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Rotated-inclined focusing monochromator with simultaneous tuning of asymmetry factor and radius of curvature over a wide wavelength range

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A new single-crystal focusing monochromator for protein crystallography has been developed. In order to achieve simultaneous tuning of the beam demagnification rate and the radius of curvature for focusing over a wide wavelength range, the surface of an inclined monochromator was cylindrically bent. The monochromator is able to tune these two parameters simultaneously with a single rotation of the crystal about the azimuthal axis. A new monochromator incorporating this idea has been developed for beamline BL6B at the Photon Factory. The monochromator was designed for use in the wavelength range 0.87–1.90 Å, and beam focusing was tested at wavelengths of 1.04, 1.38 and 1.74 Å.

Keywords: focusing monochromators; protein crystallography.

1. Introduction

Many beamlines for protein crystallography use single bent asymmetric-cut silicon or germanium crystals to achieve a focused monochromatic beam (Lemonnier *et al.*, 1978). The demagnification rate of a beam passing through an asymmetric-cut crystal monochromator depends on the angle between the beam and the crystal surface with the asymmetry factor, b, given by

$$b = \sin(\theta_B + \alpha)/\sin(\theta_B - \alpha), \qquad (1)$$

where θ_B is the Bragg angle and α is the angle between the Bragg plane and the crystal surface (Kohra *et al.*, 1978). If one uses a conventional single bent asymmetric-cut monochromator, the asymmetry factor or beam demagnification rate alters as shown in (1) with θ_B when the crystal is rotated to change wavelength. The ideal asymmetry factor, b, for a monochromator under the Guinier focusing condition should be

$$b = p/q, \qquad (2)$$

where p and q are the distance from the light source to the monochromator and from the monochromator to a focal point, respectively. For this reason, several monochromator crystals of different asymmetric angle, α, are usually necessary to cover a wide wavelength range. A rotated-inclined, or rotated Fankuchen, monochromator can cover a wide wavelength range keeping the beam demagnification rate constant because it is able to change the asymmetric angle by rotation about an azimuthal axis [see equation (8) below]. However, such a monochromator has no focusing capability. An asymmetrically cut focusing monochromator should be bent to the radius of curvature, R, given by

$$2/R = \sin(\theta_B + \alpha)/p + \sin(\theta_B - \alpha)/q, \qquad (3)$$

if the monochromator is used under the Guinier condition. Thus we have conceived a new design in which the surface of a rotated-inclined monochromator can be variably bent into a cylindrical figure.

In this paper, we describe the newly developed monochromator which allows tuning of the asymmetry factor, or beam demagnification rate, and radius of curvature of the crystal simultaneously through rotation about the azimuthal angle only.

2. Theoretical considerations

In order to achieve tunability of the two parameters, namely asymmetry angle, α, and radius of curvature, R, simultaneously, we have improved a rotated-inclined monochromator to have a focusing capability.

As shown in Fig. 1, the origin of the azimuthal angle, φ, is defined as the direction which gives the usual asymmetric angle, α_0, of a Fankuchen monochromator, and the offset angle, ψ, is the angle between the slope of the inclined surface of the asymmetrically cut crystal and the axial direction of the cylinder. This offset angle is necessary in order to make possible the simultaneous tuning of the two parameters with only a φ-axis rotation.

The radius of curvature, R, of the rotated cylindrical surface at any φ can be calculated as follows. If a cylinder of radius R_0 is cut by a plane at angle φ as in Fig. 2(a), the intersecting section is an ellipse given by

$$u^2 + v^2 \cos^2 \varphi = R_0^2, \tag{4}$$

and the radius of curvature at any point is given by

$$R = [(1 + (\mathrm{d}u/\mathrm{d}v)^2]^{3/2} / (\mathrm{d}^2u/\mathrm{d}v^2). \tag{5}$$

The radius of curvature of the crystal surface along the beam is the radius of curvature at $v = 0$ of the ellipse shown in Fig. 2(a). Therefore, if there is an offset of ψ as shown in Fig. 1, the radius of curvature is described by the function

