress in ultrahigh energy resolution EELS; Ultramicroscopy Volume 203 (**2019**), 60.

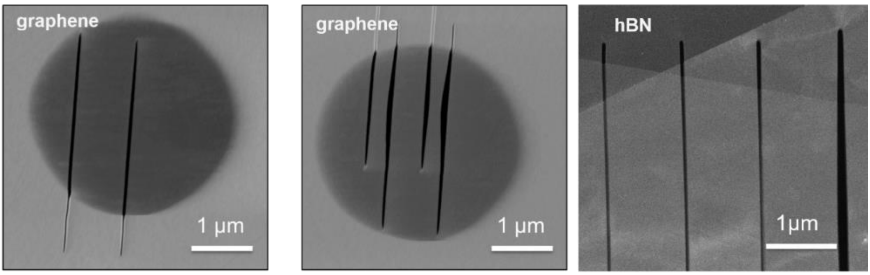

Fig. 1: SEM images of an example graphene flake and hBN flake after He ion beam patterning with different doses.

High throughput tips manufacturing for active piezocantilevers with xenon ion beam with mass control

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Microcantilevers equipped with orthogonal tips are basic tool in many scanning probe microscopy (SPM) techniques, especially in atomic force microscopy (AFM). Tips are medium for transferring interactions between surface and appropriate transducers, their durability determines life span of a tool. However, it is possible to preserve the tool by regeneration of the tip. While original tips are manufactured with use of mass production methods, regenerated tips require unitary approach. Therefore throughput of regeneration method becomes valid parameter.

For regeneration of tips focused ion beam (FIB) family of techniques can be used. It allows both for removing (milling) and building (depositing) the material. With use of nanomanipulators auxiliary materials can be applied [1]. Manufacturing the tip requires time, as required amount of material has got to be moved. To increase the throughput of process inert gas plasma beam can be utilized. Plasma beam offers greater currents than liquid metal sources or gridless ion sources. Furthermore, use of heavy element such as xenon greatly improves processing speed, as Xe ions have higher sputtering yield (2.3 atom/ion on silicon on normal incidence) than e.g gallium (2.05) and also lower penetration depth (mean 11,1 nm versus 12.2 nm for gallium) [2].

To get a hold on rapid process, external measuring circuit is applied. Active cantilever is actuated at the resonant frequency and it can be viewed as a simple harmonic oscillator (SHO). Material deposition delivers mass to the SHO, therefore reducing its resonant frequency; material milling during process raises resonance. Vibrations are observed with help of amplifying circuit on the oscilloscope, where in situ assessment of process' result is possible with measurements of mass

In this setup conductive diamond tips were delivered onto active piezoresistive cantilevers. We present used setup with results in form of sharpened tips.

[1] E. Gacka, P. Kunicki, A. Sikora, R. Bogdanowicz, M. Ficek, T. Gotszalk, I. Rangelow, K. Kwoka; *Focused ion beam-based microfabrication of boron-doped diamond single-crystal tip cantilevers for electrical and mechanical scanning probe microscopy*; Measurement (**2022**), 1

[2] R.P. Webb, I.H. Wilson; *Problems using the Sigmund formula for the calculation of sputtering yields*; Vacuum (1989), 1163

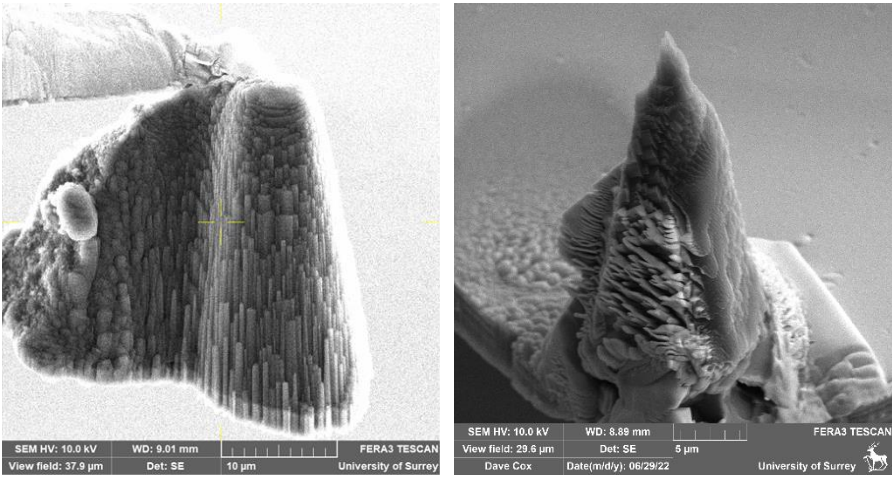

Fig. 1: Diamond tip before mounting, carried with nanomanipulator (left) then mounted and sharpened (right)

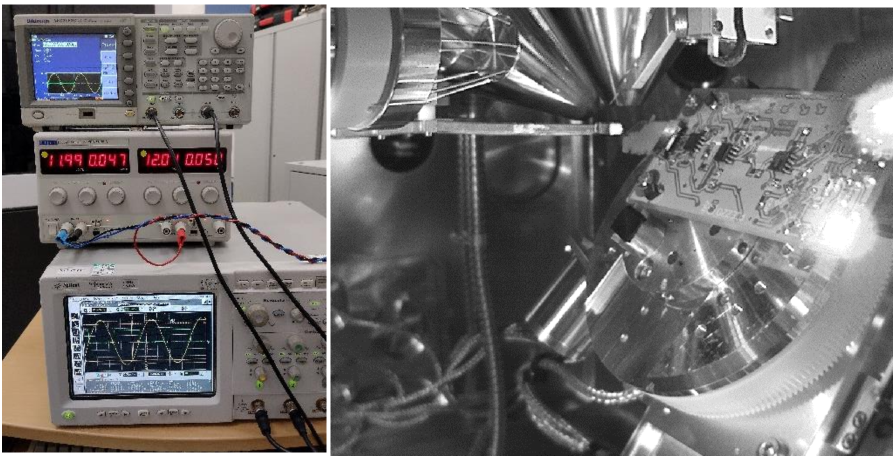

Fig. 2: Tip placement control setup consisting of: power source, function generator, oscilloscope outside chamber (left) and circuit board of amplifiers inside the chamber (right)

Crystalline anisotropic curtaining effect in Bismuth

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Elemental bismuth is a semi-metallic material with inherently strong spin-orbit coupling and high carrier mobility. These unique electronic properties makes it highly important for both fundamental researches and future applications[1]. Motivated by the recent development on higher-order topological insulator phase in Bismuth[2], it is desirably needed to explore the possibility of artificially writing topological conduction channel using focused-ion beam (FIB) technique. However, due to its low crystallization temperature, the local heating by ion beam assists the growth of Bismuth nanowires along the beam direction and therefore leads to strong curtaining effect on the polished surface, which is a major challenge for microstructure fabrication. On the other hand, we found that this curtaining effect is highly anisotropic due to the preferential growth of bismuth nanowires along different crystalline directions. By changing the ion beam direction from trigonal to bisectrix direction, the curtaining effect is strongly suppressed and therefore a clean lamella can be obtained. This sets the basis for the future development of FIB-printed topological microcircuits. Moreover, the nanowire growth along bisectrix direction sheds the light on an interesting method to develop integrated bismuth nanowire array, the pattern of which can be exactly controlled by the ion beam condition.

[1] Asish K. Kundu et al., *Quantum size effects, multiple Dirac cones and edge states in ultrathin Bi(110) films*; arXiv: 2106.13943 **(2021)**.

[2] Frank Schindler, Zhijun Wang et al., *Higher-Order Topology in Bismuth*; Nature Physics 14 **(2018)**, 918.

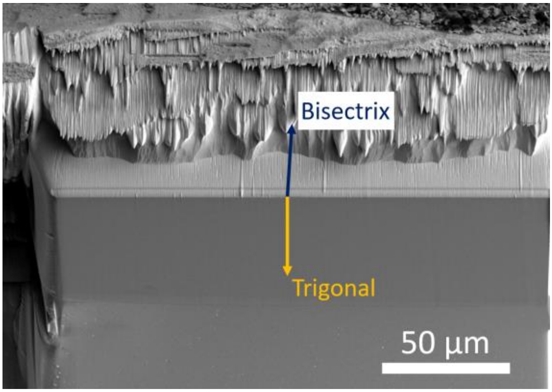

Fig. 1: Crystalline anisotropic curtaining effect in Bismuth.

