ID13: A Micro/Nano-focus Beamline diffraction for Advanced Scanning Analysis.

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The ID13 beamline at the European Synchrotron Radiation Facility (ESRF) is a state-of-the-art scattering SAXS/WAXS facility providing high spatial resolution diffraction fast scans (up to 500 Hz). With its microfocus (≈2×2 µm beam) and nanofocus (≈100×100 nm beam) branches, ID13 provides monochromatic x-ray beams (from 12.7 keV to 17 keV) and excels in exploring large parts of reciprocal space with high sensitivity and low background noise. In addition to scattering, the beamline can cover other techniques such as X-ray fluorescence (XRF), Bragg ptychography and provides various sample environments such as humidity control cell, nanocalorimetry, microfluidics and low temperature environments. To enable fast scans in all directions, the microfocus stage is equipped with a translation stage with a minimum step size of 0.5 µm. The nanofocus branch is equipped with a hexapod mounted on a rotary stage for sample orientation and a piezo translation stage with a 20 nm minimum step size for fast scanning. Both setups can be extended by adding a goniometer that allows the sample to rotate around 2 other axes for advanced 3D data acquisition modes such as texture tomography.

This specific environment with ID13 has been used for significant research, including studies on biological fibres and tissues (Liu et al. 2024), polymers (Rosenthal et al. 2017),cultural heritage (Ghirardello et al. 2022), and materials for energy (Flatscher et al. 2024). For example, the ability to obtain a SAXS/WAXS signal with high sensitivity and fast scans allows the study of biological samples and, in particular, tensor tomography of bones, which requires a large number of scattering patterns and a short exposure time. Thanks to the capabilities of ID13, major steps have been made in the field of texture tomography, with mapping the 3D orientation of nanocrystals and nanostructures in human bone, revealing new structural features (Grünewald et al. 2020). These studies are evidence of the beamline's impact on the advancement of scientific knowledge in a wide range of fields.

To go further, future improvements for ID13 include planned upgrades to equipment and capabilities to reach higher energies up to 20 keV with a new undulator (with potential to explore energies up to 35 keV), an online microscope, and new online analysis techniques such as automated integration and machine learning data processing. These enhancements will help further consolidating ID13's contribution to tomography based and texture analysis.

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« Multi-scale characterization of carbon fiber composites using Small Angle X-Ray Tensor Tomography»

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Carbon fiber composites are high-performance hierarchical materials for sustainable energy, transportation, and security applications. Their physical properties are directly related to the material's hierarchical structure which is influenced by their processing parameters. Thus, understanding the hierarchical structure and its correlation with physical properties would further enable the processing optimization of the materials targeting desirable applications. This project employs synchrotron scattering X-ray techniques to delve into the material's structure ranging from the nano- to the macro-scale. Small-angle X-ray Scattering Tensor Tomography (SASTT) is employed to reconstruct the tensor in each volume unit (voxel) to represent the reciprocal space map and retrieve 3D structural and orientational information of the materials [1,2]. Thus, SASTT is ideal for 3D analysis of the fiber, yarn, and composite inner structure, such as the density of carbon-based fibers, porosity, defects, alignment, and polymer matrix interfaces. As a first step, three composite samples manufactured under different processing conditions were characterized at the cSAXS beamline at the Swiss Light Source. The reconstruction obtained with the Mumott software package [2,3] results in a shell of the 3D reciprocal space map (Figure 1 left), from which the degree of orientation and the main orientation of the scatterers along the measured composite volume can be extracted (Figure 1 right).

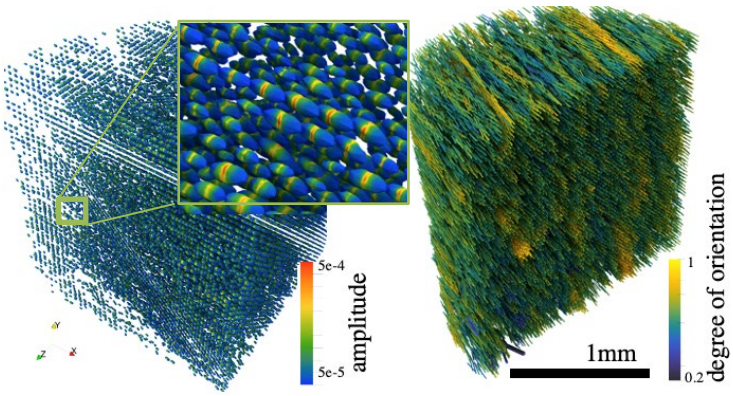

Figure 1. (Left) shows the SASTT reconstruction from the carbon fiber composite where each voxel contains a shell of the 3D reciprocal space map for a q-range of 0.0028 – 0.0035 nm^{-1}, from which the direction and the degree of orientation of the filaments can be determined with cylinders representing the direction and the degree of orientation represented with the color bar (Right).

