ESRF

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Microbeam Small-Angle X-Ray Scattering Tomography

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Report: Microbeam Small-Angle X-Ray Scattering Tomography as an invasive, but non-destructive technique allows one to gain information about the local nanostructure inside a sample. SAXS microtomography with a real space resolution of 3µm is demonstrated. We acquired SAXS tomograms of natural and artificial samples. Therein the main focus have been ancient parchment samples with ink.

For the microfocus Beryllium parabolic refractive lenses were used, for the first time with a radius in the apex of $\leq$ 50 µm. Therefore, a setup to achive a spot size of 1 µm in the horizontal scanning direction was build up. While a beam size of 0.2 x 1.14 µm^2 was generated that way, it could not be used for the SAXS experiment due to mechanical contraints of the setup, i. e., the guard slits did not fit at the right position. This difficulty can be overcome in future constructions. For the current experiment we changed to a 3µm focus setup. Using 8.02 keV and 0.1 mm horizontal gap for the primary slits, the 10 Be Lenses provide a focal distance of 0.455 m and a horizontal spot size of 2.5 µm, veryfied by knife edge scans. The samples were put on a gioniometer head and in the focal plane, slits block parasitic scattering infront of the sample, cf. Figure 1.2. In L_{DS} = 2.44 m distance the SAXS patterns were recorded by a ‘FreLon 2k’ detector, provided by the ESRF detector group. The main beam was blocked by a beamstop. High-resolution translation and rotation stages allowed us to use this µ-SAXS setup to perform tomographic scans of the sample. We acquired 3 data sets of ancient parchment samples with and without ink, 2 dried drops of nano composite polystyrol particles of various sizes, and human hair creatine. Also high resolution scans were recorded of sheep, gloat, and deer parchment as well as bovine leather in various thicknesses.

As example for the experimental results an ancient parchment sample from the year 1765 is shown, cf. Figure 1. One small piece with ink <300 µm in size at the scanning slice was cut out of the sample and positioned in the focal plane of the lenses.

Despite an unexplained slowly fluctuating beam intensity we were able to reconstruct the data. For an overview, the reconstructed integrated scattering intensity is plotted in Figure 1.4. The virtual slice though the sample shows the loose tissue of the parchment as well as the strong scattering from parts with dried ink. The high resolution is needed to analyse the region, into which the ink penetrated. This is best viewed by tomographic imaging avoiding destructive cutting of the sample.

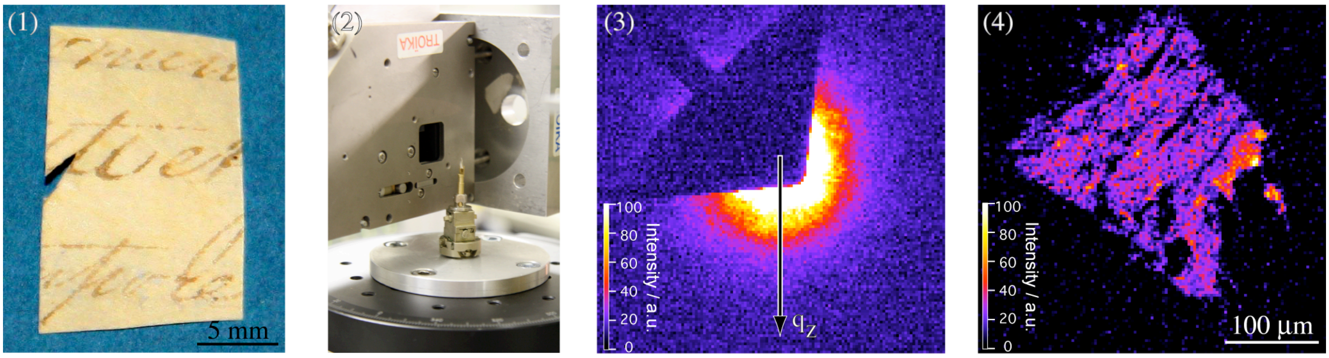

Figure 1: (Color)(1) A piece of a writing on parchment from the year 1765. A small cut thereof is the sample in the experimental setup of image (2). A typical SAXS pattern is displayed in figure (3). SAXS Microtomography leads to a virtual slice through the sample plotting the reconstructed integral scattering intensity with 3µm pixel size (4).

We thank T. Wess and K. Thomas (Structural Biophysics Group, School of Optometry and Vision Science, Cardiff University, UK) for providing the parchment samples.

A detailed analysis of the highly scattering region concerning the development of the scattering distribution is still ongoing, as well as the comparison of the different samples. The reconstruction is rigorous along the rotation axis of the sample corresponding to the q_z direction. A full reconstruction can be performed if the SAXS pattern is rotational invariant in each tomographic voxel, which is given to good approximation for some of the samples (cf. Fig. 1.3). The data analysis for other samples is still ongoing.

With the experiments made, we could address both radiation damage and modelling issues: Some experimental conditions lead to strong radiation damage, resulting in immediate or time delayed destruction of the sample. From this we can estimate radiation damage effects in these kinds of samples. All the SAXS patterns show speckle, indicating a high degree of coherence in the focused beam. In our current reconstruction, this is not taken into account. However, by increasing the coherence beyond the beam size (diffraction limit), scanning SAXS techniques merge into pychographic imaging techniques, allowing the reconstruction of the object on the nanoscale.

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