Micromachined samples for uniaxial strain studies with laser-ARPES

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Uniaxial strain is an important tuning parameter in condensed matter physics, as modest pressures can induce fundamentally different characteristics in materials reversibly and reproducibly [1-3]. However, it has long been a technical challenge to systematically study the effects of uniaxial strain in experiments such as angle resolved photoemission spectroscopy (ARPES) [4-6].

We introduce a novel and generally applicable route to studying uniaxial strain by macroscopically shaping a sample with a Helios G4 PFIB such that it gains a tapered profile. This profile allows us to induce a variation in strain within the sample by applying pressure with a thermally actuated pressure cell. The induced strain gradient can then be resolved in experiments with a spatially local probe, unlocking the potential for systematic studies of the effect of uniaxial strain. We present micro-focused laser-ARPES results for Sr_2RuO_4 – a keystone material in condensed matter physics – under uniaxial strain to study the evolution of a van Hove singularity across the chemical potential.

[1] C. Lin *et al.; Visualization of the strain-induced topological phase transition in a quasi-one-dimensional superconductor* $TaSe_3$; Nature Materials **20** (2021), 1093.

[2] H. Kim *et al.; Uniaxial pressure control of competing orders in a high-temperature superconductor;* Science **362** *6418* (2018), 1040.

[3] A. Steppke *et al.; Strong peak in Tc of Sr2RuO4 under uniaxial pressure;* Science **355** *6321* (2017) eaaf9398.

[4] J. A. Sobata, Y. He, Z. X. Shen; *Angle-resolved Photoemission studies of Quantum Materials;* Reviews of Modern Physics **93** (2021), 025006.

[5] S. Ricco *et al.; In situ strain tuning of the metal-insulator-transition of Ca2RuO4 in angle-resolved photoemission experiment;* Nature Communications **9** (2018), 4535.

[6] V. Sunko *et al.; Direct observation of a uniaxial stress-driven Lifshitz transition in Sr2RuO4;* npj Quantum Materials **4** (2019) 46.

In-situ sample preparation of oxidizing and contaminating samples for high quality EDS and WDS quantification using FIB-SEM

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Energy dispersive and wave dispersive X-ray spectroscopy (EDS and WDS) are very important tools in materials research to obtain information about the chemical composition of a sample. Many materials like thermoelectrics show a significant loss of efficiency with elemental compositions slightly differing by a few percent. In quantitative measurements conductivity, contamination and, above all, homogeneity and the geometry of the sample have a significant influence on the quantification results with EDS or WDS systems. Especially for WDS the quality of the sample surface is very important to achieve an accuracy below 1%. Many materials are susceptible to oxidation (Fig. 1, right) or carbon contamination on their surface decreasing the accuracy and quality of WDS and EDS measurements. Furthermore, this prevents the quantification of initial oxygen or carbon contents.

The use of FIB allows the preparation of smooth cross-sections with very good smoothness and planarity (Fig.1, left). With an EDS/WDS system installed at a FIB-SEM, an in-situ, site specific preparation followed by analysis without leaving high vacuum is feasible. This enables a contamination- and oxidation-free preparation and analysis, significantly improving the quality of the measurement.

Cross-sections prepared with the FIB are usually not perpendicular to the electron beam, which is a pre-requisite for a proper quantitative EDS or WDS analysis. In this study, a new procedure is presented allowing an accurate quantification with EDS and WDS by using different tilt angles for the preparation and analysis step.

Initially, the cross-section of a thermoelectric sample is prepared at a tilt of -10° producing a flat angle between sample and Ion beam on a system including an angle of 52° between electron and ion column. By tilting the sample to +28° afterwards, the cross-section is orientated perpendicular to the electron-beam enabling a quantification with EDS and WDS (Fig: 2). The results sum up to a total weight of 100.23% indicating a reliable combined EDS/WDS measurement (Tab. 1, left). For the sample surface produced via metallographic state-of-the-art procedures, which is oxidized due to atmospheric exposure, a weight of 92.23% is measured indicating a non accurate measurement caused by the high oxygen contamination layer on the surface (Tab. 2, right). This comparison indicates that this procedure significantly improves the quality of EDS and WDS quantitative compositional results for fast oxidizing and easy contaminating samples.

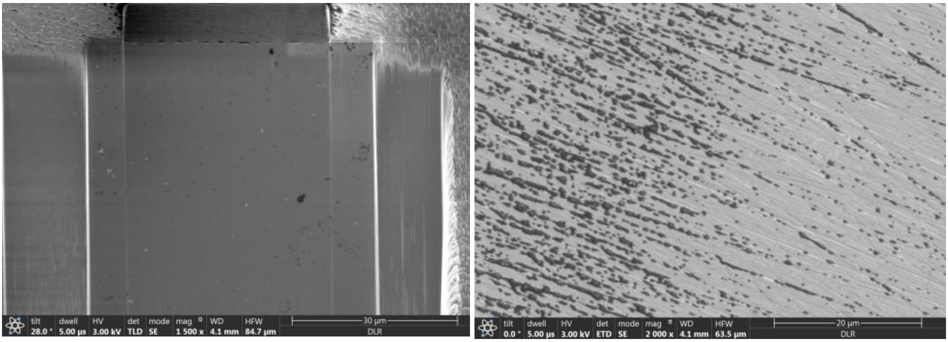

Fig. 2: Left: Surface of FIB prepared Cross-Section in a flat angle. Right: Oxidized surface of the sample

	FIB Prepared		Metallographic Preparation	
Element	Weight [%]	Atom [%]	Weight [%]	Atom [%]
Mg+	8.82	31.28	8.74	33.14
Ag	40.74	32.65	37.53	32.06
Sb	50.67	35.97	45.96	34.79
Total	100.23	100.00	92.23	100.00

Tab. 1: Left: Quantification with WDS of MgAgSb in a FIB prepared cross-section with corrected tilt. Right: Quantification with WDS of the same sample, but on its surface prepared by conventional metallography procedures. Mg was measured by WDS, AG and Sb by EDS.

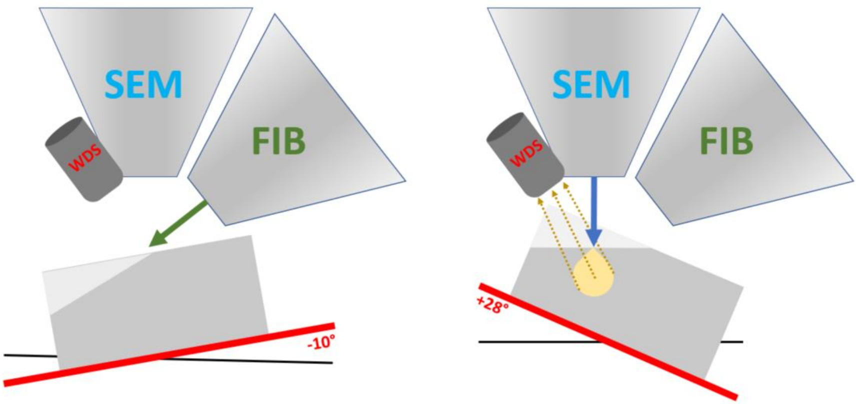

Fig. 2: Left: Cross-Section with -10° stage tilt resulting in a -28° tilt compared to the sample surface; Right: Stage tilt of +28° to achieve the geometry necessary for WDS quantification.

Dual focused ion beam nanofabrication of V-grooves in monocrystalline gold for efficient excitation of organic single photon emitters

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Single dibenzoterrylene (DBT) molecules in anthracene nanocrystals have been shown to emit single photons without blinking, bleaching or spectral diffusion. Efficient integration of these molecules into photonic circuits will pave the way toward the realization of quantum optical networks. We incorporated anthracene nanocrystals containing single DBT molecules into plasmonic V-grooves milled with a focused ion beam in monocrystalline gold flakes. The fabricated V-grooves exhibit high-quality low-loss guiding of channel plasmon polaritons with the propagation length reaching ~14 µm at a wavelength of 800 nm. For DBT molecules coupled to the V-grooves, we observe enhanced emission decay rates with up to 50% of the emission being funneled into channel plasmon polaritons [1].