This project aims to obtain a detailed statistical analysis of the orientation distributions of the structures in a full q-resolved analysis, which would allow obtaining complete structural information from these samples and investigating the effect of the different processing parameters employed for their fabrication.

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Shock mosaicism: observing microstructures developed by hyper-velocity impacts with dark-field X-ray microscopy

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Shock metamorphism is commonly formed by hyper-velocity impacts during collisions of two or multiple terrestrial planetary bodies. It is a vital deformation mechanism in the solar system, and it has been widely observed in meteoritic materials. The duration of shock is within milliseconds to seconds when shock pressure is unloaded instantaneously in the impactile, resulting in an extreme strain rate up to 10^5/s. Shock mosaicism, as a critical texture observed in shocked silicates, reflecting disordered crystal lattices formed by the randomly distributed dislocation during the pressure unloading. In this work, we present the systematic comparison of differently deformed samples, composed by terrestrial deformed kimberlitic olivine, low to moderately shocked olivine from ureilite, and heavily shocked olivine in the martian shergottite, from macroscopic scale of petrographic texture to quantitative analysis of 2-dimensional micro-X-ray diffraction (2D-XRD), to mesoscale of electron backscatter diffraction (EBSD), and to nano-meso scale of novel dark-field X-ray microscopy (DFXM). We show the coherence among each method, and we provide the first direct imaging observation on the shock mosaicism by using DFXM. We demonstrate the superior effectiveness of applying DFXM on probing dislocation distribution in the shocked silicates, proposing the dislocation network concept, formed during the shock pressure unloading, that prevents dislocation from further migrating upon the pressure uploading, unless a sufficient heating and annealing are applied.

«Characterization of macrozones in Ti-6Al-4V alloy»

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Depending on thermomechanical processing conditions, metals can develop strong crystallographic texture which in turn give rise to anisotropic mechanical properties of the material. Forged Ti alloys, used in aeronautical applications, can develop such pronounced local textures ("macrozones") which extend over mm (up to cm) sized regions. Figure 1 show an example of such a macrozone, which has been characterized by a combination of serial sectioning and EBSD.

Here we present first results obtained from a 4x4x4mm sized sample of Ti-6Al-4V alloy using both, Texture Tomography [1] and s-3DXRD [2] at the materials science beamline at ESRF.

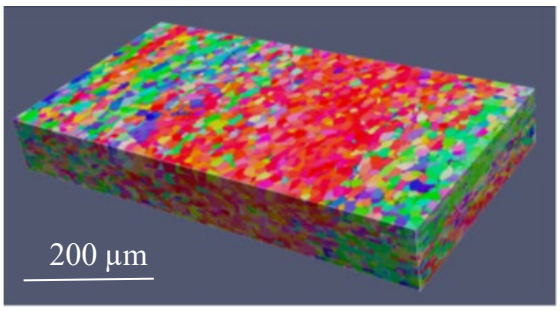

Figure 1: Example of macrozone in TA6V, characterized by serial sectioning and EBSD
(data courtesy J.S. Lecompte, LEM3, Metz, France)

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LaueMAX: X-ray Laue Diffraction Microscopy for Materials Science and Nanoscience

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LaueMAX is a new setup for X-ray Laue microcopy imaging which has been installed on the french CRG-IF BM32 beamline at the European Synchrotron (ESRF). This upgraded caracterisation and metrological tool allows one to capture the structural parameters in materials and microstructures by means of raster scans of a specimen under investigation illuminated by a 300x300 nm² sized polychromatic and intense X-ray beam. This advanced instrumentation is open the scientific community and still unique in Europe.

