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X-ray measurements with micro- and nanoresolution at BESSY ☆

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Abstract

Capabilities of the KMC-2 beamline at BESSY for spatially resolved X-ray measurements with micro- and nanometer resolution have been reviewed. A combination of experimental methods of X-ray fluorescence analysis and extended X-ray absorption fine spectroscopy with X-ray standing waves technique was applied for the depth profiling of Si/W/Si layers with sub-nanometer resolution. The investigated layers were placed into the waveguide structure formed by two Au films to increase sensitivity and accuracy of the measurements. In-depth resolution on the order of 1 nm for the structure measurements has been obtained.

Keywords: X-ray fluorescence analysis; X-ray absorption fine structure; X-ray standing waves; X-ray waveguides

1. Introduction

The use of X-rays as a tool for microscopy and nanometer technology is developing very rapidly. This can be attributed to the development of synchrotron radiation sources and on the progress in optics elements and methods for the X-rays beams control such as focusing and space modulation using diffraction phenomena in nanostructures. X-ray microprobes are widely used in combination with the most modern experimental technique, such as extended X-ray absorption fine spectroscopy (EXAFS), X-ray absorption near-edge structure (XANES) and micro-fluorescence analysis [1].

– Three-dimensional micro-fluorescence analysis has reached spatial resolution on the order of 10 μm [2]. This exploring glass capillary optics for an X-ray beam focusing on a sample as well as capillary lens placed in front of the detector to provide in-depth spatial resolution.
– An elliptically shaped glass monocapillary with a spatial resolution of 5 μm has been used for the fine two-dimensional focusing of the pre-focused X-ray beam produced by the graded-crystal monochromator beamline KMC-2. The microprobe has been used in the energy range of 3.5 keV–15 keV. XANES and EXAFS measurements for a large variety of samples were done [3,4].

These measurements required a large energy scan range, which especially for EXAFS exceeds of 1000 eV. Therefore, X-ray polychromatic focusing optics is required to cover the necessary energy range.

Among others, the capillary X-ray optical system with a lateral resolution down to 5 μm has been used at BESSY beamlines for:

- the microfocus XAS measurements on volcano-shaped manganese deposits at the cell wall of the green alga *Chara coralline*. The evolutionary origin of the manganese complex of oxygenic photosynthesis and possible formation of a bicarbonate precursor complex has been investigated [5].
- the investigation of oxidation and migration processes of inorganic compounds in ink corroded manuscripts. A model

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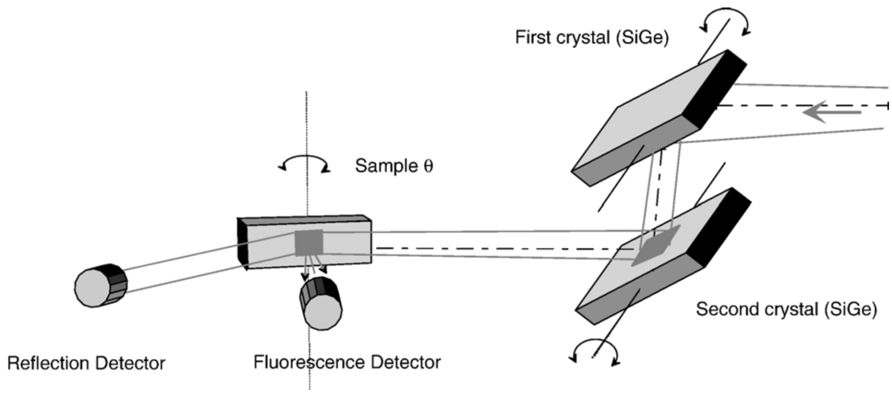


Fig. 1. Experimental setup, top view.

of the ink corrosion mechanism has been suggested based on the experimental results of a combination of μXRF and μXANES methods. The influence of restoration treatment on ink-corroded manuscripts was carefully investigated [6].
- the study of glasses containing industrial waste by means of μXRF mapping and μXANES. An inhomogeneous distribution of Fe was observed in the samples. Also found using μXAS was the alteration of the local environment around the Fe atom, which tends to occupy tetrahedral sites in the glass matrix for the regions with low Fe concentration [7].

