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Imaging X-ray fluorescence microscope with a Wolter-type grazing-incidence mirror

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A Wolter-type grazing-incidence mirror was used as an objective for an imaging X-ray fluorescence microscope. The microscope was constructed at the beamline 6C2 of the Photon Factory. The shortest wavelength used was ~0. 1 nm, which was limited by the grazing-incidence angle of the mirror. To demonstrate the possibility of recording X-ray fluorescence images, several fine grids were used as test specimens. Characteristic X-rays emitted from each specimen could be clearly imaged. Spatial resolution was estimated to be better than 10 μm.

Keywords: imaging; X-ray fluorescence; microscopes; Wolter-type mirrors.

1. Introduction

X-ray fluorescence has been widely used for trace-element analysis for many years. Recent developments of high-brightness electron storage rings have stimulated the construction of various types of X-ray microprobes for X-ray fluorescence microanalysis. Relatively high-energy X-rays are used to produce X-ray fluorescence because the X-ray fluorescence yield becomes larger for heavy elements. Typical X-ray focusing devices are a Kirkpatrick–Baez grazing-incidence mirror (Suzuki & Uchida, 1992), a thick Fresnel (Kamijo *et al.*, 1997) or Bragg–Fresnel zone plate (Erko *et al.*, 1994), and a capillary tube (Bilderback *et al.*, 1994). A new compound refractive lens (Snigirev *et al.*, 1996) has also been reported. All these devices, however, have been used only for producing small spots in the hard X-ray region. As a result, all X-ray fluorescence images were obtained with scanning X-ray microscopes. Owing to the lack of an efficient objective lens for recording amplitudes and phases of X-ray fluorescence, an imaging X-ray fluorescence microscope has not been demonstrated.

In this paper, we propose an imaging X-ray fluorescence microscope with a Wolter-type grazing-incidence mirror and present some preliminary results.

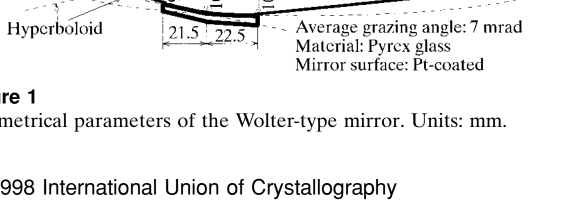

Figure 1
Geometrical parameters of the Wolter-type mirror. Units: mm.

2. Optical systems

Among many optical devices, a Wolter-type grazing-incidence mirror (Wolter, 1952) is one of the most suitable objectives for hard X-ray imaging. Because it has small coma and no chromatic aberration, it can be used for polychromatic X-rays like X-ray fluorescence. In addition, its numerical aperture is not as small as a zone plate, which means that the resolving power is relatively large. Owing to technological difficulties, these factors have not been taken advantage of. Only large Wolter-type mirrors have been successfully used for X-ray astronomy. Recent developments of nanometre fabrication technology, however, enable us to produce small Wolter-type mirrors for hard X-rays.

Fig. 1 shows the parameters of the Wolter-type grazing-incidence mirror we designed and fabricated for hard X-rays. The mirror consists of an axisymmetric hyperboloidal and ellipsoidal mirror. It is known that X-rays can be totally reflected at a grazing incidence angle smaller than the critical angle (θ_c) which is given by

$$\theta_c = 1.6 \times 10^{-2} \, \lambda \rho^{1/2}, \tag{1}$$

where λ is the wavelength in nanometres and ρ is the density of the reflection surface in g cm^{-3}. With a platinum-coated mirror, 0.1 nm X-rays can be reflected at a grazing angle of 7 mrad. The mirror optics satisfy two basic imaging conditions: one is Rayleigh's quarter-wavelength rule and the other is Abbe's sine condition. Ray-tracing calculations show that the field of view is about 0.6 mm or 0.2 mm in diameter, assuming that the spatial resolution is better than 10 μm or 1 μm, respectively.

We fabricated a small Wolter-type mirror by using the glass replica technique which had been developed to produce the same kind of mirrors for soft X-ray microscopes (Aoki *et al.*, 1992). First

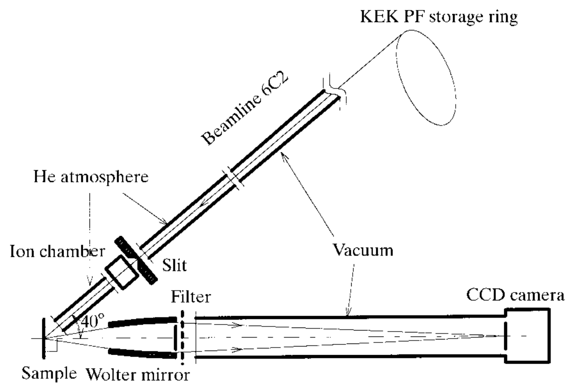

Figure 2
Schematic diagram of the experimental arrangement.

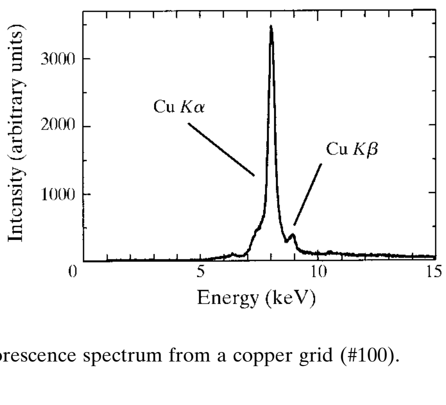

Figure 3
X-ray fluorescence spectrum from a copper grid (#100).

