

Applications of X-ray Tomographic Microscopy (XTM) in materials and life sciences

Due to the high beam brilliance at synchrotron light sources like SLS, ESRF and HASYLAB, the spatial resolution of computed tomography has been extended down to the 1 μm range. A dedicated X-ray tomographic microscopy (XTM) station with two detectors and a novel testing device for compressive loading has been developed and commissioned at SLS for samples from materials research and life science.

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X-ray based computed tomography (CT) is well established in medicine, in materials science and in industry. Tomography with X-ray tubes specialized for industry and materials science applications reaches a spatial resolution of about 10 micrometers for an object size of a few millimeters. Investigations of material properties, biological specimen and microsystems often require a resolution of one micrometer or better. Tube based CT-systems do not reach this limit at feasible exposure time. The reason is the by far too small X-ray photon flux emitted from a tube target spot as small as one micrometer. Synchrotron sources, however, do not have this limitation and deliver a highly brilliant beam. High brilliance means high intensity and small divergence. The X-ray sources of the SLS have these characteristics and are very well suited for XTM.

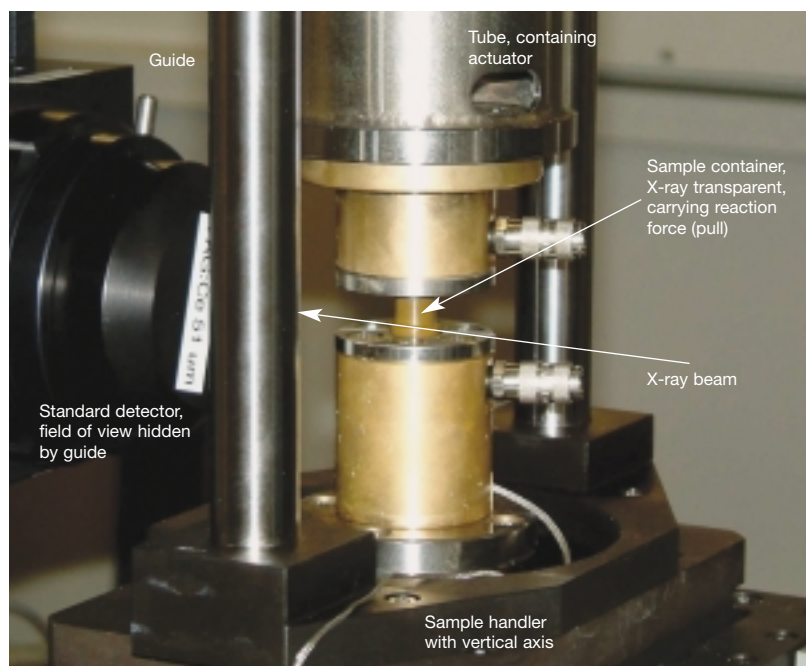
The instrumentation for XTM was designed and built as a common project of EMPA as project leader, IBT of ETHZ/UNIZ, SLS/PSI, and financially supported by ETH-Council. Major design criteria were sam-

ple rotation with smallest runouts, stable rotation, low irradiation doses of the samples, minimum data acquisition and transfer time for the approximately 1000 full frame exposures required for tomogram reconstruction, a spatial resolution of 500 nanometer to 5 micrometer depending on field of view, and the best possible X-ray absorption and phase contrasts. The sample handler had not only to fulfil very tight mechanical tolerances but also to offer a sufficient load capacity to support an in-situ mechanical compression and staining device (IMCSD) (Fig. 1). A novel Bragg magnifier detector had been included to access sub-micrometer resolution range. The first six months of operation included commissioning, test runs and already several experiments. After some corrections on beam-line, apparatus and control software, the expected performance was achieved.

In Fig. 2, we show results from osteoporosis research with single bone trabeculae, mouse femurs and stained trabecular bone samples (Fig. 2). The applied lead-uranyl-acetate stain penetrates all voids and microcracks within the structure. Fig. 3 shows that microcrack formation and propagation induced by cyclic compressive loading at different load amplitudes can well be visualized by XTM using the IMCSD, which has been designed and built by EMPA. The direct mounting of the IMCSD onto the sample handler guaranteed that the position of the sample relative to the coordinate system of the tomography rotation axis remains unchanged between scans at different load steps. This simplifies very much the comparison of the structures before and after loading, which is required for failure modeling of bone structure. In the future, also cell distributions in bone will be visualized. Novel staining techniques have been developed for this task using a model system.

Three dimensional inspection of micromechanical high precision tools is shown in Fig. 4. Attempts of the manufacturer to characterize the inner geometry of these wire bonding capillaries by using scanning electron microscopy as well as a variety of other methods had not delivered sufficiently accurate data. First investigations with 3D tomography now accurately reveal the deviations of diameter and details of shape of the bores. This demonstrates that XTM will be a well suited method for investigating the inner structure of MEMS. In combination with IMCSD robustness, local deformation parameters and reliability of micro-devices can now be investigated non-destructively.

Fig. 1: In-situ Mechanical Compression and Staining Device (IMCSD) of EMPA mounted on sample handler of XTM station.



