Generation of an X-ray microbeam for spectromicroscopy at SPring-8 BL39XU

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A pair of elliptical mirrors (KB mirror) was designed and fabricated to realize an energy tunable x-ray microbeam for spectro-microscopy at SPring-8 BL39XU. As is commonly recognized, the obtainable beam size with the aspherical total reflection mirrors is strongly affected with the slope error of the mirror. Considering that the extremely high brilliance of the undulator radiation from the SPring-8, the small mirror size and the small mirror-to-focus distance were employed to minimize effects of the slope error. Preliminary evaluation of the KB mirror was carried out using 10 keV monochromatized undulator radiation. Alignment of the mirror was assisted by the beam monitor system composed of a scintillator and a CCD, and the beam size less than 5 μm can be easily achieved even when the source was fully used. The beam size obtained with this experiment was 2 x 4 μm^2 with the photon flux of 1 x 10^{10} photons/s. Smaller beam size may be expected with the use of intermediate slits. Characterization of trace elements with the spatial resolution will be carried out by using x-ray fluorescence (XRF) analysis and x-ray absorption fine structure (XAFS) measurements with XRF yield method.

Keywords: KB mirror; elliptical mirror; spectromicroscopy; SPring-8; trace characterization.

1. Introduction

Generation of an x-ray microbeam has received great attention among the third generation synchrotron light sources, and the importance of the energy tunable x-ray microbeam for the spectromicroscopy is now widely recognized. To realize trace characterization with spatial resolution, a hard x-ray microprobe system was developed at SPring-8 on BL39XU (Hayakawa et al., 1998). X-ray fluorescence (XRF) analysis, x-ray absorption fine structure (XAFS) measurements and high resolution x-ray fluorescence spectroscopy using a crystal spectrometer have been carried out with the spatial resolution between 10 μm to 200 μm by employing undulator radiation defined by a pinhole or slits (Hayakawa et al., 2000). To realize better spatial resolution, an x-ray focusing system has been designed.

Though fairly good spatial resolutions are reported with zone plates (Kamijo et al., 1997; Yun et al., 1999), it is commonly known that the stabilization of the beam position on the sample is difficult during the energy scan because the zone plate should be translated along with the optical axis to maintain the focusing condition (Warwick et al., 1998). In the sense of the energy tunability, elliptical figured total reflection mirrors are ideal when combined with the fixed exit monochromator. However, the realization of the sub-micron beam is difficult because the requirements for the slope error are so severe (Susini, 1995). One of the ways to overcome with this problem is to use bent mirror system to produce elliptical figures (Padmore et al., 1996; Yang et al., 1995) because the smaller slope error can be achieved with the flat mirror. Recently a differential deposition technique was employed to produce the elliptical figure from the cylindrical figure, and the beam spot of 0.37 x 0.53 μm^2 was reported (Ice et al., 2000). Another practical way is to employ small mirror-to-focus distance to reduce the effect of the slope error though the smaller mirror size results in the smaller acceptance by the mirror. Considering that the extremely high brilliance of the undulator radiation from the SPring-8, the crossed elliptical mirrors (KB mirror) with the small aperture of 160 μm square was adapted. This paper describe the parameters and preliminary experimental results with the KB mirror (Kirkpatrick & Baez, 1948) system designed for spectromicroscopy in the hard x-ray region.

2. Kirkpatrick and Baez (KB) mirror system

Fig. 1. shows a schematic layout of the KB mirror and its dimensions. The M1 mirror focuses vertical divergence and the M2 mirror focuses horizontal divergence from the light source. The designed horizontal and vertical source size (FWHM) is 890 x 78 μm^2. However, effective or real source size itself is still under investigation. The demagnification of the mirrors are quite large, and the practical beam size at the focus may be determined by the slope error. The additional blurring caused by the slope error can be estimated with the following expression.

$$s = 2L\Delta\theta \qquad (1)$$

where L is the distance from the center of the mirror to the focus (the image distance), $\Delta\theta$ is the slope error. Considering that the practical slope error of around 5 μrad, L should be less than 100 mm to maintain the beam size less than 1 μm. To satisfy this condition, the dimension of the mirror was limited to 40 mm in length. To cover x-rays up to 20 keV, the mirror surface is coated with Rh and the central glancing angle was set to be 4 mrad. Therefore, the resultant aperture of the KB mirror is approximately 160 μm square when the mirror is fully illuminated.