X-rays are highly penetrating radiations and, therefore, any information obtained through X-ray-based measurements is averaged over a depth of several microns. However, X-ray-based techniques can be made depth-selective by generating standing waves inside the nanostructure of interest by making use of the phenomenon of total reflection [8,9]. X-ray intensity is localized in the anti-nodal regions, the position of which inside the nanostructure can be varied by varying the angle of incidence. Use of such X-ray standing waves (XSW) in elemental depth profiling or XANES measurements with nanometer depth resolution has been demonstrated [10].

For the first time, angular dependences of the fluorescence yield from a single organic monolayer on a solid substrate modulated by a standing wave in a total external reflection conditions has been measured experimentally. Scanning by standing wave field of a single organic molecule has been done; the depth positions of particular ions in the molecule structure have been determined [11]. In this experiment, a depth resolution on the order of 1–5 nm has been achieved.

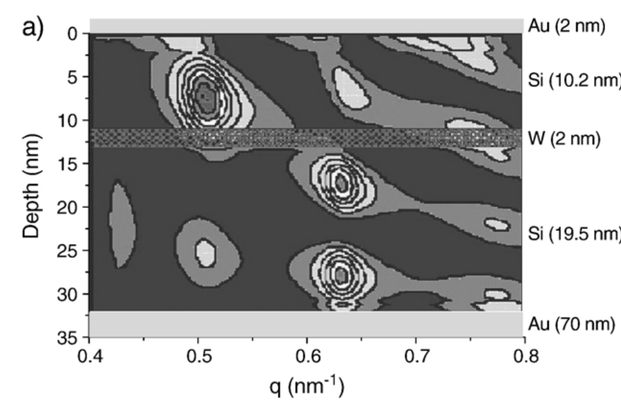
Depth selectivity can further be enhanced by making use of waveguide structures [12,13].

For the first time at BESSY, depth-selective EXAFS studies have been performed. The absorption spectra of Fe and W nanolayers were recorded with in-depth resolution on the order of 1 nm. This method is combining total external reflection standing waveguide mode and EXAFS measurements.

As an example of such measurements done at BESSY beamlines, in the following we present the results of depth resolved tungsten XAFS measurements in a Si/W/Si three-layer embedded in a waveguide structure.

2. Experimental setup

The graded-crystal monochromator beamline KMC-2 was used to set-up of the in-depth nanoprobe EXAFS system [14]. The X-ray beam in the energy range of 10 keV–14 keV was monochromatized by the double-graded-crystal monochromator and collimated in both directions using two slit systems and collimating mirror shown in Fig. 1. The beam size on the sample position was 100 μm horizontal and 700 μm vertical. The beam divergence was obtained on the order of 20 arc sec by the rocking curve measurements of a Si (111) reference crystal. The



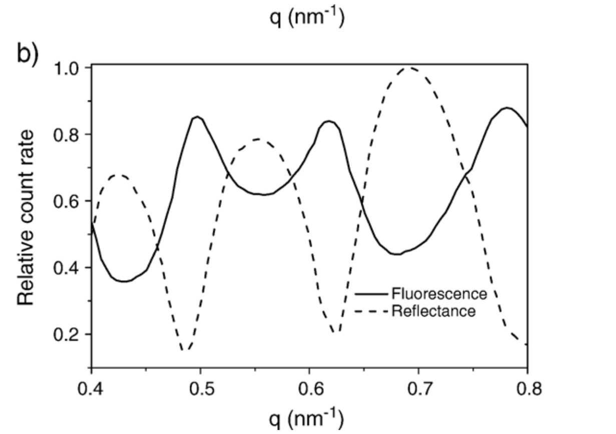


Fig. 2. (a) The multilayer sample structure. The contour plot represents the intensity of X-rays as a function of depth and q. The hatched strip marks the position of W. (b) Normalized intensity curves of reflected radiation and W Lα fluorescence radiation at 10.3 keV incidence energy.