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Author(s)	Matsuyama, S.; Emi, Y.; Kino, H. et al.
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Development of achromatic full-field X-ray microscopy with compact imaging mirror system

S. Matsuyama^{*a}, Y. Emi^a, H. Kino^a, Y. Sano^a, Y. Kohmura^b, K. Tamasaku^b,
M. Yabashi^b, T. Ishikawa^b, K. Yamauchi^{a,c}

^a Department of Precision Science and Technology, Graduate School of Engineering,
Osaka University, 2-1 Yamada-oka, Suita, Osaka 565-0871, Japan

^b RIKEN/SPring-8, 1-1-1 Kouto, Sayo-cho, Sayo-gun, Hyogo 679-5148, Japan

^c Research Center for Ultra-Precision Science and Technology, Graduate School of Engineering,
Osaka University, 2-1 Yamada-oka, Suita, Osaka 565-0871, Japan

ABSTRACT

Compact advanced Kirkpatrick–Baez optics are used to construct a microscope that is easy to align and robust against vibrations and thermal drifts. The entire length of the imaging mirror system is 286 mm, which is 34% shorter than the previous model. A spatial resolution test is performed in which magnified bright-field images of a pattern are taken with an X-ray camera at an energy of 10 keV at the BL29XUL beamline of SPring-8. A line-and-space pattern having a 50-nm width could be resolved, although the image contrast is low.

Keywords: advanced Kirkpatrick–Baez optics, Wolter mirror, full-field X-ray microscopy, X-ray mirror

1. INTRODUCTION

A full-field X-ray microscope forms magnified images of samples illuminated by X-rays. It generally consists of illumination optics, imaging optics, and an X-ray camera. It provides a level of detail that cannot be visualized by other microscopes. One advantage is its higher spatial resolution compared to optical microscopes: theoretically subnanometer and in practice 20–100 nm owing to the short wavelength^{1,2,3}. In addition, its high penetration power enables observation of thick samples and the interior of materials. Samples can also be immersed in aqueous solutions or gaseous atmospheres owing to the large penetration.

The most common imaging optical element used in a full-field X-ray microscope is a Fresnel zone plate. Commercially available devices can image X-rays with 50-nm resolution. Many important advances using zone plates have been reported^{1,2,4,5}. However, they are subject to chromatic aberration, so that polychromatic X-rays do not get focused to the same point. As a consequence, nonmonochromatic X-rays and X-ray fluorescence emitted by an object cannot be handled. To our knowledge, there has been no development of imaging optics that do not suffer from chromatic aberration and that can achieve sub-50-nm resolution. Such achromatic high-resolution imaging would enable nanospectroscopy in the X-ray region.

In this paper, the use of advanced Kirkpatrick–Baez (KB) optics⁶ is proposed, consisting of four total-reflection mirrors. Total reflection avoids chromatic aberration and the four-mirror system can have a wide field of view (FOV) because it satisfies the Abbe sine condition. In addition, it has great potential to realize high-resolution x-ray microscopy, because it consists of nearly planar mirrors that can be fabricated with a figure accuracy of one nanometer order using existing techniques.

Recently, advanced KB optics have been developed that consist of ultraprecise total-reflection mirrors finished by elastic emission machining (EEM)⁷. A demagnification imaging test showed that it has a resolution of 50 nm at 11.5 keV^{8,9}, which is near the diffraction limit. Bright-field images with a 150-nm resolution at 10 keV in the magnification setup were successfully obtained¹⁰.

*matsuyama@prec.eng.osaka-u.ac.jp; phone/fax +81-6-6879-7286; <http://www.dma.jim.osaka-u.ac.jp/view?l=en&u=4001>

In the research described here, compact advanced KB optics are designed and fabricated. Reduction in the size of the advanced KB optics would make a microscope less prone to vibrations and temperature fluctuations. Also, it would be easier to precisely align the mirrors because the calibration of the required autocollimators and laser displacement meters is improved when the length is small. Finally, compact mirrors are cheaper to fabricate. In addition, illumination optics consisting of elliptical and plane mirrors are developed. Using these imaging and illumination optics, a magnification imaging test is performed to characterize the minimum spatial resolution at SPring-8. The resulting images are quantitatively analyzed in terms of a modulation transfer function (MTF).