Laue X-ray microdiffraction is a well established technique for the studies of crystallised materials at the nanoscale (some 100 nanometers lateral spatial resolution) with a high angular resolution (0.01%) for quantitative assessment of local orientation and strain state of crystallographic unit cell. This technique is non invasive and suited to any cristallised materials and objects (oxides, metals, semiconductors), either isolated or in assembly.
On the software side, the structural parameters (orientation, lattice parameters, strain, stress) are determined by the analysis of Laue patterns recorded on 2D images composed of Laue spots. Due to the X-ray penetration or small crystal smaller than the beamsize, the net Laue pattern is the superimposition of numerous single Laue pattern corresponding to individual crystal scattering signal. Recently, the segmentation of images by individual single Laue patterns has been boosted by the implementation of a new indexing technique based on an artificial intelligence approach [1,2]. Complementary experimental measurements can be performed to assess the local full strain/stress tensor [3] and also obtain the location of crystals to reconstruct 3D map and depth resolved strain/orientation gradients [4]. Finally, Laue microdiffraction experiments can also be carried out in situ or operando (temperature, mechanical load, light emission, current injection, voltage, …) for a better understanding of properties and mechanisms involved during materials elaboration or under operation.

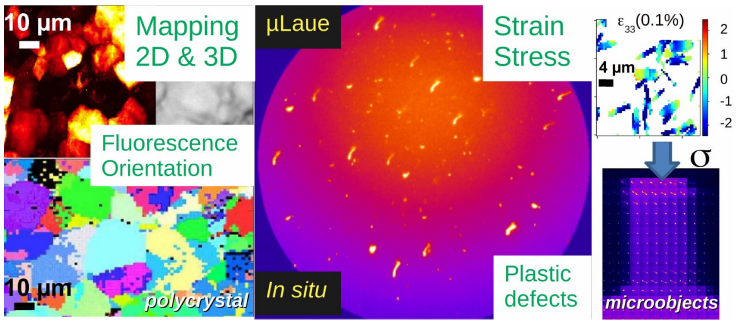

Figure 1: Summary of X-ray Laue Microscopy

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«Photonic Science and Engineering ltd Laue single crystal orientation system – TexTOM workshop 2025»

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Laue single-crystal orientation systems are crucial for crystal cutting, polishing, preparation for large facility experiments and many industrial applications such as single crystal turbine blade manufacturing. Key performance of these systems is measured by the accuracy of the alignment, cycle time and ease of use of Laue software.

Photonic Science and Engineering Ltd combines several key innovations to deliver the next generation of Laue single-crystal orientation. A high-resolution, high-sensitivity and robust X-ray CCD camera has an unbeaten active area for measuring Laue diffraction spots. A custom air-cooled X-ray beam solution eliminates the need for a water chiller while delivering a high-brilliance X-ray beam with a spot size of 450 µm for a standard system.

Our system comes with a dedicated software suite allowing users to drive motorised stages, the X-ray source, camera, and perform Laue analysis. The Laue analysis tool is crucial for speeding up the orientation of single crystals with automatic peak indexing, making it a truly user-friendly and efficient tool. The use of our solution been pivotal for several successful publications [1-3].

This combination of high detector resolution, bright diffraction peaks and small spot size leads to a typical exposure time of 1-3 seconds, shown in figure 1, with an accuracy of 0.1 degrees for a Si reference sample. The result is a fast and accurate Laue single-crystal orientation tool designed with ease of use in mind, delivering the next generation of Laue single-crystal orientation systems.

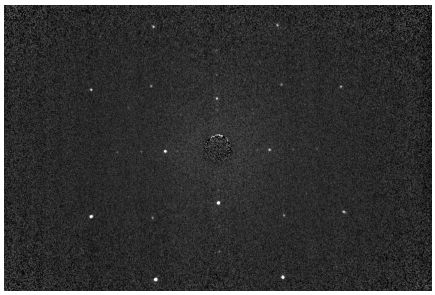

Figure 1: Back scattered Laue diffraction measured on a reference Si sample in 3s on the PSEL Laue single crystal orientation system.

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ForMAX - multiscale structure characterisation of hierarchical materials

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ForMAX is a new beamline at the MAX IV Laboratory, allowing multiscale structure characterisation of hierarchical materials by combining full-field microtomographic and scattering-based imaging in tandem [1]. Moreover, the custom detector configuration allows simultaneous small- and wide-angle x-ray scattering (SAXS and WAXS, respectively) in imaging mode [2], a particularly appealing features for the TexTOM community. Here, we will briefly describe the ForMAX beamline and exemplify its potential for multiscale structure characterization of bio-based materials.