In particular we utilized a two-step focused ion beam lithography process to fabricate V-grooves with defined dimensions, smooth sidewalls and inclined end-faces on monocrystalline gold flakes. The subsequent application of gallium and helium focused ion beams (GaFIB/HeFIB) provided by a commercial instrument (Zeiss Orion Nanofab) allowed us to fabricate waveguides of the required size and shape at reasonable fabrication speed.

[1] Kumar, S.; Leissner, T.; Boroviks, S.; Andersen, S. K. H.; Fiutowski, J.; Rubahn, H. G.; Mortensen, N. A.; Bozhevolnyi, S. I. Efficient Coupling of Single Organic Molecules to Channel Plasmon Polaritons Supported by V-Grooves in Monocrystalline Gold. Acs Photonics 2020, 7 (8), 2211-2218.

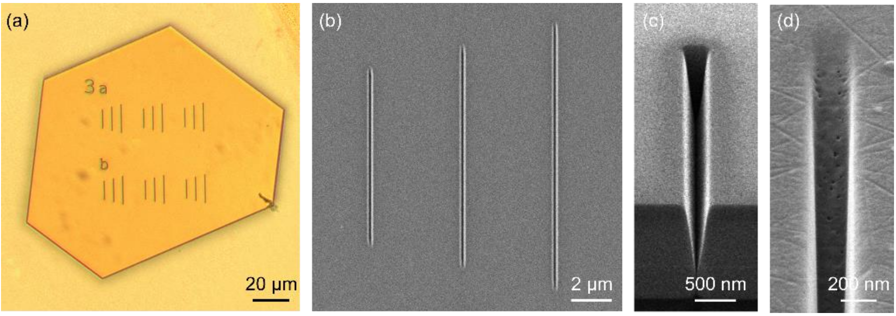

Fig. 1: (a) Microscope image of a gold flake containing V-grooves of different sizes. (b) HIM image of V-grooves. (c) HIM image of a cross-section of a V-groove together with a nano mirror. (d) HIM image of the nano-mirror at one of V-groove ends.

Novel FIB nanofabrication strategies facilitated by light and heavy ions from GaBiLi Liquid Metal Alloy Ion Sources

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Focused Ion Beam (FIB) direct nano-patterning has become established as versatile, and precise fabrication method of smallest features at the nanoscale [1]. Applications in nanoscale science are manifold and require high resolution fabrication techniques at high fidelity, accuracy, and reproducibility. As a result, high demands concerning sputter yield, beam stability and patterning resolution are made on the ion beam for direct FIB nano-patterning. Liquid Metal Alloy Ion Source (LMAIS) is an emerging FIB source technology that provides a versatile solution to deliver light or heavy and fast or slow ions from a single source for FIB nanofabrication [2]. We present unique direct nano-patterning results and a novel workflow using GaBiLi LMAIS. This workflow allows taking advantage of the benefits of the different beams. Gallium, Bismuth and Lithium ions are emitted simultaneously with subsequent ion separation in an ExB filter.
A Lithium-ion beam has the smallest beam diameter and enables highest patterning resolution as well as imaging resolution of all ions available from LMAIS [3] whereas Bismuth ion beam provides higher sputter yield at higher depth resolution [4].
To overcome challenges during patterning of sub-10nm metallic nanogaps, we present a 2-step fabrication process for bowtie nano-antennas. This approach takes advantage of large volume material milling with a Bismuth ion beam at high sputter yield to speed up the entire fabrication process and subsequent lateral fine shaping using a Lithium beam from the same ion source.
Beyond direct nanopatterning, Lithium as the lightest ion available from LMAIS provides excellent ion beam imaging capabilities. Latest results of 3D milling and ion imaging with Bismuth and Lithium ions for 3D sample tomography and reconstruction will be presented.

[1] P. Li, S. Chen, W. Peng, W. Shan, and H. Duan, Recent advances in focused ion beam nanofabrication for nanostructures and devices: fundamentals and applications, Nanoscale 13, 1529 (2021).
[2] J. Gierak, P. Mazarov, L. Bruchhaus, R. Jede, L. Bischoff, Review of electrohydrodynamical ion sources and their applications to focused ion beam technology, JVSTB 36, 06J101 (2018).
[3] N. Klingner, G. Hlawacek, P. Mazarov, W. Pilz, F. Meyer, and L. Bischoff, Imaging and milling resolution of light ion beams from helium ion microscopy and FIBs driven by liquid metal alloy ion sources, Beilstein J. Nanotechnol. 11, 1742 (2020).
[4] A. Tolstogouzov, P. Mazarov, A.E. Ieshkin, S.F. Belykh, N.G. Korobeishchikov, V.O. Pelenovich, D.J. Fu, Sputtering of silicon by atomic and cluster bismuth ions: An influence of projectile nuclearity and specific kinetic energy on the sputter yield Vacuum 188 110188 (2021).

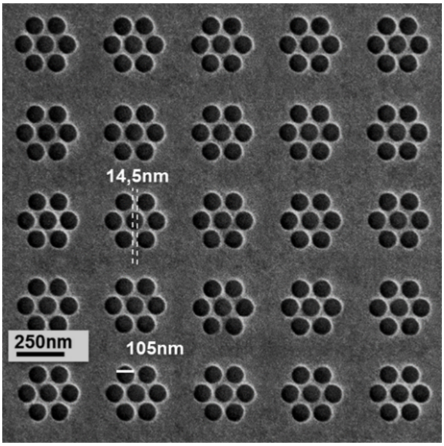

Fig. 1: *Li FIB patterning of heptamer-arranged nanohole (HNH) arrays in 50nm Au film on SiO_2 and subsequent Li ion beam imaging*.

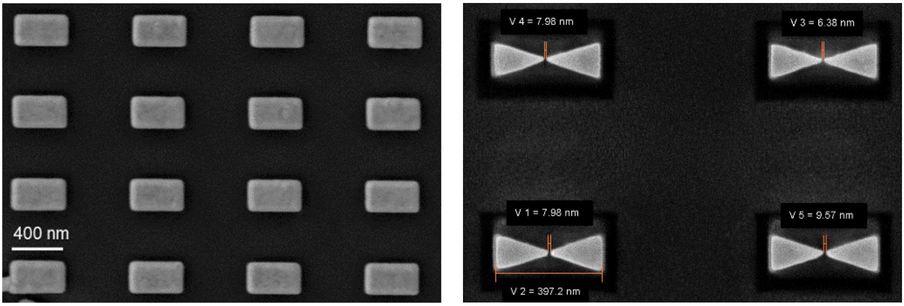

Fig. 2: *Stepwise fabrication of nano bowties in 50nm Au film on Si: a) Bi FIB for large volume milling to excavate rectangular boxes, b) Li FIB for fine shaping bowties structures at highest resolution*.

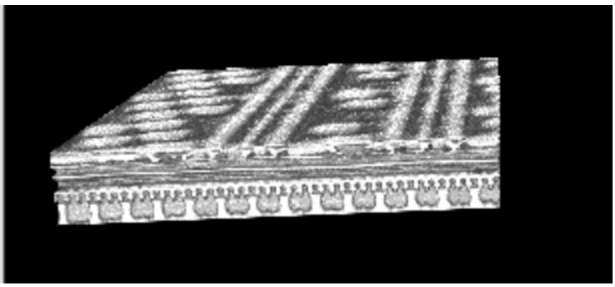

Fig. 3: *3D reconstruction (5µm x 5µm x 0.85µm) of semiconductor chip layers by Bi milling and Li imaging.*

Effect of Focused Helium-ion Beam on Surface Morphology of Polypropylene Thin-films for Power Capacitors

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Polypropylene (PP) films are one of the widely used dielectric materials for power capacitors in high-frequency and high-power applications due to their excellent dielectric strength, ease of processability, reliability, and low dissipation factor [1]. The rapid development of high-performance capacitors requires new methods of modification and advanced characterization of the PP films at the nanoscale level to enable their further miniaturization and to improve their performance. In this work, we investigated the effect of ion-beam irradiation on the surface morphology of PP films. Commercially available and deposited with spin-coating PP films were irradiated with a focused He^{+}-ion beam (He-FIB) in a Zeiss Orion NanoFab Helium Ion Microscope at landing energy of 25 keV with doses in a range of 5.4×10^{-5} nC/μm^2 to 8.07×10^{-3} nC/μm^2. Prior to irradiation, all samples were metalized with a very thin layer of a metal alloy. He-FIB was used to construct surface patterns similar to those fabricated in our previous studies [2,3] of polymer materials irradiated with FIBs. Atomic force microscopy (AFM) and optical microscopy were used to analyze the details of surface modification. The obtained results show that the irradiation of the PP films with He-FIB results in shrinkage of the polymer material and negligible surface sputtering effects. Surface ripples were observed at the borders between the irradiated and non-irradiated regions, which is attributed to the mechanical strain induced by the material modification. Among other things, our study shows that, alongside the known modification of dielectric and electric properties of PP material, the changes in the film shape in the irradiated regions should be taken into account as a geometrical factor that affects capacitance.