$$R = R_0 / \cos^2(\varphi - \psi). \tag{6}$$

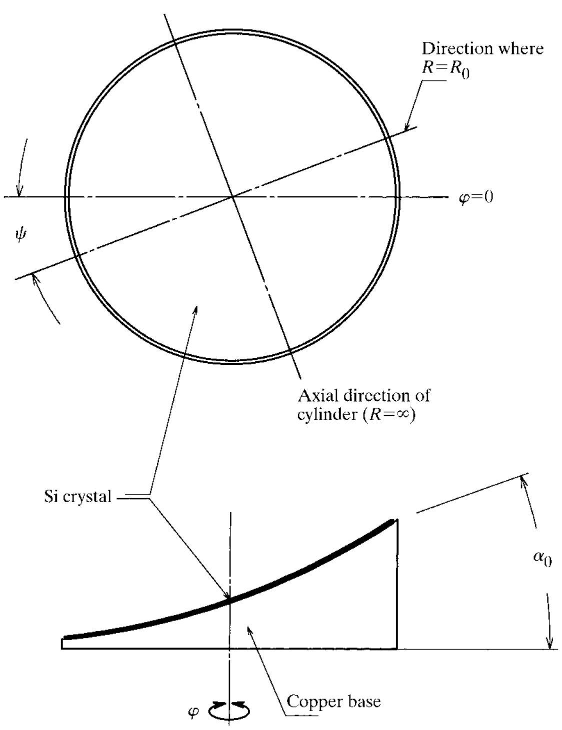

Figure 1
Relation between the rotation axis and parameters of the monochromator. The origin of the azimuthal axis, φ, is defined as the direction which gives a maximum asymmetric angle, α_0, and the offset between $\varphi = 0$ and the axial direction of the cylinder is $90^\circ - \psi$. The azimuthal axis, φ, is perpendicular to the reflection plane.

Furthermore, the asymmetric angle α at any φ is determined as follows. Consider a plane inclined by α_0 relative to the x-y plane as shown in Fig. 2(b). Then z is given by

$$z = r \cos \varphi \tan \alpha_0. \tag{7}$$

Therefore the azimuthal angle, φ, and asymmetric angle, α, are related according to

$$\tan \alpha = \cos \varphi \tan \alpha_0. \tag{8}$$

3. Design and applications

The monochromator was designed for use at the TARA beamline, BL6B, a bending-magnet beamline at the Photon Factory. Beamline BL6B is equipped with a bent plane platinum-coated silicon mirror providing vertical focusing of the beam located at 19.5 m from the source. The monochromator is situated 23.0 m from the bending magnet. The focus position of the beam is 24.38 m from the source. Therefore the optimal asymmetry factor, b, should be 16.7 for this beamline as given by equation (2). The theoretical value of α at any wavelength can be calculated using equation (1) with this b value.

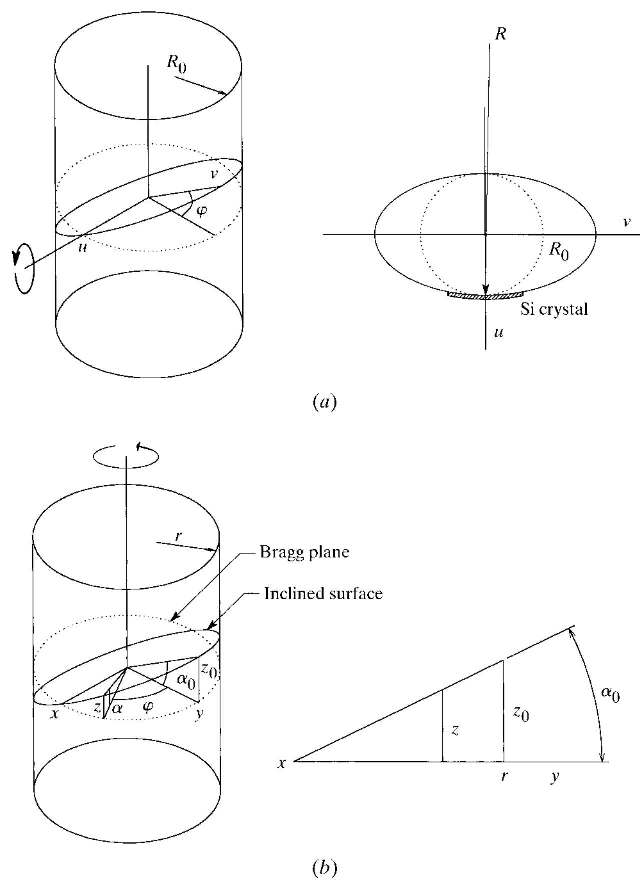

Figure 2
Schematic drawing indicating (a) the relationship between φ and the radius of curvature and (b) the relationship between φ and the asymmetric angle α.

Table 1
Parameters of the monochromator.