2. ADVANCED KIRKPATRICK–BAEZ OPTICS

New advanced KB optics are designed with the following specifications. The mirrors are to have a radius of curvature greater than 20 m, a reflectivity greater than 87% at 10 keV, and a length of between 30 and 120 mm. The microscope is to have a magnification of at least 200, a working distance (WD) of 35 mm, and a numerical aperture exceeding 1.2×10^{-3} . The curvature of the mirrors is constrained by the metrology system. A steeply curved shape cannot be measured using a relative angle determinable stitching interferometer (RADSI)¹¹. The above numerical aperture is needed to achieve sub-50-nm resolution. The layout of the four mirrors, and their shapes and slopes are shown in Figs. 1 and 2, respectively. Their detailed parameters are listed in Table 1. The system has a length of 286 mm, which is 34% shorter than the previously constructed one⁸ with a length of 435 mm. That previous system had a nested arrangement of optics, with two elliptical mirrors aligned downstream of two hyperbolic mirrors, so that mismatches in the magnification in the vertical and horizontal directions could be canceled. In the new system, a tandem arrangement is employed, in which the vertical and horizontal imaging mirrors are aligned in series, as shown in Fig. 1. The arrangement has an advantage that a mirror manipulator used to align the four mirrors precisely can be simple. To estimate the performance of the design, the point spread function (PSF) and FOV characteristics were calculated using a wave optical simulator^{9,12,13}. Unlike ray-tracing simulators, it can handle nearly-diffraction-limited situations. The results are presented in Fig. 3, confirming that sub-50-nm resolution is possible with a wide FOV of 13×20 (V \times H) μm^2 .

The four mirrors were figured on quartz glass substrates by numerically controlled EEM with peak-to-valley figure accuracies of better than 2 nm, as graphed in Fig. 4. The figure errors were measured using a microstitching interferometer (MSI)¹⁴ and a RADSI. Surface characterization using a phase-shift interference microscope (Zygo NewView 200CHR) confirmed that they have a root mean square roughness of better than 0.2 nm over an area of $64 \times 48 \mu\text{m}^2$, which is sufficiently small that any reduction in the reflectivity caused by roughness can be neglected. After figuring their shapes, the mirrors were coated with a thin chrome binder layer and a 80-nm-thick platinum layer by magnetron sputtering.

Table 1. Design parameters of the mirror.

	M1	M2	M3	M4
Shape	Hyperbolic (for vertical imaging)	Elliptical (for vertical imaging)	Hyperbolic (for horizontal imaging)	Elliptical (for horizontal imaging)
a^* (m)	20.51×10^{-3}	22.57	68.57×10^{-3}	22.66
b^* (m)	0.3111×10^{-3}	13.34×10^{-3}	1.044×10^{-3}	23.60×10^{-3}
Incident glancing angle (mrad)**	4.6	5.5	4.8	5.5
Distance from object (mm)**	50	90	161	268
Mirror length (mm)	30	38	92	111
Magnification factor	658		214	
Numerical aperture	1.51×10^{-3}		1.46×10^{-3}	

* Ellipse $x^2/a^2 + y^2/b^2 = 1$ or hyperbola $x^2/a^2 - y^2/b^2 = 1$.

** At the center of the mirror.

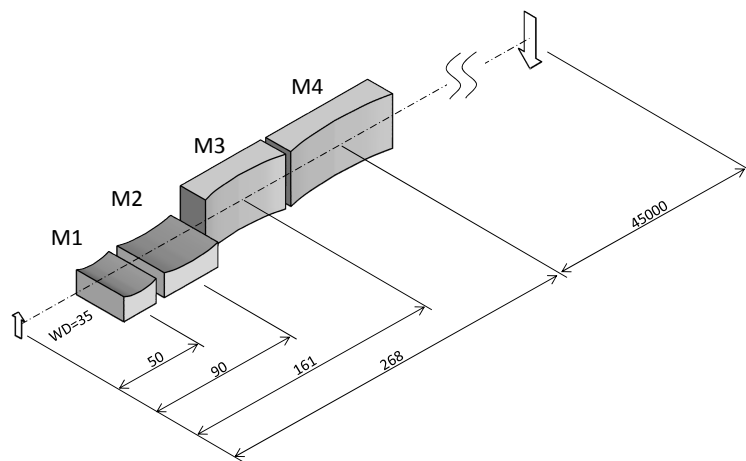


Figure 1. Schematic of the advanced KB optics. The distances are in millimeters.