References:

[1] J-W. Zha, M-S. Zheng, B-H. Fan, Z-M. Dangd; *Polymer-based dielectrics with high permittivity for electric energy storage: A review;* Nano Energy, 89 (2021).

[2] S. Chiriaev, L. Tavares, V. Adashkevich, A. J. Goszczak, H-G. Rubahn; A. Author, B. Author; *Out-of-plane surface patterning by subsurface processing of polymer substrates with focused ion beams;* Beilstein journals of nanotechnology, 11 (**2020**), 1693-1703.

[3] L.Tavares, S.Chiriaev, V.Adashkevich, R.Taboryski, H-G.Rubahn; *Height patterning of nanostructured surfaces with a focused helium ion beam: a precise and gentle non-sputtering method*; Nanotechnology, 31(2020),145303

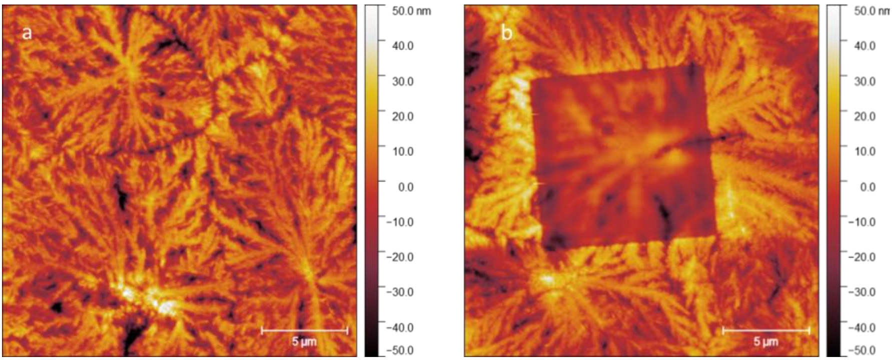

Figure 1: AFM images of the surface of spin-coated PP film, before (a) and after (b) FIB-He irradiation. The irradiated region is the dark square in b).

Fault Localization in FIB/SEM – combining delayering and EBAC into a cohesive workflow

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Fault localization and fault isolation are important, recurring tasks in many Fault Analysis laboratories. The workflow involves various techniques for deprocessing and delayering the sample including CNC milling, various polishing methods, and lately, with the advent of smaller and smaller technology nodes, PFIB delayering. The latter method yields precise control over the number of layers removed while allowing the operator to open large windows into the sample.

Once the target layer is exposed, electrical testing methods such as EBIC (Electron Beam Induced Current) or EBAC (Electron Beam Absorbed Current) as well as nanoprobing experiments for transistor characterization can be performed. Depending on the obtained results, further delayering may be required. Or, if the fault is located, a TEM slice may be prepared in order to further elucidate the fault's root cause.

Thus, it is advantageous to combine the electrical characterization measurement and the PFIB delayering into a single tool – thereby circumventing the need to move the sample back and forth between tools. This minimizes the sample's exposure to air (which minimizes contamination on the sample) and significantly speeds of the time to result. The images in Fig. 1 and Fig. 2 show the proposed setup as well as a micrograph of a typical sample with probes in place for EBAC analysis.

In this work, the proposed tool configuration for performing the combined workflow described above will be presented.

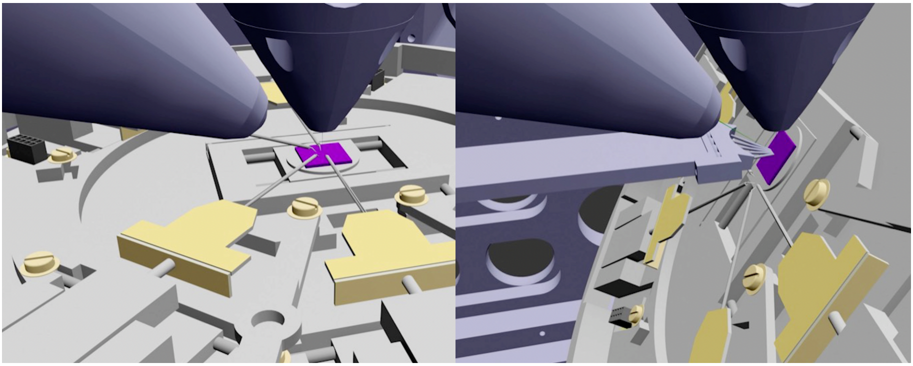

Fig. 1: EBAC configuration (left) and delayering configuration (right) with the stage tilted to FIB angle and the GIS inserted. Note that the sample was shifted using the integrated substage such that the distance between the probe tips and the FIB is maximized in order prevent contamination of the probe tips.

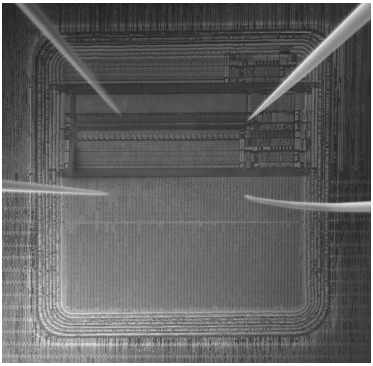

Fig. 2: Four probe tips placed in close vicinity to the sample in preparation for an EBAC analysis (courtesy AMD Singapore)

Second order Zeeman interaction and ferroquadrupolar order in $TmVO_4$

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$TmVO_4$ exhibits ferroquadrupolar order of the Tm 4f electronic orbitals at low temperatures, and is a model system for Ising nematicity. A magnetic field oriented along the *c*-axis constitutes a transverse effective field for the quadrupolar order parameter, continuously tuning the system to a quantum phase transition as the field is increased from zero. In contrast, in-plane magnetic fields couple to the order parameter only at second order, such that orienting along the primary axes of the quadrupole order results in an effective longitudinal field, whereas orienting at 45 degrees results in a second effective transverse field. Nuclear magnetic Resonance (NMR) studies of these effects are hampered by increased linewidth of the spectra due to inhomogeneous demagnetizing fields but can be minimized by cutting the sample to an ellipsoidal shape using a Xe plasma FIB.

Previous NMR measurements of $TmVO_4$ identified a scaling between the spin–lattice relaxation rate and the shear elastic stiffness constant, c_{66}, suggesting that the ^{51}V ($I = 7/2$) nuclear spins couple to the Tm orbitals through the electric field gradient (EFG), giving rise to a quadrupolar relaxation channel [1]. However, the spectra were significantly broadened by inhomogeneous demagnetization fields and the anisotropic *g*-factor of the Tm ground state doublet. In order to better discern the spectra and relaxation mechanisms at play, we reshaped a single crystal of $TmVO_4$ to an ellipsoidal shape, with a homogeneous demagnetization field (see Fig. 1). We utilized a Xe^{2+} plasma focused ion beam (FIB) by Thermo Fisher Scientific with a 30 kV, 1 μA beam to cut our sample with the long-axis along the *c*-axis of the crystal. Sample damage from the beam is only expected on the surface within a depth of 30–40 nm and energy dispersive X-ray analysis (EDX) of a test surface verifies the unchanged composition of $TmVO_4$ below. The final sample diameter is 0.4 mm and the length of 1.3 mm require a total cutting time in excess of 25 h of each side. The magnetic broadening was dramatically reduced in the FIB crystal, such that each of the seven peaks separated by the quadrupolar splitting are clearly resolved. The ability to resolve all seven peaks is important because it enables us to extract details of the magnetic and quadrupolar contributions to the spin–lattice relaxation rate that would otherwise be inaccessible, as discussed above in the section on Spin-lattice relation [2].