Asymmetric angle α is calculated using equation (1). The azimuthal angle of rotation, φ, at optimal α is calculated using equation (8). The radius of curvature, R, at that φ angle is calculated using equation (6). R_{opt} is calculated using equation (3). The expected values of the spectral resolution, $\delta\lambda/\lambda$, at each wavelength were calculated after Lemonnier *et al.* (1978). $\varepsilon = |R - R_{opt}|/R_{opt}$.

Wavelength (Å)	α (°)	φ (°)	R (m)	R_{opt} (m)	ε (%)	$\delta\lambda/\lambda$
1.07	8.73	64.6	72.5	72.3	0.28	3.1×10^{-4}
1.38	11.3	56.0	56.7	56.5	0.35	2.4×10^{-4}
1.74	14.4	44.3	45.0	45.4	0.88	1.9×10^{-4}

The parameters α_0, ψ and R_0 were determined to optimize α and R between 0.87 Å (Kr K-absorption edge) and 1.90 Å (Mn K-absorption edge), which correspond to 8.0 and 17.6° of θ, respectively, for Si(111). The parameters optimized for BL6B were α_0 = 19.7°, ψ = 20.9° and R_0 = 37.9 m. Plots of α and R calculated using these values are shown in Fig. 3. This figure shows that it is possible to tune both asymmetry angle, α, and radius of curvature, R, simultaneously with only one φ-axis rotation. Calculated values of α, R and φ at the three wavelengths where the performance of the monochromator was tested are also shown in Table 1. The error of the curvature, ε, is expected to be less than 1% for all three wavelengths.

The cylindrical surface of the copper block, having a radius of 37.9 m, was fabricated using a high-precision diamond-cutting machine (Higashi *et al.*, 1992). A 1 mm-thick silicon wafer was cut from a 5 inch ingot with an asymmetric angle of 19.7° from the (111) plane and finished by mechano-chemical polishing. In order to fit the crystal to the curvature of the copper block, the crystal was stuck onto the copper cylinder surface using mineral oil (ALDRICH 16,140-3) and the assembly placed into a polyethylene bag. The bag was then evacuated so that the crystal was pushed onto the copper cylinder by atmospheric pressure. Once the crystal was shaped to the copper

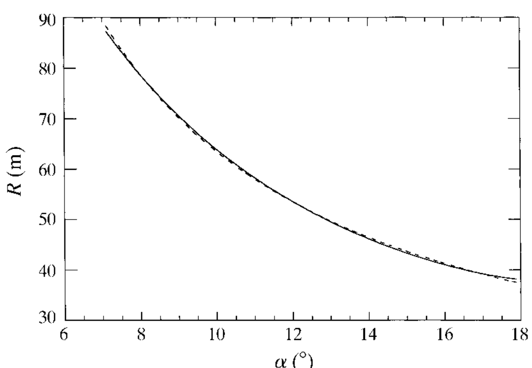

Figure 3
Plot of asymmetric angle, α, and radius of curvature, R. The ideal relation between them (dashed line) is also calculated using equations (1) and (3).

Figure 4
Photograph of the monochromator crystal fixed on the copper cylindrical base.