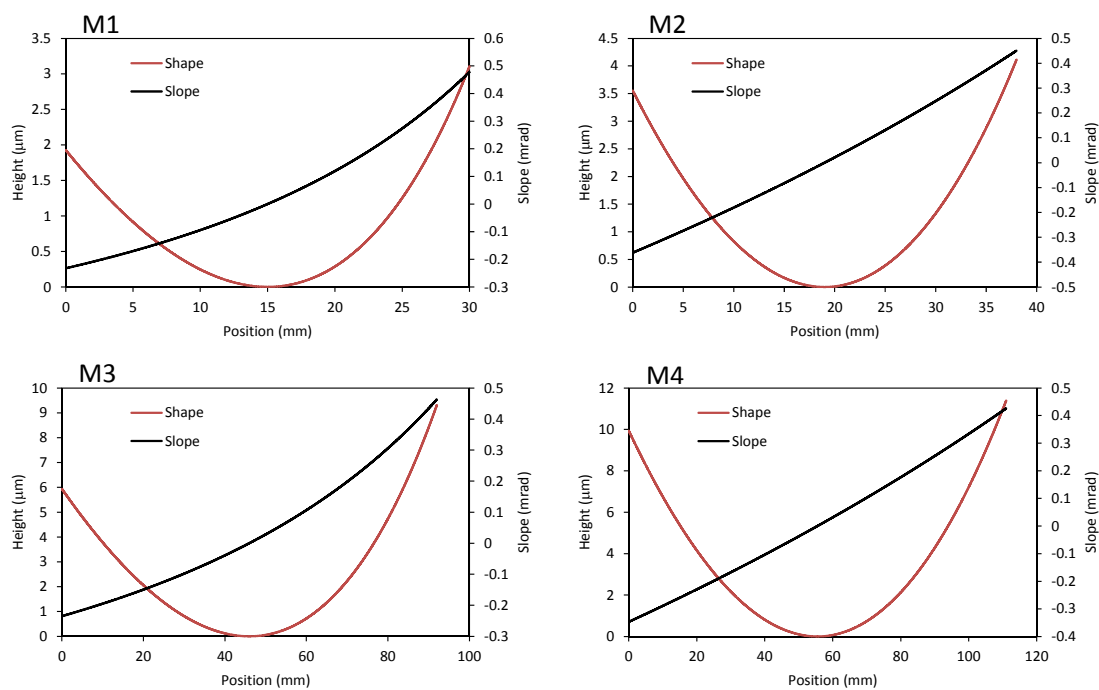


Figure 2. Designed mirror shapes and slopes.

