ESRF	**Experiment title:** Nanotomography based on hard x-ray microscopy with Be parabolic refractive lenses	**Experiment number:** MI-715
Beamline: ID22	**Date of experiment:** from: July 13, 2004 to: July 19, 2004	**Date of report:** August 30, 2005
Shifts: 18	**Local contact(s):** Pierre Bleuet	*Received at ESRF:*

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Report:
The aim of the experiment was to setup a hard x-ray microscope based on beryllium parabolic refractive x-ray lenses and record tomographic data sets at a spatial resolution well below 1μm, i. e., < 200nm. We setup this microscope at ID22, using both experiment hutches. The sample was illuminated by monochromatic (Si 111 double crystal monochromator) undulator radiation in the range between 14 and 18keV that was reflected off a flat mirror (Pd coated, reflection angle 0.14°) to reduce the contribution of higher harmonics. To reduce the lateral coherence of the illumination and to match the aperture of the objective lens, a rotating diffuser (B_4C powder between two thin glass plates) was placed a short distance before the sample. The sample located at 41.2m from the source in EH1 was imaged by an objective lens placed $L_1 \approx 1$m behind the sample onto the FReLoN2000 camera that was located $L_2 = 23.9$m behind the lens in EH2. In this way, an image of the sample was formed on the CCD with 23.9 fold magnification. Theoretically, a diffraction limited resolution of 95nm is expected for this setup.
As part of the sample stage of the x-ray microscope a high precision rotation stage with air bearing allows one to rotate the sample around an axis perpendicular to the beam with low eccentricity, flatness (both < 100nm), and whobble (< 2.5μrad). This degree of freedom allows one to record a tomographic data set of magnified projection images.
In a first step, we have focused the microscope with a gold line test pattern. It turned out, however, that the spatial resolution measured was significantly reduced compared to the expected result, i. e., $\approx 0.9\mu$m. This result can not be explained by aberrations of the x-ray optic, since with the same optic at least 250nm resolution were achieved

in an experiment performed at APS and 140nm resolution were obtained in a compact geometry at ID22 (cf. MI-506). However, it is believed that the image quality was deteriorated in the beam path between the two experiment hutches. This was confirmed by independent measurements of the lateral coherence that was significantly reduced in the second hutch compared to the first [P. Bleuet, private communication]. This problem could not be resolved during this beamtime and all data acquisition with the x-ray microscope was affected by this.
Nevertheless, we recorded tomographic data with the x-ray microscope of a set of uranium particles provided by the beamline and two fragments of an AMD Athlon XP 2000 microprocessor. In addition, a limited angle tomogram of a full (unthinned) AMD chip was recorded in an angular intervall of $\pm 60°$.
For comparison, we recorded several projection tomograms (without magnification) at $E = 18$keV with the standard tomography setup in EH2. Data sets of one of the fragments of the microprocessor and of concrete filled into a thin capillary were recorded.
Besides the spatial resolution issue that could not be resolved during the experiment, two other issues concerning tomographic microscopy were identified:

- At high spatial resolution, stable sample fixation is not trivial. In particular, glues take a long time to settle into a final state. The uranium particles were fixed with two-component glue long before the experiment, avoiding this instability. However, this glue was not radiation hard, forming color centers and deforming measurably under irradiation, although it had been used in previous microprobe experiments. Therefore, a sample fixation where glue is illuminated by x-rays is not suited for tomographic microscopy. If the glue is not illuminated, as for example in the case of the microprocessor, the problem does not arise. Similarly, radiation hardness of the sample is required.
- For the microprocessor samples, the thin object approximation seems to break down, as small periodic structures can significantly refract a ray away from its straight path assumed for parallel projection. This leads to artifacts in the tomographic reconstruction. The issue is currently investigated theoretically, considering the limits of tomographic microscopy.

In previous experiments, it has been shown that magnified tomographic imaging with refractive lenses is possible with spatial resolution in the sub-micrometer range [1, 2]. Despite the difficulties encountered during this experiment, several issues were identified that will be addressed to improve the technique in the future.

References

[1] C. G. Schroer *et al.*, Appl. Phys. Lett. **81**, 1527 (2002).

[2] C. G. Schroer *et al.*, in *Developments in X-Ray Tomography IV*, Vol. 5535 of *Proceedings of the SPIE*, edited by U. Bonse (SPIE, Bellingham, 2004), pp. 701–708.