ESRF	**Experiment title:** Hard X-Ray Nanoprobe for Fluorescence Tomography, Nanodiffraction, and Coherent Diffraction Imaging	**Experiment number:** MI-836
Beamline ID 13	**Date of experiment:** from: 2009-02-25 to: 2009-03-04	**Date of report:** 2011-02-10
Shifts 33	**Local contact(s):** S. Schoeder, M. Burghammer, C. Riekel	*Received at ESRF:*

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Report:
The principal object of this experimental session of the long-term project MI-836 was to implement and test the optics module for the newly installed nanoprobe setup of ID 13. In the time before this session, the prototypical nanoprobe (designed and built by TU Dresden) was brought from Dresden to Grenoble and temporarily installed at ID 13. The experience with that prototype was used to install an advanced nanoprobe instrument in the extension hutch EH 3 at ID 13, for which we delivered the optics module with nanofocusing lenses (NFLs). The plan was to integrate the optics module into the nanoprobe, to align the NFLs, and to characterize the nanobeam with absorption and fluorescence knife-edge scans and with far-field measurements. In addition, we planned to systematically measure the flux gain of several two-stage focusing schemes and the resulting coherence properties. Further, we tested a new generation of nanofocusing lenses made of diamond (Fig. 1). Moreover, we performed a number of experiments, as, for example, fluorescence element mapping of geological and biological samples, coherent x-ray diffraction imaging with nano-sized gold particles, and we started a first attempt to carry out an x-ray ptychography scan. Subsequent to our experimental session, we supported other users with their experiments, providing the nanobeam generated by the aligned nanofocusing lenses. During this experimental session lots of new instrumental components were successfully tested and many new methodical issues were treated. We could demonstrate that the new nanoprobe setup at EH 3 in combination with the NFL optics module provides a stable nanobeam ready to be used in many experiments. After the NFLs had been aligned, we characterized the nanobeam and used the results for identifying aberrations of the lenses [1, 2]. We also performed systematic prefocusing tests, from which we gained important information about the influence of prefocusing on flux and coherence. The flux in the nanofocus, for example, could be increased by a factor of $\sim$20, while the focus size increased by a factor of $\sim$1.5, only.
This experimental session was also used to test nanofocusing lenses made of diamond. We cooperate with Diamond Materials in Freiburg, Germany, in order to fabricate diamond lenses with improved x-ray optical properties compared to silicon lenses. The manufacturing process is quite challenging, and thus, the shape of the diamond lenses is not ideal, yet. Still, we could demonstrate a focus size of 360 nm FWHM generated with NFLs made of diamond. This focus is dominated by spherical aberrations. In the future, improvements in the fabrication should lead to a significant reduction in beam size.

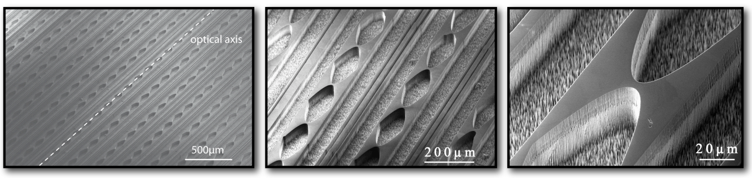

Figure 1: SEM images of nanofocusing lenses made of diamond [1].

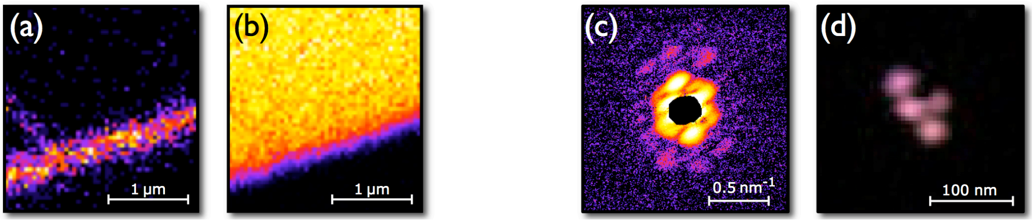

Figure 2: Results of fluorescence element mapping and CXDI. (a) and (b) show the two-dimensional element distribution of Yb and Y near a grain boundary within a model sample from geology. (c) The diffraction pattern of a cluster of 4 gold particles with 50 nm diameter. (d) Reconstructed electron density of the gold particles. (Published in [3].)

We carried out fluorescence element mapping on a model sample from geology. We investigated the region near the boundary between two grains of the object with the intention to study the grain boundary diffusion versus bulk diffusion of Yb in YAG. Fig. 2(a) and (b) show distribution maps of the elements of interest. The spatial resolution was about 100 nm in both, the horizontal and the vertical direction, conforming with the size of the nanobeam.
We performed coherent x-ray diffraction imaging on a cluster of 4 gold particles with a size of 50 nm each. The particles were illuminated by the $100 \times 200\,\text{nm}^2$ sized nanobeam, while the diffraction pattern was recorded by a FReLoN CCD detector in 2×2 binning mode at a distance of 1850 mm behind the sample (sum of 20 images with 60 s exposure time, each). Fig. 2(c) shows the diffraction pattern from which the projected electron density was reconstructed as seen in Fig. 2(d).
X-ray ptychography is another method using coherent x-ray diffraction, combining CXDI with scaning techniques. Ptychography wins over CXDI in several aspects. First, in contrast to CXDI, the sample investigated by ptychography is allowed to be larger in transverse direction than the illumination. Second, ptychography needs no prior information about the illumination function. Third, ambiguities of the reconstructed object are avoided by ptychography. And, last not least, ptychography provides the complex illumination function (phase and amplitude) in addition to the object function. We used the nanoprobe setup in order to carry out ptychography scans and tried to evaluate the data. Unfortunately, the quality of the data was not sufficient, and the reconstruction failed. But we learned much about this new trend-setting method and we are going to pursue this technique during the long-term project MI-836.
Besides performing our own experiments, we also supported other users, providing nanobeam expertise and aligning the nanofocusing lenses. For example, we contributed to the fluorescence experiments by Vince, et al. with their specimens from the stardust mission. In addition, we supported Hanke, et al. with their investigation of individual SiGe/Si(001) dot molecules. In that experiment, single dot molecules were illuminated by the nanobeam, and the fluorescence signal was recorded in parallel with the scattering signal [4].

References

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