[1] Z. Wang, I. Vinograd et al.; *Anisotropic nematic fluctuations above the ferroquadrupolar transition in $TmVO_4$; Phys. Rev. B* **104**, 205137 (**2021)**.

[2] I. Vinograd, K. R. Shirer et al; *Second order Zeeman interaction and ferroquadrupolar order in $TmVO_4$; npj Quantum Materials* **7**, 68 (**2022)**.

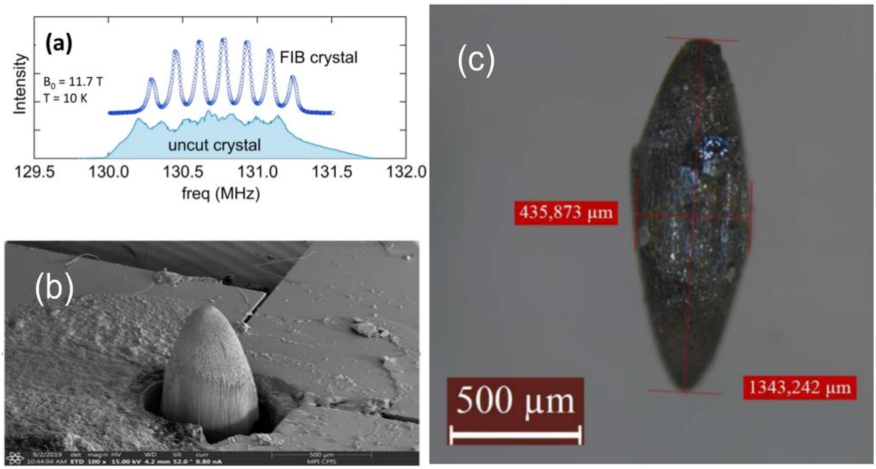

Fig. 1: (a) ^{51}V-NMR spectrum with and without ellipsoidal crystal shape. (b) Scanning electron microscopy (SEM) scan of the sample during the FIB process. (c) Crystal after FIB. Al and C are deposited on the sample surface layer during the FIB processing, but do not contribute to the NMR signal.

Using Electron Beam Curing (EBC) for the Controlled Bending of 3D-Nanoprinted FEBID Structures

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Additive manufacturing via Focused Electron Beam Induced Deposition (FEBID) is an increasingly relevant technique for depositing high-fidelity architectures on the nanoscale. While most such structures in the past were of a meshed nature [1], recent developments towards building closed (sheet-like) elements have opened up the field for a whole new range of possibilities [2]. In a next step we now explored post-growth electron beam curing (EBC) [3], where the structures are locally irradiated without precursor gas present. This process impacts the inner structure and the overall volume of exposed elements and, if only applied partially, enables controlled deformation. We therefore performed experimental series, analyzed via SEM and TEM and complemented by Monte Carlo Simulations to explore and identify ideal parameters for smooth, stable and reproducible morphological bending. Figures 1a and 1b show a vertical wall with a width of 1 µm and a height of 2 µm that was bent via electron beam irradiation within a defined area across the structure. Figure 1c shows a more complex (originally straight) screw element where two areas have been exposed to EBC, clearly illustrating the bending effect towards the incidence direction. We attribute this “forward” bending to smaller interaction volumes of the incoming electrons compared to the wall thickness, mainly influencing the front part of the elements in comparison with the back side. We evaluated the impact for a variety of parameters, such as voltage, point pitch, dwell time, overall dose and beam incidence angle to achieve controlled and reproducible results. The expansion to more complex EBC patterns leads furthermore to more sophisticated bending as will be presented as well (see Fig. 2). We thereby extended the post-growth treatment possibilities of FEBID, showing the flexibility of EBC for various applications in research and development, some of which clearly go beyond the capabilities of sole 3D FEBID (e.g. spatially tuned mechanics).

[1] R. Winkler et al.; 3D nanoprinting via focused electron beams; Journal of Applied Physics 125 (**2019**), 210901.

[2] A. Weitzer et al.; Expanding FEBID-Based 3D-Nanoprinting toward Closed High-Fidelity Nanoarchitectures; ACS Applied Electronic Materials, 4 (2) (**2022**), 744.

[3] F. Porrati et al.; Tuning the electrical conductivity of Pt-containing granular metals by postgrowth electron irradia-tion; Journal of Applied Physics 109 (**2011**), 063715.

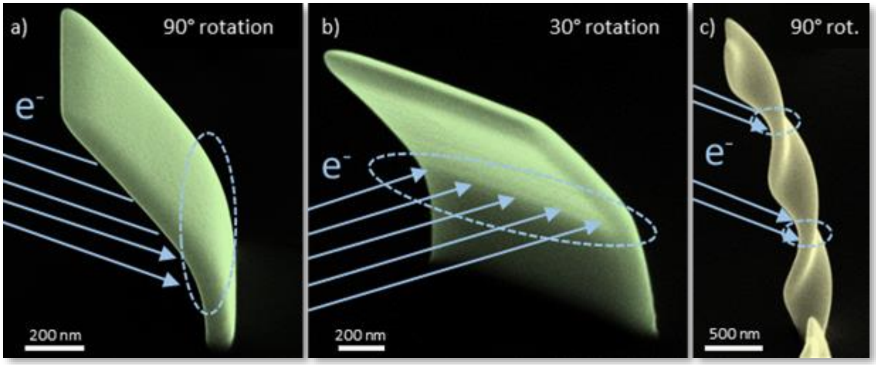

Fig. 1: Bending of sheet-like 3D FEBID elements. Bent wall from a side angle (a) and from a 30° rotated point of view (b) and twofold bent screw structure (c).

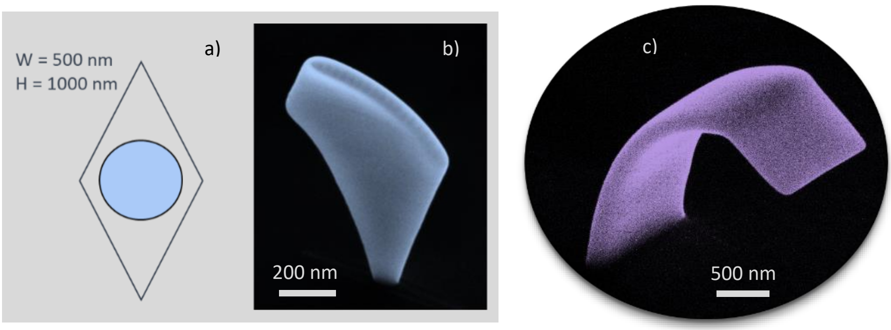

Fig. 2: More advanced electron beam curing with (a) a schematic for the bending of a diamond architecture via a circular curing pattern, (b) the corresponding SEM image of the real structure and (c) a wall element that was bent to an overhang.

High-Precision 3D-Nanoprinting for Sheet-like Structures via FEBID

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Among the few additive-manufacturing techniques capable of creating 3-dimensional objects on the nanoscale, 3D nanoprinting via Focused Electron Beam Induced Deposition (3D-FEBID) is an increasingly relevant technology for building high-fidelity nanostructures. Its capabilities of depositing feature sizes below 20 nm under optimized conditions and below 100 nm on a regular basis and its flexibility both in terms of substrate as well as precursor materials make it a unique technology with many possibilities and yet unexplored applications. While it has been used and developed further for a few years now, most fabricated structures in the past have been meshed [1], meaning a combination of differently oriented, individual nanowires, connected at specific points in 3D space according to the target application. This work leverages 3D-FEBID to the next level by expanding its capabilities from mesh-like towards closed (sheet-like) structures with a high degree of precision. The main challenge and source of most deviations from target shapes is thereby based on local beam heating and its implications on local growth rates. While well-understood in meshed structures, closed objects revealed additional dependencies on the dimensions of built objects and the XY pixel position within the structures. Furthermore, electron trajectories are more complex in closed objects, introducing additional proximity effects. To address these problems, we combined finite-difference simulations with 3D-FEBID experiments and developed a Python-based compensation tool, capable of stabilizing the growth for each XY pixel point in individual patterning planes by pre-determined parameter adjustments (Fig. 1a). The gained insight allowed further expansion, now being applicable for different element widths and -heights, as demonstrated by more advanced structures (Fig. 1b). In a last step we introduced trapezoid and inclined elements into our compensation code (Fig. 2a), which we then combined to a "construction kit" tool that is able to build compound structures (Fig. 2b-d). By that, we crucially expanded FEBID-based 3D nanoprinting by opening up design possibilities for closed and consequently mixed objects for novel applications in various fields of research and development.

