The microXAS beamline at the Swiss Light source: towards nano-scale imaging

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Abstract. The microXAS beamline is a dedicated hard X-ray microprobe facility allowing a combination of fluorescence, spectroscopy and diffraction techniques in an energy scale from 4 to 23 keV. This paper presents a short review of the beamline, including the recent developments towards nano-scale imaging.

1. General description of beamline
The small source size and low emittance of the new 3rd generation synchrotron sources in combination with recent advances in the design and production of hard X-ray focusing optics opens up new possibilities for high-flux hard X-ray microprobe beamlines.

The microXAS beamline is located at the long straight section X05L of the SLS ring. A minigap in-vacuum undulator (U19) serves as radiation source providing high brightness X-rays in the energy range from 4 to 23 keV. The photon flux delivered at 12 keV is $3 \times 10^{12}$ photons/sec, while the optical elements used ensure an energy resolution of $\Delta E/E < 10^{-4}$.

The optical layout of the beamline is composed of several pairs of slits, a bendable toroidal, horizontally deflecting mirror and a monochromator. The vertical mirror unit serves three main purposes: (i) to collimate the beam in the vertical dimension, (ii) to allow for dynamic demagnification in horizontal dimension, and to act as a low-pass filter with an energy cut-off of ~23 keV given by the Rh coating. The horizontal focusing corresponds to the first part of a two-step focusing strategy that offers two main advantages: a secondary source with flexible size adjustment by precision slits (the capability of dynamical focusing and the possibility of optimizing the overall acceptance of the subsequent microfocusing optical system. The fixed–exit double-crystal monochromator is equipped with three different pairs of crystals: Si(111), Si(311) for higher energy resolution and Ge(111) for higher flux throughput.

2. Micro-probe setup
The entire micro-probe setup is installed on a stable optical table. For achromatic focusing in the entire energy range of 4-23 keV we use an elliptical shape mirror pair in the Kirkpatrick-Baez (KB) geometry, commercially available from Xradia. The mirrors are 10 cm in length, coated with Rh and enclosed in a He chamber that ensures their thermal stability. The acceptance of the horizontal and vertical mirrors at a nominal incident angle of 3.1 mrad is ~ 0.3 mm for both directions. The respective nominal focal distances are 150 mm and 260 mm for the centers of horizontally and vertically
focusing mirrors, respectively. Since the source is strongly asymmetric, the horizontal and vertical dimensions of the focal spot are not the same. Highest total photon flux density is obtained for a 6×1 µm² spot. A more symmetric spot size near 1 µm² can be obtained by dynamically reducing the secondary source size. Figure 1 shows typical beam profiles as measured by means of knife edge scans. ‘Reasonable effort’ spot size values measured using monochromatic beam are 1.3 (H) × 0.9 (V) µm², while using a white beam the spot size is 1.3 (H) × 0.75 (V) µm². The monochromatic photon flux measured in the focal spot is in the order of 10¹⁵ photons/sec.

Figure 1. Beam size measured by knife-edge scans for micro-focused white light (left panel) and a monochromatic micro-beam (right panel).

Several types of detectors and beam monitors are available at the beamline corresponding to the needs of the different experimental setups available. For the micro-spectroscopy setup we use a single element Si(Li) detector (Ketek) with an active area of 50 mm². The XRF map in Figure 2 shows the spatial distribution of Ni fluorescence intensity obtained from an 80 nm thick Ni patterned film fabricated by electron beam lithography using lift-off technique. The size of the grid lines was 1 µm in both horizontal and vertical directions, with a period of 10 µm. The sample is mounted on a fully encoded XYZ manipulator, with encoder resolution of 50 nm for the horizontal axis and 100 nm for the other two directions. Additional X-Y piezo nanopositioners, as well as 2 sets of rotation stages, can be mounted on the sample manipulator in case precise sample positioning is needed (for example in the micro-XRD setup). For monitoring the incoming micro-beam we use an ion chamber of reduced secondary source size. Figure 1 shows typical beam profiles as measured by means of knife edge technique. The size of the Ni grid lines is 1 µm and a period of 10 µm. The middle panel shows an average of 5 unsmoothed µ-EXAFS scans acquired at the intersection of two Ni lines. The third panel shows the corresponding EXAFS oscillations and the corresponding fit.

The µ-EXAFS at the Ni K-edge was performed at the intersection of two grid lines, using a beam size of 1.3 µm (H) x 0.5 µm (V). The average of 5 unsmoothed scans is shown in Figure 2 (middle panel), while the corresponding EXAFS oscillations in k-space are shown in the right panel. The fit shown with continuous line was performed using the IFEFFIT package, indicating the presence of 6 Ni nearest neighbors at a distance of 2.45(7) Å, as expected for bulk Ni²⁺.
3. Nano-probe setup

Focusing of soft X-ray beams with Frenel zone plates (FZP) has proven to give focal spots with excellent spatial resolution combined with low background [1]. The diffraction limit for the resolution of a FZP is on the order of the smallest, outermost zone width, meaning that nanolithography processes with very high resolution is required. Efficient focusing of hard X-rays is more difficult, as additional problems must be overcome: to achieve acceptable diffraction efficiencies, the zone plate structures should be made from heavy materials and the required height of the structures needs to be a micron or more. This means that the structures have to be 10 or even 20 times higher than their width, when both high resolution and efficiency is needed. Lensless X-ray microscopy has been successfully demonstrated at our beamline at 6-8 keV [2]. Here we describe recent results on the focusing of 15 keV X-rays using gold FZPs with an outermost zone width of 100 nm and a profile height of 1 µm. The fabrication process of gold FZPs with an aspect ratio of more than 10 is described in reference [3].

A schematic drawing of key components of the setup used at the micro-XAS beamline is shown in Figure 3. Experiments were performed in air at atmospheric pressure using an X-ray energy of 15 keV. The X-ray beam passes first through an aperture and then it is focused by the FZP. In order to reduce the background caused by the divergent radiation of the higher diffraction orders, a 5 µm order selecting aperture (OSA) is put 3 mm downstream of the FZP. The coherence length of the X-ray beam at this energy is ideally about 100 µm in vertical direction and 20 µm in horizontal direction. In order to cut the incoherent part of the radiation we used a 20 µm coherence defining aperture in front of the FZP. The FZP has a diameter of 30 µm and an outermost zone-width of Δr·N = 100 nm and therefore, only zones down to Δr·N = 150 nm were illuminated through the aperture. In case of fully coherent illumination, a focal spot of \( \delta = 1.22 \times \Delta r = 182 \) nm in the first diffraction order could be achieved in our case.

![Figure 3. Schematic drawing of the focusing setup and SEM-image of the test pattern used for imaging (right) and of the reconstructed fluorescence map (left). Only zones down to 150 nm were illuminated through a 20 µm aperture in front of the zone plate.](image)

A number of improvements can be done to increase the aspect ratio of the structures of the FZPs, and therefore further reduce spatial resolution at higher energies, as further described in reference [4]. The future development plans at the microXAS beamline include full implementation of hard X-ray nano-imaging capabilities, starting with fluorescence imaging and going to nano-spectroscopy in a later stage.

References

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