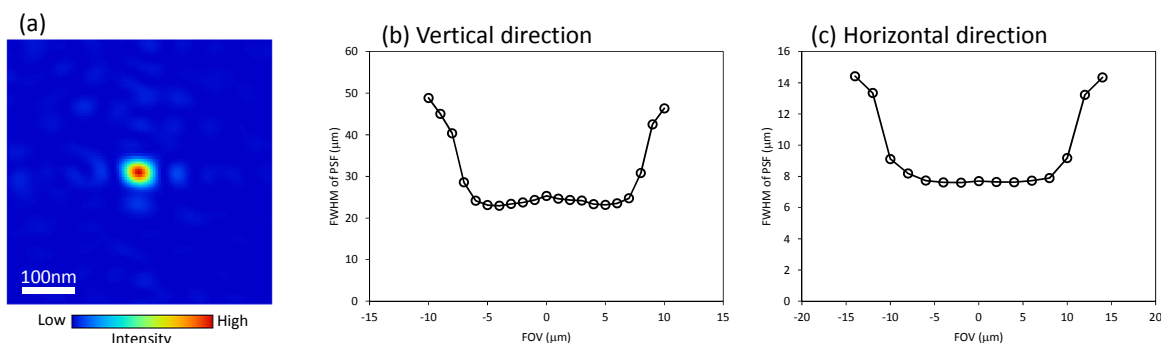


Figure 3. (a) Point spread function and (b)–(c) field of view characteristics, as calculated using the wave optical simulator at 11.5 keV. The PSF is computed by demagnifying a point source with the imaging optics. The full width at half maximum (FWHM) is 36×44 ($V \times H$) nm^2 . The actual PSF at the image plane is obtained by multiplying the calculated PSF by the magnification. In panels (b) and (c), the vertical axis plots the FWHM of the PSF observed at the image plane, and the horizontal axis plots the FOV at the object plane.

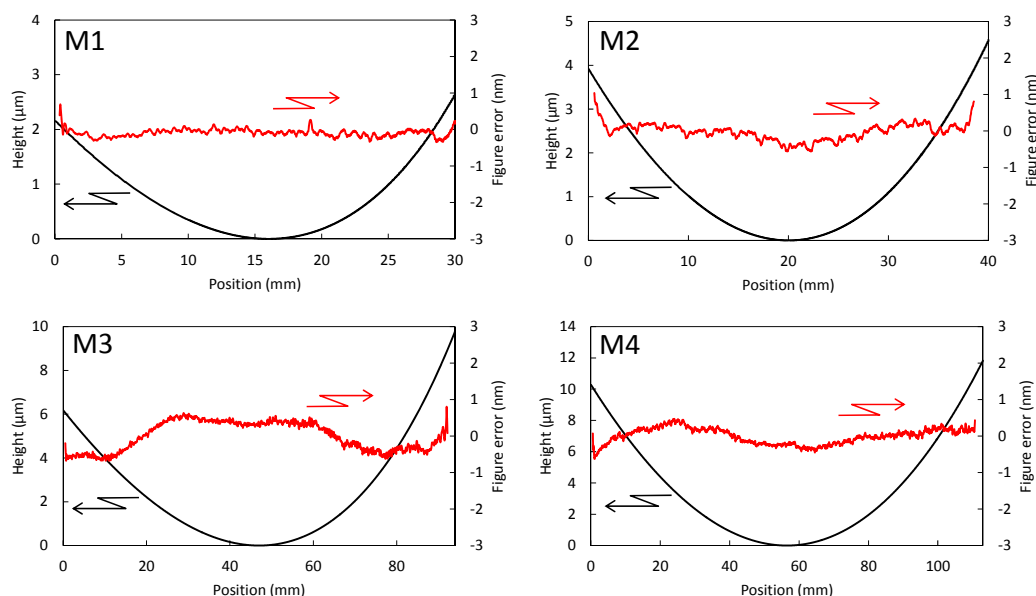


Figure 4. Shapes and residual figure errors of the fabricated mirrors.

3. EXPERIMENTAL TEST OF THE OPTICS

An imaging test was performed at BL29XUL¹⁵ of SPring-8 at an X-ray energy of 10 keV. This beamline has four experimental hutches at different distances from the undulator ranging between 50 m and 1 km. The first and third hutches were selected for the imaging optics and X-ray camera, because high magnifications can be obtained using a distance of ~ 45 m. The hutches are connected by a long vacuum duct. The experimental setup is sketched in Fig. 5. The X-rays were monochromatized using a double crystal monochromator. In the most upstream area of the first hutch, a homemade diffuser consisting of fine sandpaper mounted on a motorized rotating table was positioned to decrease the coherence of the X-rays. Illumination optics composed of two elliptical mirrors aligned perpendicular to each other were placed before the samples. They can focus X-rays down to ~ 80 μm at the sample by detuning the focal length by 16 mm to enlarge the illuminated area. The parameters regarding the mirrors are summarized in Table 2. A commercial line-and-space pattern (XRESO-50, NTT Advanced Technology Corporation) was used to evaluate the spatial resolution. The pattern is made of tantalum and has a minimum line width of 50 nm and a thickness of 200 nm. An optical microscope