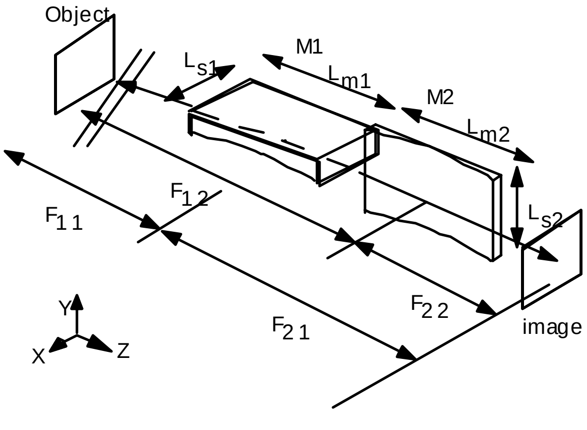

Figure 1
A schematic layout of the Kirkpatrick and Baez (KB) mirror system.
The object and image distances are labeled F_1 and F_2. Mirror sizes in meridional and sagittal directions are labeled L_m and L_s.
M1 : F_{11}=49950; F_{21}=90; L_{m1}=40; L_{s1}=30
M2 : F_{12}=50000; F_{22}=90; L_{m2}=40; L_{s2}=30

Two mirrors are made of fused quartz because they are used for monochromatized undulator radiation and the heat load is not so severe. Figures and surfaces of the mirrors were investigated at each step of the mirror fabrication process. After the machining process, 10 - 20 nm thick Cr was coated to the mirror surface preceding to the coating of 100 nm thick Rh. The evaluated surface roughness (rms) of the M1 and M2 mirrors are 0.684 nm and 0.771 nm, and the evaluated slope errors (rms) of these mirrors are 3.16 μrad and 5.25 μrad, respectively. Utilizing equation (1) and the image distances listed in Fig.1, the additional blurring of 0.57 (vertical) x 0.42 (horizontal) μm^2 can be predicted

3. Experimental

Experiments were carried out at SPring-8 on the BL39XU. Fig. 2 shows experimental setup. Though the KB mirror and mirror alignment mechanism are designed to be used inside the vacuum chamber of the x-ray microprobe, the preliminary evaluation of the mirror performance was carried out on the optical bench inside the experimental hutch. The x-ray energy was tuned to 10 keV with the undulator gap of 14.471 mm. The size of the front-end-slit placed 29 m from the source was 0.5 x 0.5 mm^2. A standard water cooled Si (111) double crystal monochromator was used for monochromatization, and the higher order harmonics of the undulator radiation were rejected by using a Pt coated flat deflection mirror of the beamline. To align the optical elements, the x-ray beam was monitored with a peltier cooled digital CCD coupled with a fast plastic scintillator and a lens system. A commercial NE102A plate (25 x 25 mm^2) of 0.3 mm thick was used as the scintillator, and it was placed approximately 100 mm downstream the focus (sample position) of the KB mirror. X-ray beam was converted into the visible light of around 420 nm, and the image can be observed on the CCD (KX1E, Apogee) with the unit magnification. A pixel size of the CCD is 20 μm square, and the number of effective pixels are 768 and 512 in horizontal and vertical direction, respectively. To avoid direct exposure of x-rays to the CCD, a plane deflection mirror is installed to the optical axis of the lens system. The deflection mirror can be switched to the off-axis position when the x-ray beam intensity is monitored by the ionization chamber.

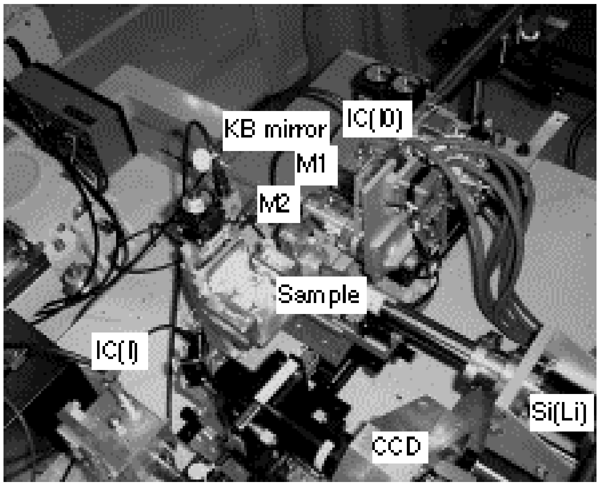

Figure 2
Photograph of the experimental setup for preliminary evaluation of the KB mirror system at SPring-8 BL39XU.

Fig. 3 shows images on the CCD obtained during the alignments. The incident beam was defined by the slits of 100 μm square, and the difference of the beam position between the direct beam and the reflected beam was observed at the position 100 mm downstream the focus. Glancing angles of the M1 and M2 mirrors were adjusted to satisfy the difference of positions between the direct and reflected beams as designed, and the beam intensity was optimized with the slight translation of the each mirror. Initial alignment of the KB mirror can be easily finished with the procedure mentioned above, and it takes within two hours.