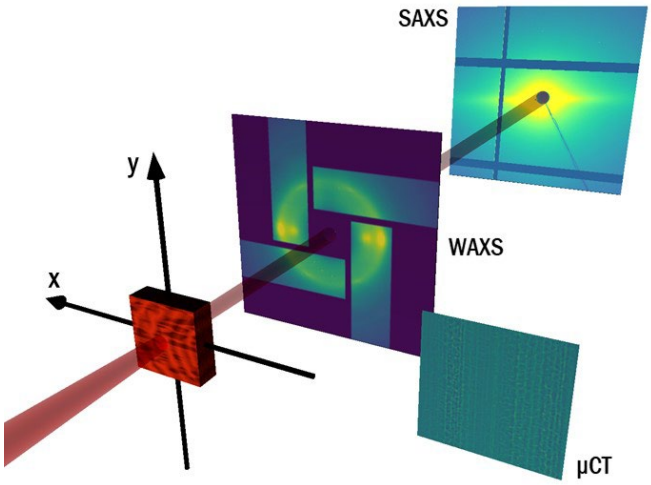

Figure 1: Custom detector configuration at ForMAX. The WAXS detector has a hole in the center that passes through the SAXS signal, allowing simultaneous SWAXS studies. The full-field microscope can be translated in and out of the x-ray beam path, allowing full-field microtomographic and SWAXS imaging in tandem.

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«Mapping orientation variations of nanocrystals in bone near cement line using texture tomography approach»

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The hierarchical structure of bone spans across many length scales from angstrom to centimeters, where each organizational unit contributes to its mechanical performance. At the nanoscale, coaligned collagen fibers and hydroxyapatite (HAP) crystals assemble into layered structures of different patterns with varied orientations [1, 2]. The spatial organization of these patterns and heterogeneity of the mineralized matrix are a result of a constant bone remodeling process – they define the mechanical behavior of bone on larger length scales [3]. Bone remodeling also leads to formation of structural features, for example, cement lines - highly mineralized borderlines between parts of altered bone structure arrangements, namely old and newly laid-down matrix [4]. Previous results from SAXS/WAXS tensor tomography [1] showed strongly varied 3D orientational organization and spatial changes in mineral properties, when comparing regions surrounding the cement lines with the lamellar bone. While these results give invaluable insight into matrix spatial arrangement, we still need to (1) characterize the region at the submicron scale to reach the desired resolution, which is achievable with recent advances in synchrotron technology, and (2) be able to describe the distribution of different orientation near cement line and its alignment with mineral properties, which is possible with the development of texture tomography approaches (TexTOM) [5].

For the purpose of the study, a piece of femoral bone was cut to obtain a sample of ~70x90x100 μm^3 containing the cement line and glued to PMMA. Prior to that, the lab absorption tomography was done to later align the TexTOM results with the absorption tomogram. The sample was scanned at the ID13 beamline, ESRF, France at X-Ray energy of 15 keV and 750 nm step size. A total of 262 projections were collected for 10 tilt angles between 0 and 45°. For TexTOM reconstructions the algorithm of Frewein et.al [5] was implemented both with one (002) HAP diffraction peak (tensor tomography approach) and multiple peaks (TexTOM approach).

We have successfully achieved submicron step size and performed TexTOM reconstructions to get texture information. The preliminary results show the presence of different orientational domains on each side of the cement line. The extracted local misorientations relative to neighboring voxels in mineral texture match the cement line border from absorption tomography. In addition to the cement line, it was also possible to obtain orientational information around several lacunae spaces – voids in matrix that store bone cells. The XRD-CT data provides valuable information on HAP mineral properties to align with absorption and texture tomography results. Thus, we have managed to characterize at a submicron scale the distribution of orientations by identifying the orientational domains. While it gives us a preview of texture surrounding cement lines, in order to properly access texture information inside the cement line, our next experiment will be done on a smaller sample at much higher resolution.