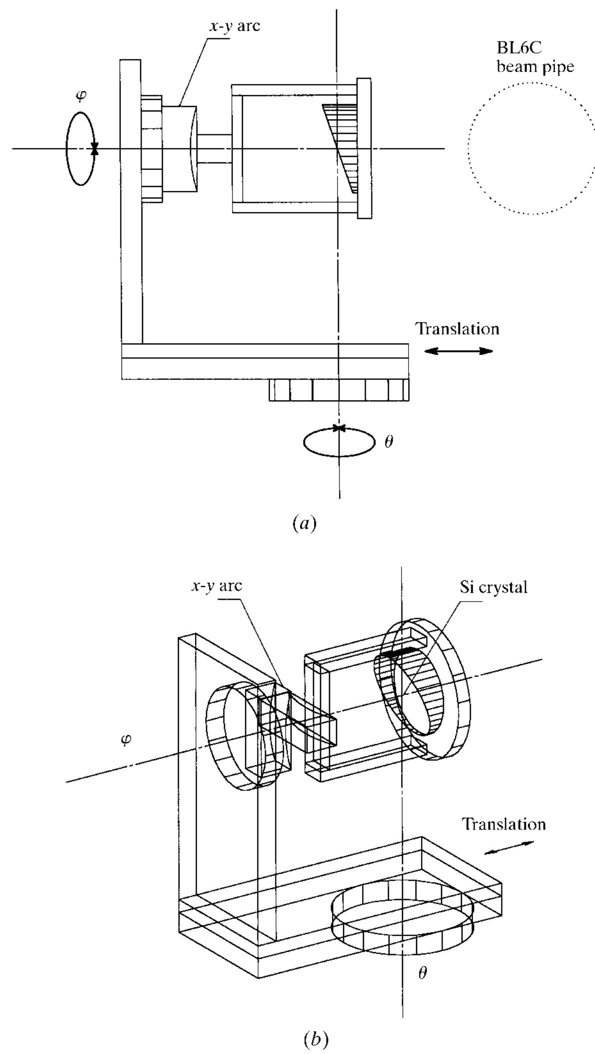

Figure 5
Schematic drawings of the monochromator. (*a*) Side view and (*b*) trimetric view of the goniometers. The azimuthal axis, φ, is on the θ goniometer. The Si crystal surface is located above the centre of the θ axis using a translation stage. Two arcs, x and y, are used to align the reflection plane perpendicular to the φ axis. The crystal is mounted on the goniometer in this way because of spatial restrictions caused by the BL6C beam pipe.

cylinder and held by the surface tension of the mineral oil, the monochromator was used without any other fixing mechanism (see Fig. 4).

Schematic drawings of the monochromator showing the layout of the goniometers are shown in Fig. 5.

4. Results

Three wavelengths (1.07, 1.38 and 1.74 Å) were chosen to demonstrate the performance of the monochromator. The horizontal slit in the beamline was opened to accept 1.0 mrad of the X-ray beam from the bending magnet. The tuning scheme of the monochromator was as follows. First, in order to set the wavelength, the monochromator was rotated around the θ axis which is parallel to the (111) Bragg plane. The crystal was then rotated about the φ axis to tune the asymmetry angle, α, and the radius of curvature, R, simultaneously as described in the previous section. The two-dimensional intensity profile of the focused beam at several φ angles was recorded using imaging plates (Miyahara *et al.*, 1986), and the result for 1.38 Å is shown in Fig. 6. To reduce the beam intensity, 6 mm of aluminium plate was placed between the monochromator and the imaging plate. The horizontal intensity profile was also measured by horizontal scanning of a 0.2 mm-wide slit which was placed at the focus position. The results for all three wavelengths are shown in Fig. 7. The expected beam profile for 1.38 Å calculated using a ray-tracing program (Takeshita, 1995) is also shown in Fig. 7(*d*).

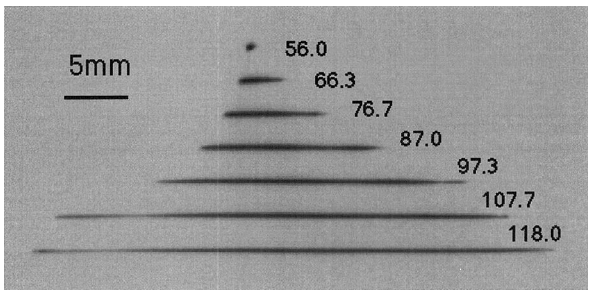

Figure 6
Observed image of the focused beam at 1.38 Å. From lower to upper, φ was rotated from 118.0°, where the surface of the monochromator is almost flat, to 56.0° in 10.3° steps. Since 6 mm of aluminium plate was used as an attenuator, mainly the higher order harmonics of 1.38 Å are recorded on the imaging plate. The imaging plate was moved 3 mm vertically between each exposure to record all images on one plate without overlap.

5. Discussion

As shown in Figs. 6 and 7, a well compressed and focused beam was obtained at all three wavelengths by rotation about the φ axis only. The monochromator could focus the X-ray beam to less than 0.8 mm FWHM in the horizontal direction. The usual width of the final slit or collimator in a protein crystallography experiment is about 0.1 or 0.2 mm. Therefore, a well compressed and focused beam is essential