[1] R. Winkler et al.; 3D nanoprinting via focused electron beams; Journal of Applied Physics 125 (**2019**), 210901.

[2] A. Weitzer et al.; Expanding FEBID-Based 3D-Nanoprinting toward Closed High-Fidelity Nanoarchitectures; ACS Applied Electronic Materials, 4 (2) (**2022**), 744.

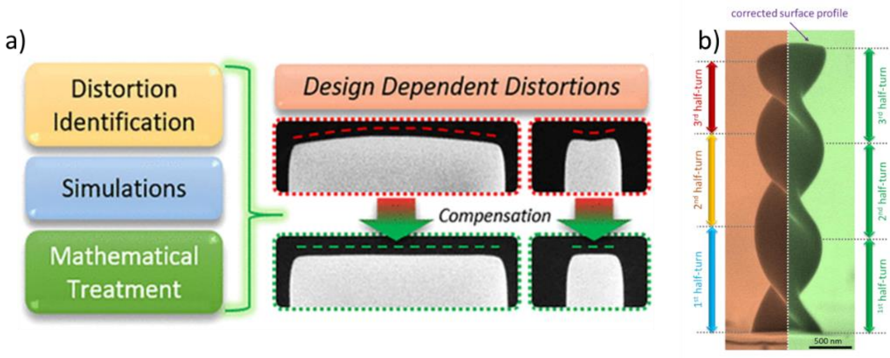

Fig. 1: Improvements due to our compensation tool on the example of (a) walls and (b) a screw [2].

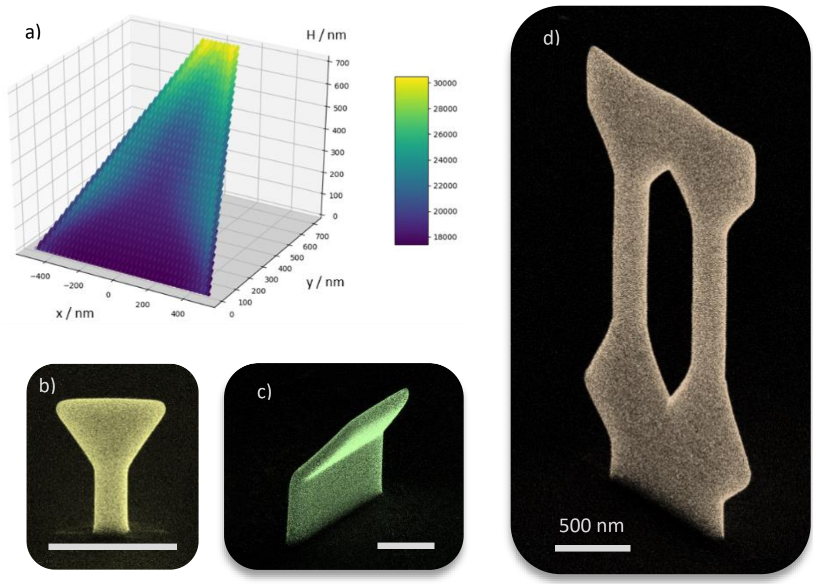

Fig. 2: Python compensation tool extensions for advanced FEBIP structures with (a) an illustration of the dwell time adjustments for a trapezoid structure and (b)-(d) SEM images of compound architectures.

Correlative Microscopy for Aviation and Aerospace

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Correlative microscopy (CM) workflows allow and aid solving a broad range of scientific and industrial problems (e.g. battery research, 3D printing, smart materials, integrated circuits, multi-physics simulation, etc.) previously unreachable by the typical experimental operando. CM workflows involve coordinated in 2D / 3D space and time (temporal 4D imaging) characterization of materials and components across a range of length scales. Various apparatus and imaging modalities contribute to the workflow, for example light, electron/ion microscopy, X-Ray computed tomography (CT), SIMS, EBSD, EDS, WDS, CL, XPS, Raman, STEM and TEM imaging and metrological techniques. These techniques use advanced cross-platform sample holders and dedicated software for automatic or guided coordinate transfer and locking solutions. CM delivers plethora of coregistrated 3D data that is often post-processed by artificial intelligence (AI) algorithms. AI-based segmentation or complex, multi-phase microstructures reduces the time-to-results from months to days or even dozens of hours. Further CM is often followed by an image-based modelling and multi-physics simulations.

As a practical example of CM for aerospace and defense industries we study a thermal barrier coating (TBC) used in the afterburner liner of turboramjet configured JT11D-20 engine (Fig. 1). The CM workflow used in this study correlates 3D data from HeliScan micro CT, femtosecond Laser Plasma FIB-SEM serial sectioning tomography across the length scales required to characterize and understand effects of long-term service on microstructural characteristics of the TBC (Fig. 2). Each system contributes structural (micro CT, Serial Sectioning) and analytical (EBSD, EDS) data directly within its characteristic length scale. The Laser Plasma DualBeam, cross-platform holder kit, Maps and Avizo software also plays a key role in integrating the workflow across the full range of length scales. Atomic scale S/TEM imaging and quantitative analysis is a logical extension of the workflow to the nano scale.

[1] B Winiarski at al., Supplement of Microscopy and Microanalysis 152 (2017), p. S4-S9.
[2] B Winiarski at al., Microscopy and Microanalysis 23 (S1/2017), p. 342-343.
[3] TL Burnett et al., Scientific Reports 4 (2014), p. 4711.
[4] SJ Randolph et al., Journal of Vacuum Science & Technology B36 (2018), p. 06JB01.
[5] MP Echlin and A Mottura et al., Review of Scientific Instruments 83 (2012), p. 023701.
[6] B Winiarski at al., Microscopy and Microanalysis 24 (S1/2018), p. 366-367.

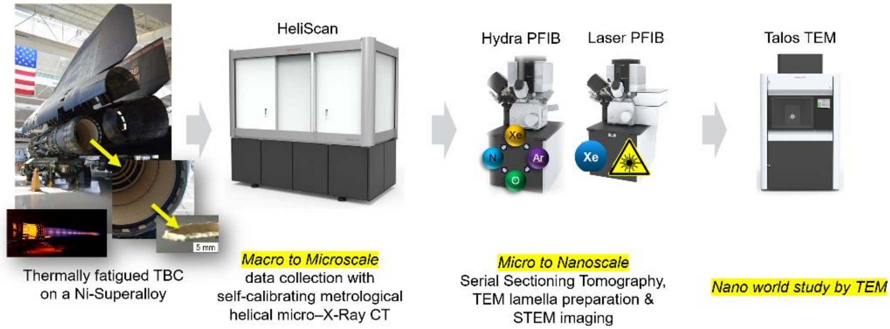
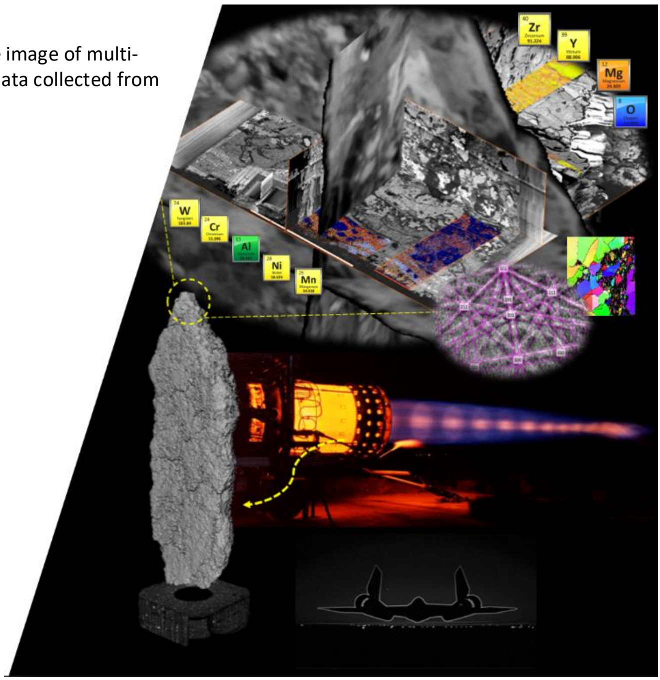


Fig. 1: Shows the experimental workflow.