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I22 at the Diamond Light Source – 3D Small Angle Scattering Tensor Tomography (3D-SASTT)

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The I22 Small/Wide Angle X-ray Scattering (SAXS/WAXS) beamline was designed by Diamond Light Source for structural investigations of soft matter and biological materials. It operates in several modes including Transmission SAXS/WAXS, Microfocus SAXS/WAXS, Grazing Incidence SAXS/WAXS and Ultra-SAXS/WAXS. The 3D-SASTT non-destructive technique is one of the new emerging modes of I22 that is a combination of two techniques – SAXS and tensor tomography. 3D-SASTT extends the characterisation expertise to investigate and understand the 3D structural variations with the high imaging resolution at the nano-scale to understand 3D structural anisotropy in complex hierarchical materials such as teeth, bones, shells etc. In 3D SASTT at I22 samples are rotated through different angles around two orthogonal axis with 2D raster scans collected at each angle. For each scan, Pilatus P3-2M and Pilatus P3-2M-L are used to collect the SAXS/WAXS scattering patterns at a sample-to-detector distance up to 10m. A monochromatic 7-20 keV x-ray beam allows accessing the *q*-space within the range 0.024 to 32.3 nm^{-1} in a standard setup, or up to 470 nm in real space depending on the energy choice.

Abstract

3D Intragranular Orientation Mapping in Cold-Rolled Ferrite at Industrially Relevant Deformation Levels

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Thermomechanical processing of metals introduces high dislocation densities, altering material properties through mechanisms such as work hardening. Non-destructive 3D mapping of deformation microstructures remains a challenge due to limitations in characterization tools. This study combines Dark-Field X-ray Microscopy (DFXM) and texture tomography (TT) to investigate deformation structures in 50% and 65% cold-rolled ferrite. DFXM enables non-destructive 3D mapping of intragranular deformation structures, resolving dislocation cells with elevated densities and misorientation values up to 5°, at a resolution of 100 nm. TT complements this by providing 3D grain orientation data, aligning grains relative to the rolling direction and bridging the effects of neighboring grains on deformation. These data are also validated with complementary Electron backscatter diffraction (EBSD) measurements. The combined approach reveals the evolution of intragranular deformation structures, cell size distributions, and strain hotspots as a function of deformation, advancing our understanding of plasticity and the interplay between local and global deformation behaviors. This framework offers critical insights into deformation mechanisms and recrystallization in metals.

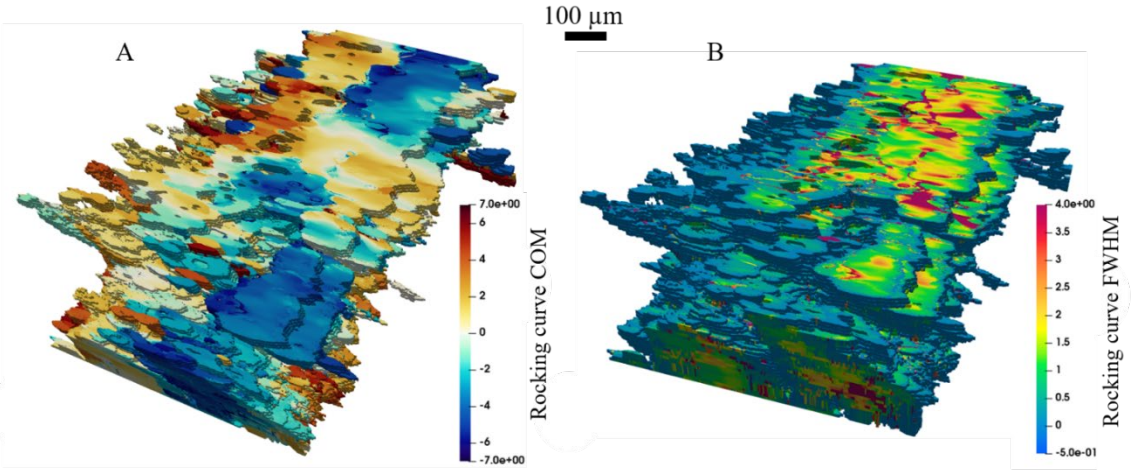

Figure 1. 3D reconstruction of deformation structures of a grain inside 50% deformed cold-rolled Ferrite. A) Rocking curve COM map B) Rocking curve FWHM map.