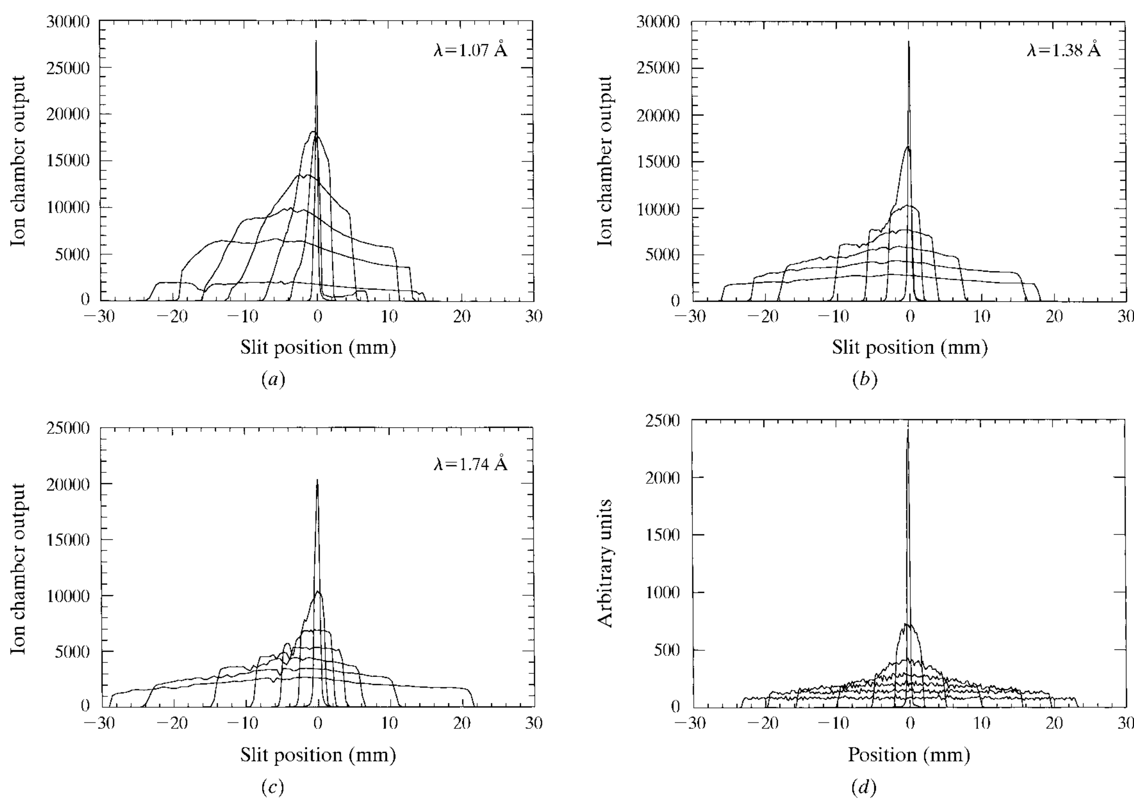

Figure 7
Horizontal intensity profile of the focused beam. A horizontal slit of width 0.2 mm was placed at the focus position and scanned. (*a*) 1.07 Å, (*b*) 1.38 Å and (*c*) 1.74 Å. Expected beam profiles calculated using a ray-tracing program for 1.38 Å are also shown in (*d*).

to obtain high throughput, especially at bending-magnet beamlines at second-generation storage rings such as the Photon Factory. The single-crystal monochromator described in this paper is convenient to use and makes the task of obtaining high fluxes easier compared with triangular bent crystal monochromators because it is able to cover a wide wavelength range with a single crystal. If a triangular single bent asymmetric-cut crystal monochromator is used, five or more monochromator crystals of different asymmetric angle, α, would be necessary to cover the wavelength range mentioned above.

Since this test, this monochromator has been used as the standard monochromator at BL6B. The output beam is quite stable and there have been no problems during conventional collection of protein data for over half a year. It is necessary, however, to find a more sophisticated method of fixing the crystal onto the cylinder surface. As shown in Fig. 7, the observed profiles have an asymmetric aspect. This asymmetry may be caused by an error in the cylinder surface. Of those tested, mineral oil was the best material for fixing the crystal. Greasy glue, such as liquid InGa, affected the focused beam profile due to difficulties in obtaining uniform thickness. We are currently investigating other methods of fixing the crystal, such as a hot isostatic pressing bonding method or an electrostatic bonding, to reduce non-uniformity of the curvature. As shown in Table 1, this monochromator has the potential to achieve a good energy resolution over a wide wavelength range if an ideal cylinder surface can be made. The monochromator is always used near the Guinier condition.

We plan to construct a new undulator beamline for protein crystallography at the 6.0 GeV PF-AR ring on the same campus. In order to realize this type of monochromator for use with the undulator beamline there, cooling techniques to cope with the heat-load problem are necessary. The mineral oil used in this study is not applicable because of its poor heat conductivity and resistance to synchrotron radiation. Once we can establish a new crystal-fixing method, many indirect cooling techniques which have already been developed will be utilized. This type of monochromator with horizontal dispersion geometry is also useful in the construction of a branch beamline if there are spatial restrictions. Spatial problems may be especially serious on compact storage rings.

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