Fig. 2: Shows composite image of multi-scale and multi-modal data collected from the TBC sample.



Plasma FIB spin milling hastens battery research

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The technology for energy storage systems is changing rapidly. In the past decade, the rapid growth of consumer electronics and electric vehicles market leads Li-ion batteries to attract significant attention. Currently two important industries, the energy and automobile industries, are involved in intensive research in this area where the most research takes place into: (a) solid state batteries [1], (b) lithium-ion batteries [2] and (c) sodium-ion batteries [3]. As the energy revolution gathers pace, batteries will be needed for energy storage in order to equalize the fluctuating power production of photovoltaic solar systems or wind turbines. The trend for electromobility places a lot of pressure to accelerate battery research.

Further advancement of their performance for higher energy and better safety is achieved by fundamental understanding of battery materials structures and chemistry throughout the life cycle. Various complex studies have been carries out in situ, ex situ and using 3D correlative multi-scale tomography and microscopy (CMT) [4]. Although CMT is very powerful methodology to gather 3D insight into material microstructure and chemistry [5, 6], it requires considerable amount of effort if compared to characterization of materials in two dimensions without prior screening with X-ray micro tomography (µCT). In many situations 2D information (SEM, EBSD, EDS, Raman, SIMS) from a cross-section of material give plethora of quantitative and statistical data. Plasma FIB-SEM opened fast access to 100's of microns wide and deep cross-sections allowing collecting multi-modal information with nanometer resolution [6]. Depending on the size of the cross-section, material and type of analyses needed the time-to-results (TTR) can vary from few dozens to hundreds of minutes. Considerable part of TTR is the site preparation time (SPT) for the cross-section: (a) MultiChem gas injection system deposition of a protective layer; (b) milling of the side trenches; (c) milling of the front trench to define of the cross-section and (d) polishing of the cross-section. For example, the SPT time for high resolution electron diffraction pattern analyses (HR-EBSD) over large areas (> 100's of µm2) is about < 25% of the TTR, while the SPT for high resolution SEM imaging is about > 95% of the TTR. The SPTs for other quantitative techniques sits in between the above percentage values of the TTR. In the recent years TTR was reduced by introducing new more sensitive detection systems, e.g. EDS, EBSD. Further, the femto-second Laser PFIB-SEM significantly reduced a cross-section preparation or serial sectioning time [7]. Currently, a new large area (<= 1 mm2) or large volume acquisition technique, so called Plasma FIB spin milling (PFIB-SM), was introduced by Winiarski at al. [8]. This technique collects data from superficial layers of a specimen surface and does requires minimum or no site preparation time.

This work presents for the first time the Plasma FIB spin milling method for applications in the battery research. As an example, a generic NMC cathode from a Li-ion battery cell was spin mill polished, where areas of 500 µm in diameter ware prepare within dozens of minutes. The PFIB-SM allows collecting curtain-free data from on-axis and off-axis location, thus accessing areas of about 50 mm2. This technique is well suited for rapid surface polishing and collecting multimodal information, while reaching an inert gas transfer condition is possible.

[1] J Nanda, C Wang, et al. MRS Bulletin 43(10):740-745.

[2] G Blomgren. Journal of The Electrochemical Society 164(1):A5019-A5025.

[3] M Liu, P Zhang at al. Nature Communications, (2019) 10:3917.

[4] R Moroni, M Börner at al. Scientific Reports 2016; 6: 30109.

[5] B Winiarski, G Pyka, at al. Microscopy and Microanalysis, Nano Supp, Dec 2017.

[6] B Winiarski, G Pyka, at al. Microscopy and Microanalysis 24(S1) 2018:366-367.

[7] SJ Randolph et al., Journal of Vacuum Science & Technology B36 (2018), p. 06JB01.

[8] B Winiarski, C Rue, at al. Microscopy and Microanalysis 25(S2) 2019:350-351.

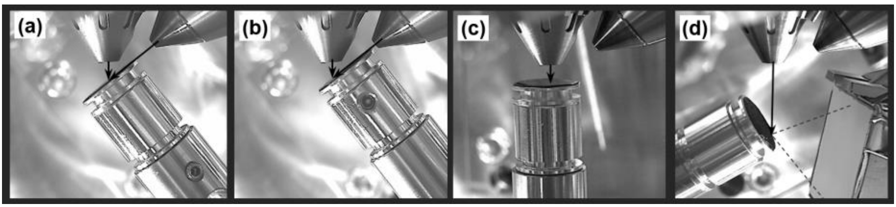

Fig. 1: Shows the experimental setup. Hydra PFIB spin milling (a) on axis and (b) off-axis; (c) SEM/EDS data collection and (d) EBSD data collection geometries.

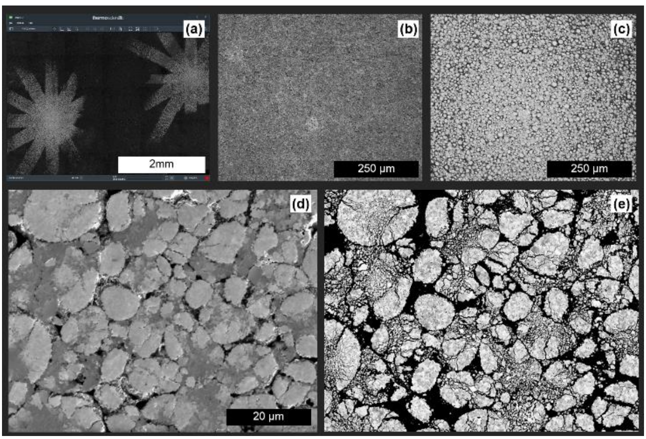

Fig. 2: Shows Xe PFIB spin milling results. (a) data is collected in a Maps project; the cathode before (b) and after spin milling (c) @ 30 kV, 60 nA, 1° glancing angle, < 60 min; (d) SEM ETD-SE 2kV, 0.8nA; (e) SEM CBS 5 kV, 1.6 nA

Plasma FIB-SEM-based Kintsugi Imaging for Battery Electrodes

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The mesostructure of porous electrodes used in lithium-ion batteries strongly influences cell performance [1]. Accurate imaging of the distribution of phases in these electrodes would allow this relationship to be better understood through simulation [2]. However, imaging the nanoscale features in these components is challenging. While scanning electron microscopy is able to achieve the required resolution, it has well established difficulties imaging porous media. This is because the flat imaging planes prepared using focused ion beam milling will intersect with the pores, which makes the images hard to interpret as the inside walls of the pores are observed. It is common to infiltrate porous media with resin prior to imaging to help resolve this issue [3], but both the nanoscale porosity and the chemical similarity of the resins to the battery materials undermine the utility of this approach for most electrodes.

In this study, a technique is demonstrated which uses *in situ* infiltration of platinum to fill the pores and thus enhance their contrast during imaging (Fig. 1). Reminiscent of the Japanese art of repairing cracked ceramics with precious metals, this technique is referred to as the *kintsugi* method. The images resulting from applying this technique to a conventional porous cathode are presented and then segmented using a multi-channel convolutional method [4] (Fig. 2). We show that while some cracks in active material particles were filled with the carbon binder phase, others remained empty, which will have implications for the rate performance of the cell. Energy dispersive X-ray spectroscopy was used to validate the distribution of phases resulting from image analysis (Fig. 2), which also suggested a graded distribution of the binder relative to the carbon additive. The equipment required to use the kintsugi method is commonly available in major research facilities and so we hope that this method will be rapidly adopted to improve the imaging of electrode materials and porous media in general.

[1] T-T Nguyen et al. Npj Computational Materials 2020, 6(1), p. 1-12.
[2] ME Ferraro at al. J Electrochem Soc 2020, 167(1), p. 013543
[3] M Biton, at al. J Electrochem Soc 2017;164(1):A6032-A6038.
[4] I Arganda-Carreras, Kaynig et al. Bioinformatics 2017;33(15):2424-2426.