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«*In situ* Texture tomography to characterize the multiscale deformation mechanisms of the Achilles tendon enthesis in 3D – TexTOM workshop 2025»

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Introduction: The enthesis is an insertion site of tendon to bone with a highly complex organization and exceptional mechanical properties [1]. Despite a large mismatch in mechanical properties, failure typically does not occur at the interface, but rather within the tendon or bone. This highly durable attachment stems from a transitional cartilaginous zone with gradients in collagen and mineral content, organization and structure on multiple hierarchical length scales. If the enthesis is injured, however, the regeneration is poor and surgical repair often fails [1,2]. Despite extensive research into the structure and mechanics of this attachment, the local deformation mechanisms are still poorly understood. This study aims to characterize the multiscale mechanical response of the Achilles tendon enthesis in 3D by combining *in situ* mechanical testing of the mouse Achilles tendon enthesis with the newly developed technique X-ray texture tomography (TexTOM) [3] as well as phase-contrast µCT (PhC-µCT).

Methods: 40 enthesis samples were dissected from 20 healthy mice and stored frozen until experiments. A loading device was designed and custom-built (Figure 1) for compatibility with TexTOM and PhC-µCT measurements at ID15 and ID19 beamlines respectively at the European Synchrotron Radiation Facility. TexTOM measurements are planned to be carried out with an X-ray energy of 40 keV, 10µm beam size and at a detector distance of 800mm to obtain a q-region of 0.12-38nm^{-1}. The sample will be scanned during preload and 3 incremental strain steps at rotations between 0-360° at 7 tilt angles between 0-45°. The TexTOM data will be reconstructed and analyzed to retrieve the hydroxyapatite orientation, particle shape (0.3-3.5nm^{-1}) and crystal plane reflections (15-35nm^{-1}), as well as collagen fibril orientation, d-spacing (0.26-1nm^{-1}) and molecular spacing (20-25nm^{-1}). PhC-µCT measurements are planned to be carried out with an X-ray energy of 26 keV, 600nm pixel size over 1.5x1.5mm^2 and with 10s temporal resolution. The sample will be imaged at preload and incremental strain steps. PhC-µCT data will be reconstructed and analyzed for mineral and collagen fiber orientation and strain. Careful assessment of radiation damage will be conducted during the setup of both experiments to determine a suitable dose-delivery regime for valid results.

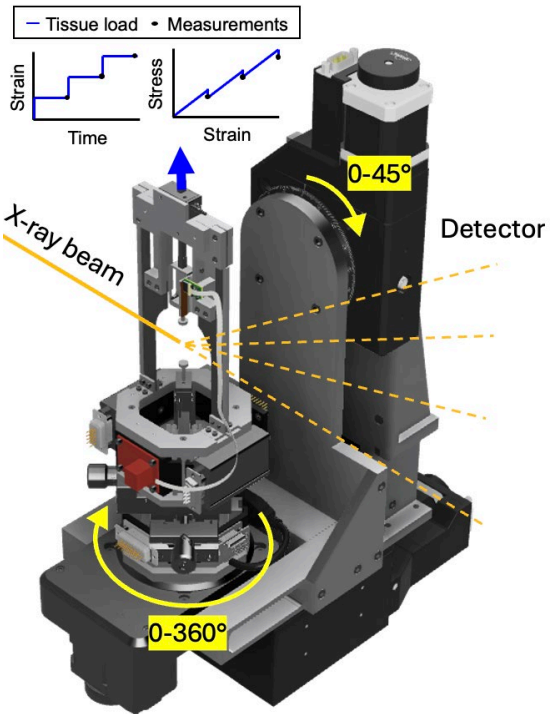

Figure 1. Device design, TexTOM measurement scheme and setup.

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«The Nanoscale Architecture of the Bone-Tendon Interface Probed with Multi-Modal, High Resolution X-ray Imaging Approaches – TexTOM workshop 2025»

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The interfacial region between tendon and bone exhibits intriguing remodeling dynamics and structural stability to overcome the bio-mechanically challenging task of connecting the soft tendon with the stiff bone tissue. In the case of the fibrocartilaginous enthesis the tissue is made up of fibrocartilage that exhibits a gradient in mineral content with a sharp mineral border called the tidemark [1, 2]. The effects of mechanical unloading and the resulting remodeling of the enthesis has not yet been elucidated on a nano-scale.

We investigate mouse Achilles heel entheses that were exposed to controlled periods of mechanical unloading as well as recovery periods in analogy to [1]. To solve the question of the changes in the nanostructure we measure the 3D crystallographic texture in the framework of the texture tomography methodology [3].