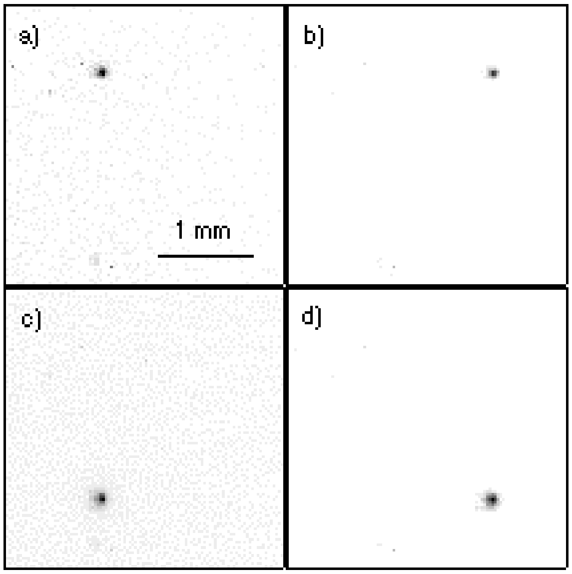

Figure 3
Images on the beam monitor placed 100 mm downstream the focus of the KB mirror. a) direct beam, b) horizontally focused beam with M2, c) vertically focused beam with M1 and d) focused beam in both directions.

4. Performance of the KB mirror system

Fig. 4 shows a scanning transmission x-ray image of a #200 grid made of stainless steel. An area of 200 μm square was scanned with the 2.5 μm step. Line profiles and their derivatives in horizontal and vertical direction measured at the marked positions are displayed beside the image. Owing to the rather huge step of the scan it is difficult to estimate the beam size precisely. However, it is easily understood that the beam sizes are less than 5 μm in both directions.

Owing to the limited beam time, the focused beam size was evaluated without the further optimization of the alignment. Edge scans of a 100 nm thick Cr evaporated resolution test pattern were carried out by monitoring Cr XRF signals. Scans were carried out at the several positions along with the optical axis to find the beam west. The estimated beam size was around 2 (horizontal) x 4 (vertical) μm^2 with the photon flux of 1 x 10^{10} photons/s. In this experiment the light source was directly imaged by the KB mirror. The beam size less than 1 μm can be expected with the use of intermediate slits to reduce the effective source size. Utilizing the energy tunable x-ray microbeam, characterization of trace elements with the spatial resolution will be carried out by using various spectroscopic techniques.

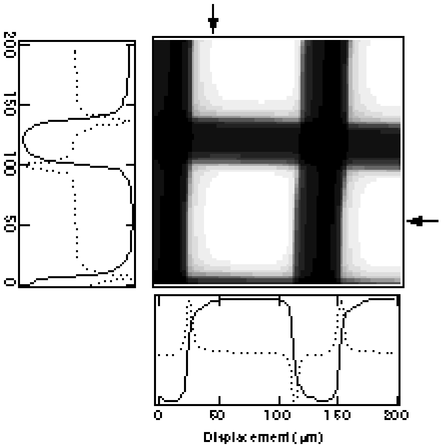

Figure 4
A scanning transmission x-ray micrograph of a stainless steel grid of #200. Cross-sectional profiles (solid lines) and their derivatives (broken lines) in horizontal and vertical directions are shown beside the image.

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References

Hayakawa, S., Goto, S., Shoji, T., Yamada, E., and Gohshi, Y. (1998) J. Synchrotron Rad. **5,** 1114-1116.

Hayakawa, S., Hirokawa, T., Gohshi, Y., Suzuki, M. and Goto, S. (2000). Proc. of XRM 99 (AIP, 2000) 92-97.

Ice, G. E., Chung, J.-S., Tischler, J. Z., Lunt, A. and Assoufid, L. (2000) Rev. Sci. Instrum. **71**, 2635-2639.

Kamijo, N., Tamura, S., Suzuki, Y., Kihara, H. (1997) Rev. Sci. Instrum. **68**, 14-16..

Kirkpatrick, P. and Baez, A. V. (1948). J. Opt. Soc. Am. **38**, 766-774.

Padmore, H. A., Howell, M. R., Irick, S., Renner, T., Sandler, R. and Koo, Y.-M. (1996) SPIE 2856, 145.

Susini, J. (1995) Optical Eng. **34**, 361-376.

Warwick, T., Franck, K., Kortright, J. B., Meigs, G., Moronne, M., Myneni, S., Rotenberg, E., Seal, S., Steele, W. F., Ade, H., Garcia, A., Cerasari, S., Denlinger, J., Hayakawa, S., Hitchcock, A. P., Tyliszczak, T., Kikuma, J., Rightor, E. G., Shin, H.-J. and Tonner, B. P. (1998). Rev. Sci. Instrum. **69**, 2964-2973.

Yang, B. X., Rivers, M., Schildkamp, W. and Eng, P. J. (1995). Rev. Sci. Instrum. **66**, 2278-2280.

Yun, W., Lai, B., Cai, Z. Maser, J., Legnini, D., Gluskin, E., Chen, Z., Krasnoperova, A. A., Vladimirsky, Y., Cerrina, F., Fabrizio E. Di. and Gentili, M. (1999) Rev. Sci. Instrum. **70**, 2238-2241.

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