ESRF	**Experiment title:** Hard x-ray fluorescence microtomography and scanning microscopy with sub-micrometer resolution using refractive x-ray lenses	**Experiment number:** MI-704
Beamline: ID13	**Date of experiment:** from: Jan. 25, 2005 to: Feb. 8, 2005	**Date of report:** March 1, 2006
Shifts: 18 + 12 + 9	**Local contact(s):** M. Burghammer	*Received at ESRF:*

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Report:

The third beam time of this long term proposal served the preparation of the nanoprobe setup for two user experiments at ID13: ME-887 and ME-889. The nanoprobe had been set up at the microprobe station in EH2 of ID13 during the previous experiment MI-704-2 shortly before the winter shutdown and remained at the ESRF until the end of MI-704-3. For details about the setup see experimental report MI-704-2.

One major task for this beamtime was the integration of the system into the SPEC control system at the beamline. For this purpose, Jens Patommel from Aachen University came to the ESRF about one week prior to the experiment to do the necessary programming together with Manuel Perez (BLISS contact). A socket-based server was written to communicate with SPEC. The control of a high resolution camera, a PIN-diode, and various mechanical degrees of freedom was implemented.

For the upcoming diffraction experiments it was crucial to shield the diffraction camera from all stray radiation produced before the sample. Therefore, additional (more extended) shielding was introduced into the setup. The nanobeam is fed through a guard pinhole (Pt, 250μm thickness, $\approx$ 16μm diameter) without touching it, efficiently removing stray radiation at the nominal energy. However, higher harmonics are still transmitted and diffracted by the guard pinhole that needed to be removed in the relevant angular range by an additional lead collimator. Near the center of the diffraction pattern, a contamination by higher harmonic remained (see exp. rep. ME-889).

The nanobeam was generated at $E = 15.2$keV (flux: $6 \cdot 10^8$ph/s). A lateral beam size of 140×150nm^2 (H$\times$V) was measured with a gold fluorescence knife-edge. Mechanical

instabilities led to fluctuations in the effective beam size of the order of 100nm. Long-term drifts were investigated by centering the knife-edge on the beam and observing the fluctuations of the gold fluorescence.
Two user experiments (ME-887 and ME-889) were successfully carried out with the setup. The main difficulties encountered during the experiments resulted from mechanical instabilities. Since this experiment, changes in the design of the device were made to address stability issues. The sample stage was redesigned to be less prone to horizontal oscillations. In the last experiment of the LTP, vibrations will be monitored and active vibration damping by means of an appropriate table will be tested. It is also planned to demonstrate fluorescence tomography with high spatial resolution.
During the last year, the record focus measured during MI-704-2 was published in Appl. Phys. Lett. [1]. The article was selected for publication in Virt. J. Nanoscale Sci. Tech. [2] and was highlighted in *Nature Materials*, nanozone news (http://www.nature.com/materials/nanozone/news/051006/portal/m051006-3.html) as well as in the ESRF Highlights 2005 [3]. The results were also presented in a talk at the 8th International Conference on X-ray Microscopy in Himeji, Japan [4] and at the XX Congress of the International Union of Crystallography (IUCr2005) [5].

References

[1] C. G. Schroer, O. Kurapova, J. Patommel, P. Boye, J. Feldkamp, B. Lengeler, M. Burghammer, C. Riekel, L. Vincze, A. van der Hart, and M. Küchler, Appl. Phys. Lett. **87**, 124103 (2005).

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[5] C. G. Schroer, O. Kurapova, J. Patommel, P. Boye, J. Feldkamp, B. Lengeler, M. Burghammer, C. Riekel, and L. Vincze, Acta Cryst. A **61**, C64 (2005).