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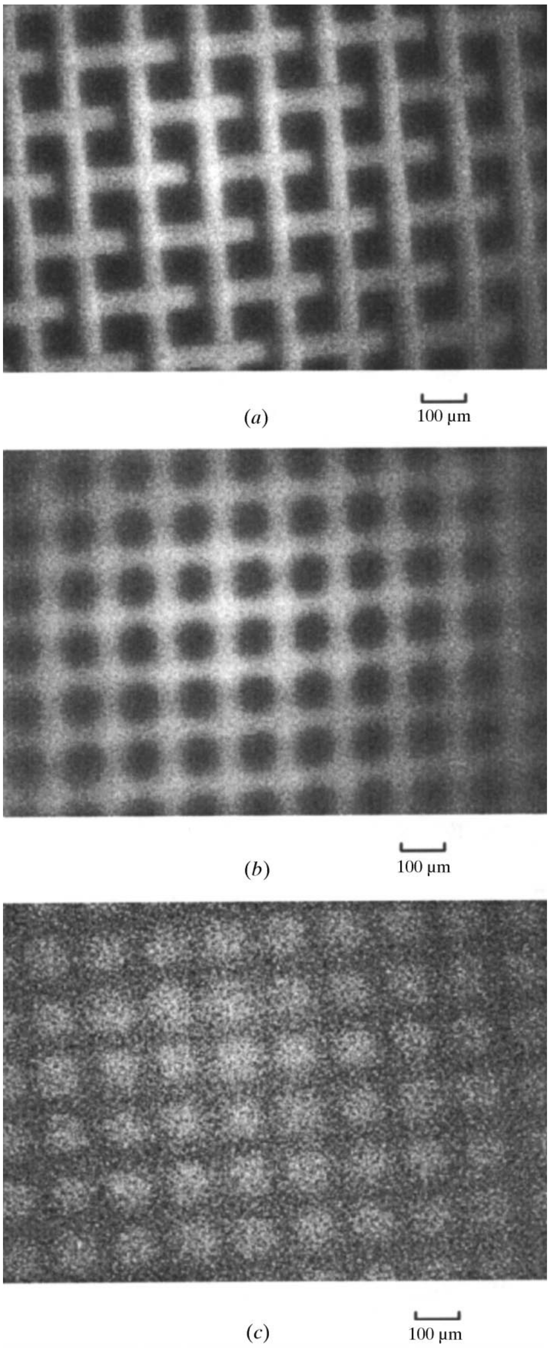

Figure 4
X-ray fluorescence images. (*a*) Stainless-steel netted grid (#200), (*b*) stainless-steel grid sheet (#200), (*c*) chromium 50 nm-thick pattern (#200).

we polished the master mandrel (tungsten carbide) and then made a glass replica (Pyrex glass). The axial surface deviation from the design parameter was better than 1 μm and the surface roughness was about 1 nm r.m.s. The inner surface of the glass replica was coated with tens of nanometres of platinum film. Details of the fabrication processes have been described in our previous paper (Onuki *et al.*, 1992).

We built an imaging X-ray fluorescence microscope by using this mirror at the Photon Factory. A schematic diagram of the experimental arrangement at beamline 6C2 is shown in Fig. 2. No monochromator was used because the mirror can reflect broad-band X-rays. Two 0.2 mm-thick and two 0.3 mm-thick beryllium plates were put in the beamline as the vacuum windows. These plates worked as high-pass filters so that X-rays above 4 keV could be incident on the samples. The surface of the sample was inclined at 50° to the incident beam and vertical to the optical axis of the objective mirror. The area detector was an X-ray sensitive CCD camera (TI, TC-215). The number of pixels was 1000×1018 and the area of one pixel was $12\ \mu m \times 12\ \mu m$. The detector was cooled by a Peltier cooler to below 253 K to reduce the dark current. The X-ray path between the mirror and the detector was evacuated.

3. Results

Several test samples were imaged to reveal the performance of the system. Fig. 3 shows the X-ray fluorescence spectrum of a copper grid (#100) obtained at the detector plane with a Ge SSD. High-energy X-rays of about 15 keV were observed. Fig. 4(*a*) shows the X-ray fluorescence image of a stainless-steel netted grid (#200). The exposure time was 50 s. The overlapped parts of the grid produced the shadow of the wire because of the inclined irradiation of the incident X-rays. Fig. 4(*b*) shows the X-ray fluorescence image of the stainless-steel grid sheet (#200). The exposure time was 90 s. There was no shadow of the grid. Compared with those in Fig. 4(*a*), the corners of the grid in Fig. 4(*b*) are round, which are not attributed to the blur of the image but their original patterns. Fig. 4(*c*) shows the X-ray fluorescence image of 50 nm-thick chromium square patterns (#200) coated on a silicon wafer. The exposure time was 30 min.

4. Conclusions

An imaging X-ray fluorescence microscope has been demonstrated with a Wolter-type grazing-incidence mirror for the first time. Although the experiment could not show the distribution of a specific element, the use of an energy-tunable monochromator or an energy-resolvable area detector can solve this problem. The microscope could image very thin film patterns, though the spatial resolution was not so good. For a further improvement of the spatial resolution, a new mirror-fabrication technique must be developed. The use of a brighter X-ray source is also recommended.

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