was used to position the sample. The X-rays transmitted by the sample were collected by the imaging optics. However, X-rays reflected from the setup do not propagate to the camera in the third experimental hutch because the vacuum duct between them is too long. Instead, two plane mirrors were set upstream of the illumination optics to direct X-rays through the duct. An X-ray camera system (AA20MOD, Hamamatsu Photonics) consisting of a thin scintillator (having a thickness of 10 μm), a relay lens ($\times 2$), and a CCD (with a pixel size of 6.45 μm) was used to record the images. The advanced KB optics were aligned offline using autocollimators and laser displacement meters. The incident angles for all mirrors (except for the relative angles between the advanced KB optics) were adjusted to an accuracy of 0.3 mrad.

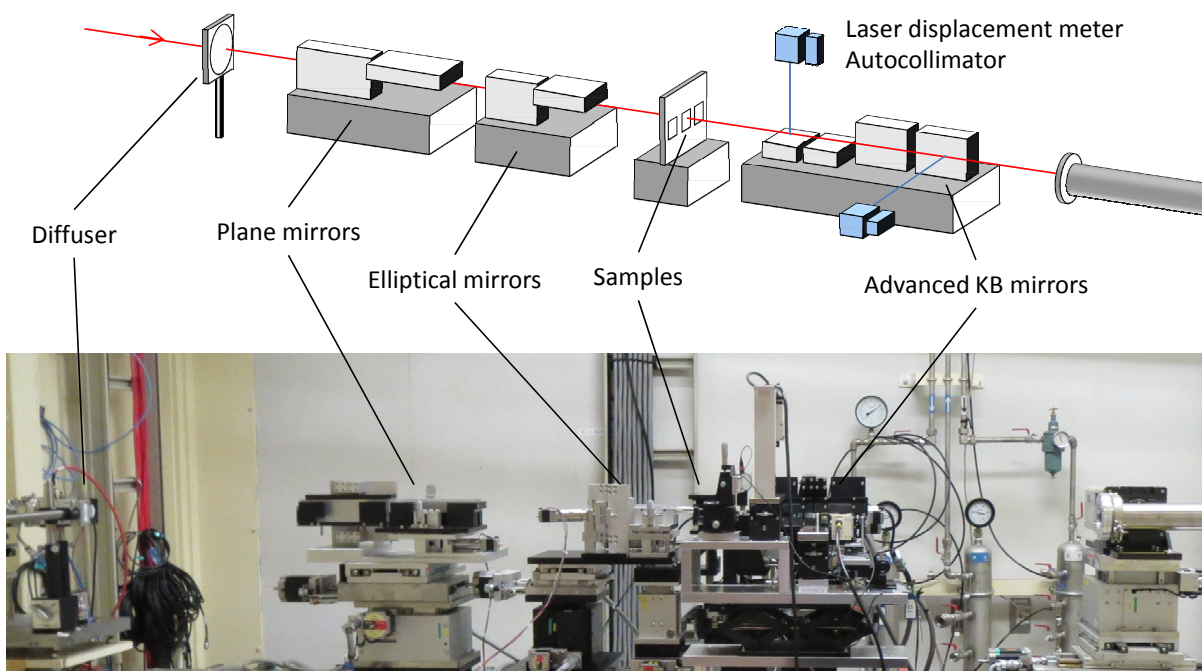


Figure 5. Experimental setup at the first beamline hutch.

Table 2. Mirror parameters for the illumination optics.

Shape	Planar (horizontal)	Planar (vertical)	Elliptical (horizontal)	Elliptical (vertical)
Incident glancing angle (mrad)*	2.97	2.75	7.8	7.4
Distance from object (mm)*	850	645	255	155
Mirror length (mm)	200	200	100	90

* At the center of the mirror.