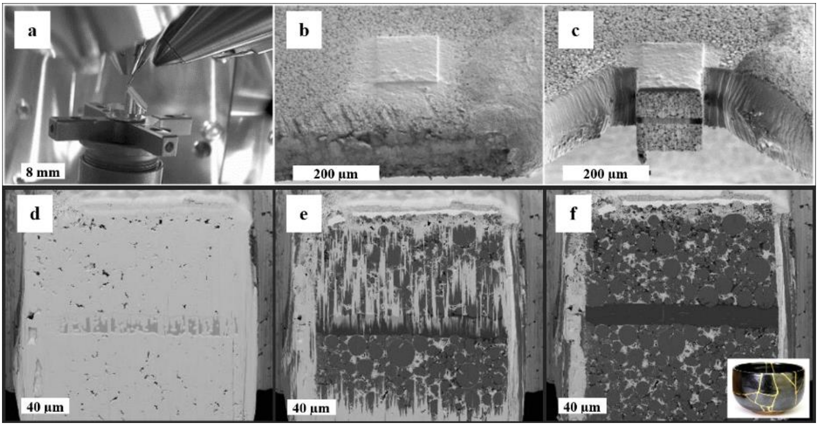

Fig. 1: The cross-section preparation workflow using Helios Hydra PFIB-SEM. (a) Image of the sample arrangement relative inside the vacuum chamber. (b) deposition of the Pt-C mix protective layer on the top surface of cathode. (c) the material block and block face preparation. (d) Pt layer deposition on the block face; (e) the automated ASV run, removing the excess of Pt layer. (f) image of the kintsugi infiltrated block face at 54 nm resolution (Inset shows an example of the pot repaired using the traditional kintsugi method). The scalebar in (a) is 8 mm; in (b, c) is 200 µm; and in (d-f) is 40 µm.

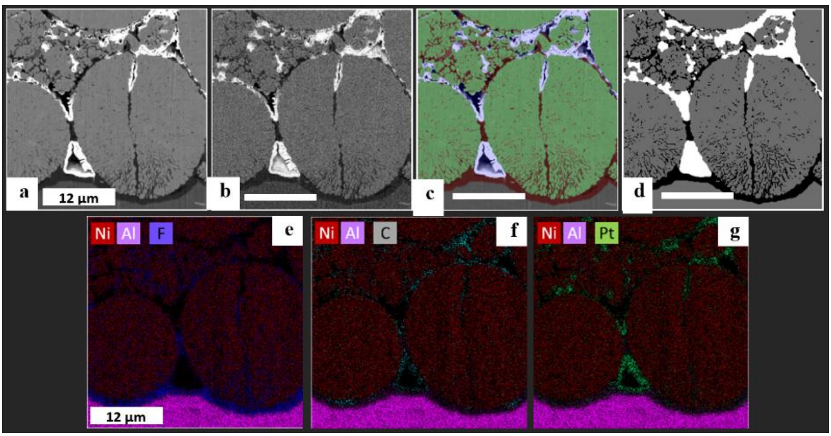

Fig. 2: Images of cathode cross-section prepared using kintsugi method. (a) SEM-BSE images using CBS detector. (b) SEM-BSE images using TLD. (c) CBS image overlaid with segmentation false colouring, where green is active material, red is CBD, and blue is pore. (d) Segmentation where white is pore, black is binder and grey is either active material or current collector. (e-g) Composite of EDS images highlighting the distributions of Al (current collector), Ni (AM particles), Pt (pores), and C (CBD), and the presence of F (polyvinylidene fluoride binder).

The FIB as 3D Nanoprinter – Overview of the Activities in Graz

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Focused particle beam microscopes are versatile tools as they allow nanofabrication in subtractive (ion beam milling, chemically assisted etching via ions or electrons) as well as in additive ways. For the latter, precursor gases are introduced by a gas injection system and locally immobilized by the focused particle beams. Those techniques, called Focused Electron / Ion Beam Induced Deposition (FEBID/FIBID), allows the deposition of nanoscale objects from different precursor materials even on extreme surface topographies and complement situations were alternative nanofabrication technologies run into their limitations (e.g. resist based lithography). Beyond planar and bulky objects, 3D printing of complex architectures becomes possible by a thoughtful control of the particle beam, leveraging both techniques into the status of a real 3D-nanoprinter [1].

In this contribution we highlight the research activities at the FELMI-ZFE in Graz (Austria), which are centered around 3D nanoprinting via FEBID. We line out actual challenges of this emerging technology on three frontiers (Fig. 1): (1) materials, (2) structure/geometries and (3) applications. For each area, we demonstrate possibilities, recent progress and remaining challenges. Furthermore, we show several applications, where such 3D-FEBID structures are used. A dedicated section presents the activities of the Christian Doppler Laboratory that focus on the direct-write fabrication of nanoprobes via 3D-FEBID with an industrial relevance. Here, we modify the tip region of Atomic Force Microscopy (AFM) cantilever with FEBID deposits [2] to enable advanced AFM modes such as conductive AFM, magnetic force microscopy or scanning thermal microscopy [3] (Fig. 2).

[1] R. Winkler et al.; *3D nanoprinting via focused electron beams.* J. Appl. Phys. (**2019**), 125, 210901

[2] H. Plank et al.; *Focused Electron Beam-Based 3D Nanoprinting for Scanning Probe Microscopy: A Review*. Micromachines (**2019**), 11, 48.

[3] J. Sattelkow et al.; *Three-Dimensional Nanothermistors for Thermal Probing*. ACS Appl. Mater. Interfaces (**2019**), 11, 22655

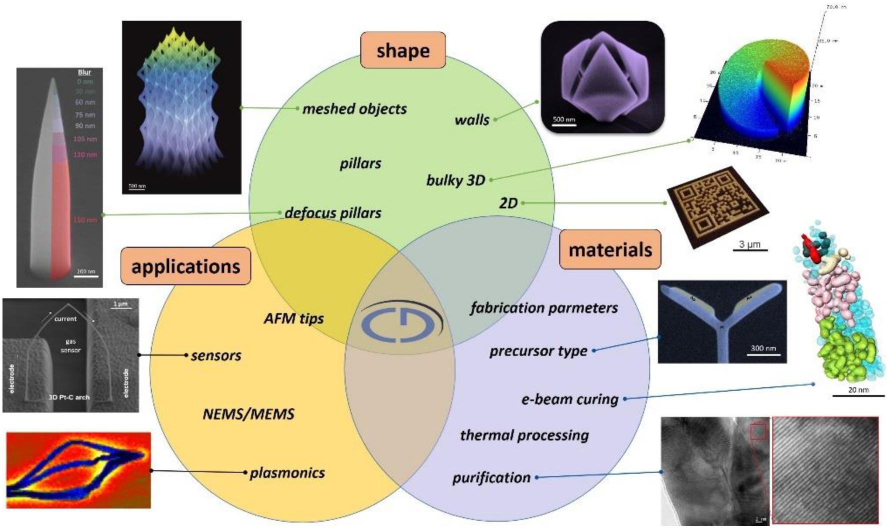

Fig. 1: Actual research areas on 3D-nanoprinting via Focused Electron Beam Induced Deposition addressed at the TU Graz, with a focus on direct-write of AFM nanoprobes.

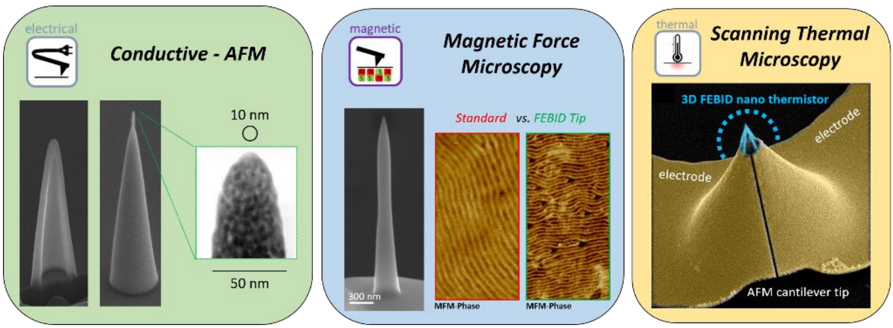

Fig. 2: Special 3D-FEBID nanoprobes for advanced AFM measurements (C-AFM, MFM, SThM), developed in the Christian Doppler Laboratory in cooperation with GETec Microscopy and Quantum Design Microscopy.