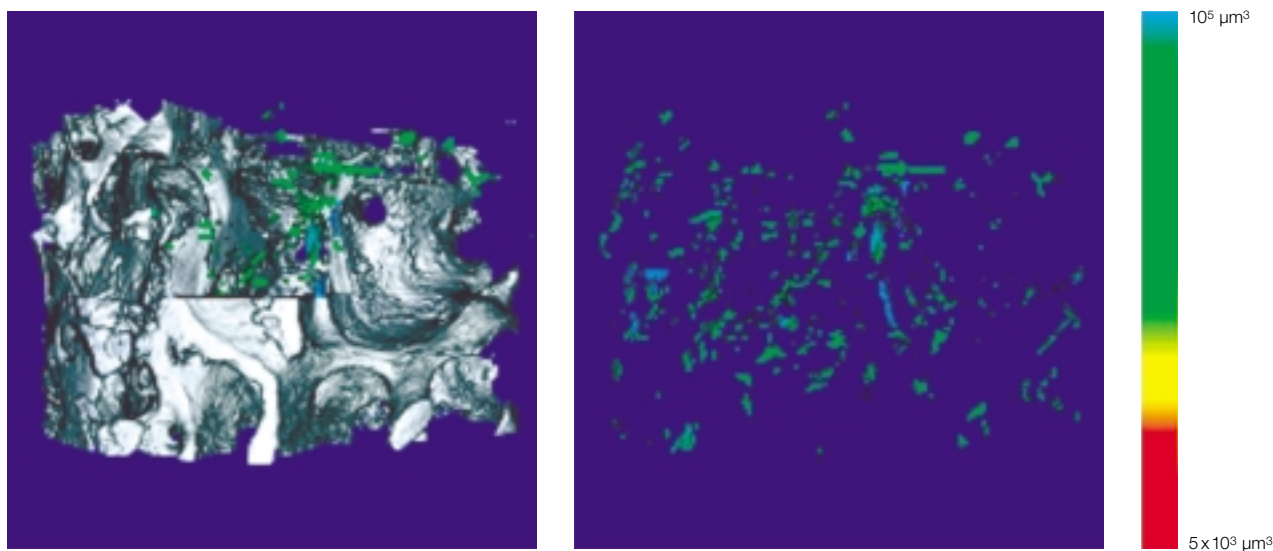


Fig. 2: 3D views of trabecular bone sample with lead-uranyl-acetate stained microcracks and cavities containing osteocytes. Microcracks and cavities are colored according to their size in μm^3 . Left: The bone is in partially cut block view. Right: The bone is fully transparent.

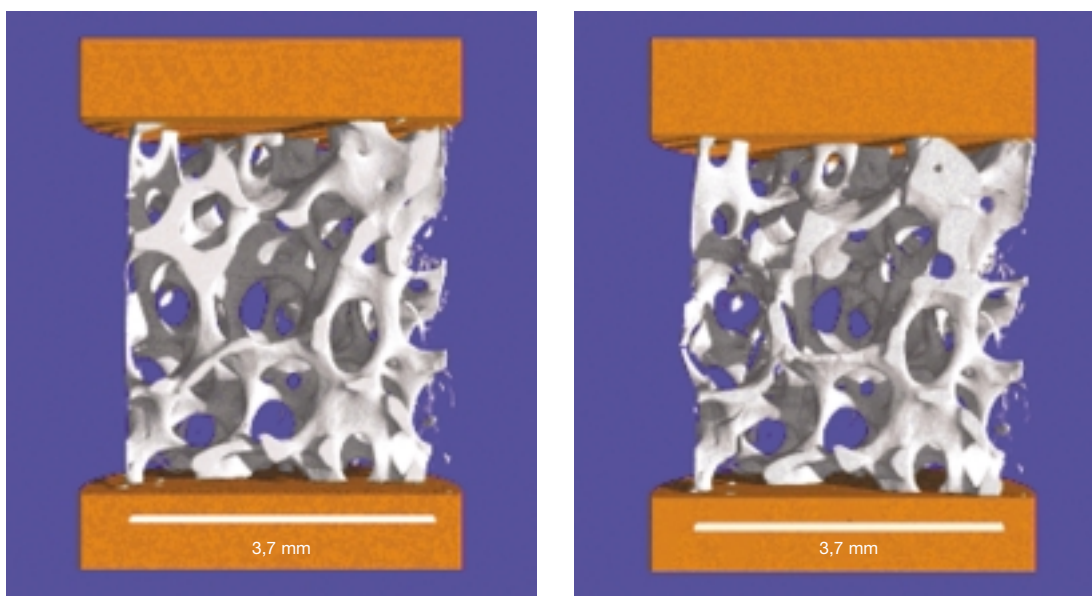


Fig. 3: 3D view of bone sample in the IMCSD before (left) and after (right) compressive loading.

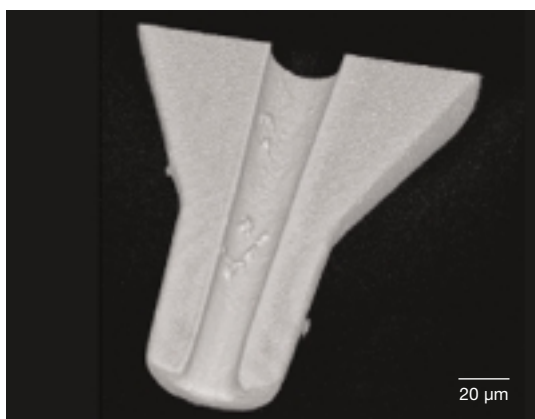


Fig. 4: 3D view of wire bonding capillaries for extracting dimensions and details of shape.

Support: ETH-Council

Links: www.empa.ch/abt173
> Nondestructive test methods

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References:

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