4. RESULTS AND DISCUSSION

Figure 6 presents images of the line-and-space pattern. They are normalized to the background collected without the object. The differing line widths in the vertical and horizontal directions result from the mismatched magnifications. Patterns having widths of 50 and 100 nm in the vertical and horizontal directions, respectively, are visible, suggesting a spatial resolution of approximately those sizes. The modulation transfer function (MTF) was evaluated. The intensity modulation is defined as $(I_H - I_L)/(I_H + I_L)$ where I_H and I_L are the intensities in the regions of the spaces and lines on the

pattern, respectively. The resulting raw modulation was normalized by the ideal modulation of 0.04 calculated from the thickness of the pattern. The normalized modulation is plotted against the half-period (i.e., the line width) of the pattern in the right-hand panel of Fig. 6. The modulation corresponding to 50 nm in the horizontal direction was estimated to be zero because that pattern was not visible.

The vertical direction has better resolution than the horizontal direction because the focus tuning was insufficient. Because this is a preliminary experiment, focus tuning was not separately performed in the vertical and horizontal directions in order to shorten the alignment time. As a result, the focal length in horizontal direction is ~ 1 mm shorter than ideal. Also, the horizontal direction has a smaller magnification than the vertical direction due to the long focal length. The camera may degrade the resolution of the microscope because the FWHM of the PSF at the object plane exceeds 50 nm, taking into account blurring of approximately $10\ \mu\text{m}$ caused by the camera. To simultaneously achieve sub-50-nm resolution and good contrast in the horizontal direction, a higher resolution camera should be used. In addition, the contrast in the vertical direction is slightly smaller than ideal. A slight vertical defocus is thus suspected.

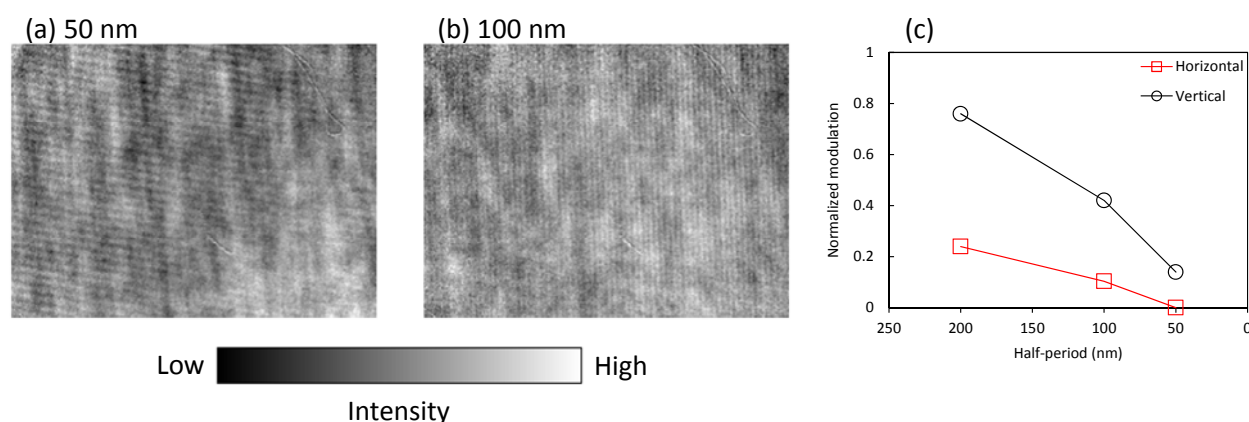


Figure 6. (a)–(b) X-ray images of 50-nm and 100-nm line-and-space patterns. Exposure time: (a) 10 min, (b) 5 min. (c) Measured modulation transfer function.

5. SUMMARY AND OUTLOOK

A full-field X-ray microscope has been developed using advanced KB optics designed to be 34% shorter than in the previous system. A preliminary imaging test at 10 keV indicates that the resolution is ~ 50 nm and ~ 100 nm in the vertical and horizontal directions, respectively. However, ideal focus tuning was not performed. Consequently, the spatial resolution in the horizontal direction did not quite reach the design goal of 50 nm and the contrast in the images was somewhat lower than predicted. The focus tuning in both directions will be optimized in a next experiment.

An achromatic full-field X-ray fluorescence microscope with sub-50-nm resolution would be effective in detecting polychromatic X-ray emissions over a wide field of view. Such a microscope would become a powerful tool for observing elemental distributions in samples.

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