The samples were prepared in an extensive milling process. The sub-100 µm ROI was approached by a succession of a coarse milling [4] followed by a fine-milling with a focused ion beam. The free-standing sample cubes were then measured with a nanometer X-ray beam in a scanning tomographic experiment analogous to [3]. We present preliminary results and an outlook towards further experiments performed in the framework of this PhD project.

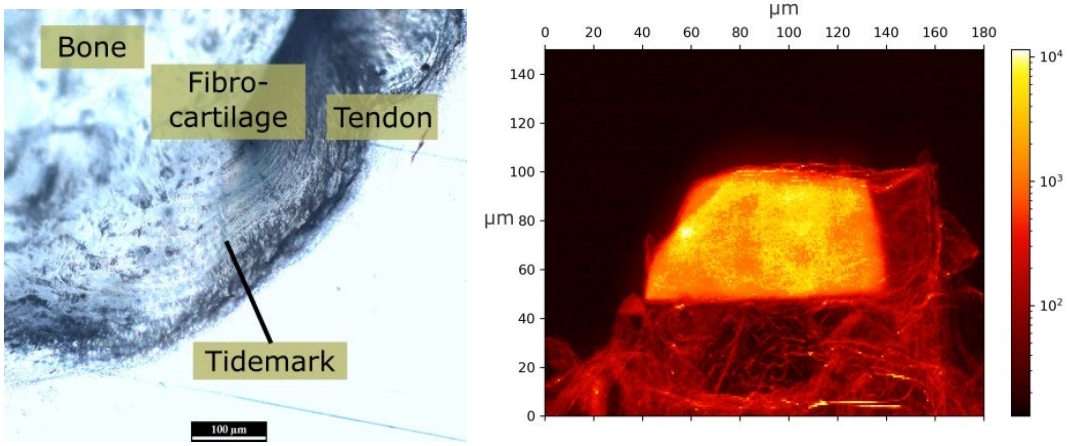

Figure 1: Left: Light microscope image of Mouse enthesis. Right: X-ray scanning SAXS microscopy image of milled sample block.

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Scanning 3DXRD at the Materials Science Beamline ID11/ESRF

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Micro- and nano-focused X-ray beams often give rise to "spotty" diffraction patterns. A suite of 3DXRD methods has been developed at ID11 to index these spots and find distinct crystal orientations in a sample. Recently the ESRF-EBS source upgrade has increased the X-ray flux by an order of magnitude, and the ID11 beamline was also equipped with a photon counting Eiger2 4M CdTe detector that runs at >500 frames per second [1]. These developments have delivered a step change in our ability to carry out fast diffraction tomography experiments.

To benefit from these upgrades there have been a lot of developments in software to process the resulting large datasets using the ImageD11 software package [2] that was historically developed at beamline ID11. Careful measurements of detector distortion [3] have been essential to extract precise strains. Figure 1 shows a reconstruction of a large dataset from a K-type thermocouple that corresponds to a scan of 2401x7220=17.3 million X-ray frames. The code has been tested on a range of other samples and datasets and it works well for samples containing undeformed crystallites giving 3D maps with 100 nm resolution [4]. The code has been extended to handle large deformations in metals [5] and also to extract maps of local strain tensors in these deformed samples. For complex mixtures of phases in geological materials, and also for very large samples, there are new developments to exploit the use of Friedel pairs during data processing [6] and locate the origin of diffraction peaks within a sample.

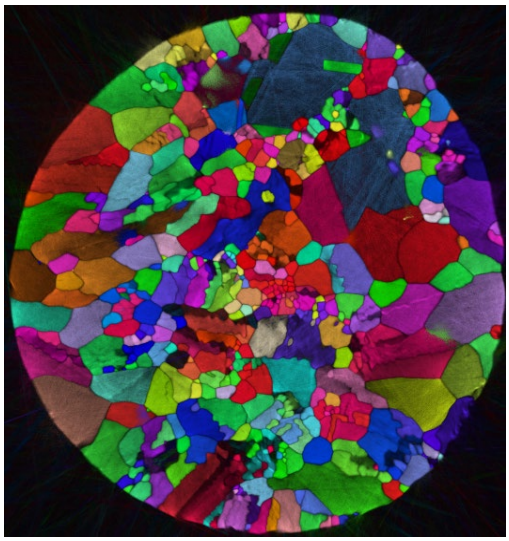

Figure 1: A cross-section of a K-type thermocouple, coloured by orientation with brightness as density.

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