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Tests of an Asymmetric Monochromator to Provide Increased Flux on a Synchrotron Radiation Beamline

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Asymmetric channel-cut monochromators have been tested at the SRS. Results from both focused and unfocused beamlines have shown a threefold improvement in flux when compared with the flux obtained from a symmetric cut Si(111) monochromator. Some problems with using such monochromators and possible modifications are described.

Keywords: asymmetric monochromators; X-ray diffraction.

1. Introduction

Many X-ray beamlines on storage rings use perfect crystals of silicon or germanium to monochromate the beam for a particular application. Single, bent, asymmetric cut crystals are often employed in X-ray diffraction experiments to compress the beam and alter the focusing conditions while retaining a small wavelength spread (Lemonnier, Fourme, Rousseaux & Kahn, 1978). X-ray beamlines using these monochromator systems cannot be rapidly tuned as the position of the diffracted beam alters as the wavelength is changed. Beamlines for rapid tunability use double-crystal monochromators. In a well designed system of this type, only small changes in beam position occur as the wavelength is changed because the second crystal restores the direction of the monochromatic beam to that of the white beam.

The intrinsic bandpass of symmetric double-crystal monochromators is suitable for many spectroscopic purposes (for example, EXAFS, XANES) as well as for some anomalous-scattering applications where it is required to resolve changes in scattering of the relevant atoms across absorption edges. However, the bandpass of these monochromators is less (by one or even two orders of magnitude) than that required for many X-ray scattering and diffraction applications. The consequence is that the potential useful photon flux (in the phase space required by the experiment) from the storage ring is not fully used. The vertical divergence of a synchrotron radiation X-ray beam is of the order 0.1–0.3 mrad, whereas the Darwin width of a symmetric Si(111) monochromator operating at 0.9 Å is 0.02 mrad. If the full vertical divergence of the radiation is used the overall bandpass is determined by the divergence of the radiation rather than the intrinsic width of the monochromator. As pointed out by Kohra, Ando, Matsushita & Hashizume (1978) the two figures can be matched by the use of an asymmetric cut monochromator in spatial expansion geometry, resulting in an increase in flux. This matching is in contrast to that achieved by bending a crystal to match the divergence and obtain a narrow bandpass (Van der Hoek, Werner, Van Zuylen, Dobson, Hasnain, Worgan & Luijckx, 1986). For many experiments, a narrow bandpass is not required and the use of double-crystal asymmetric monochromators gives a useful gain in flux, whether the overall bandpass of the system is determined by the monochromator rocking width or the synchrotron radiation divergence.

The gain of a given asymmetric double-crystal monochromator depends on the wavelength. As the wavelength is reduced, the gain increases, until the incident beam approaches the critical angle for reflection from that surface. The incident beam is then specularly reflected from the surface rather than the Bragg planes and the Bragg intensity drops (Kishino & Kohra, 1971). The gain obtained using these monochromators has already been reported in a previous paper (Nave, Gonzalez, Clark, McSweeney, Cummings & Hart, 1995) which deals more generally with single- and double-crystal monochromators. In this paper we show that, provided a suitable monochromator is constructed for a particular wavelength, the gain to a first approximation is independent of wavelength. The factors limiting the gain are discussed and a comparison is made with measurements made using an asymmetric monochromator on stations 9.5 and 7.6 on the SRS.

2. Theoretical considerations and principles of operation

The gain of the asymmetric monochromators is given by the increase in the angular width of acceptance by a factor of $b^{-1/2}$. This translates to an increased bandpass for a white

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beam. The asymmetry factor b is given by

$$b = \sin(\Theta_B + \alpha)/\sin(\Theta_B - \alpha),$$

where Θ_B is the Bragg angle and α the angle of the Bragg planes to the surface (Kohra, Ando, Matsushita & Hashizume, 1978). The convention used here is that a negative α ($b < 1$) corresponds to the situation where the input X-rays are at a small angle to the surface and are reflected at a large angle. To operate at the maximum gain the input grazing angle ($\Theta_B - \alpha$) has to be as close as possible to the critical angle for reflection Θ_C, where (James, 1948, pp. 54, 172)

$$\Theta_C = 0.00234\lambda(\rho Z/M)^{1/2}.$$

Here Z is the atomic number, M the atomic mass, λ the wavelength in Å and ρ the density in CGS units of the material in the crystal. For silicon, $\Theta_C = 2.5$ mrad (0.144°) at 1 Å wavelength.

If the monochromator is set with an input grazing angle of $n\Theta_C$ the gain is

$$[\sin(2\Theta_B - n\Theta_C)/\sin(n\Theta_C)]^{1/2}.$$

At $n = 4$, a gain of 5.5 can in principle be obtained. Note that as Θ_B and Θ_C are both approximately proportional to the wavelength, similar gains can be achieved at any wavelength provided a monochromator with the appropriate asymmetric cut is available. However, the gain also depends on how close to the critical angle one can operate before specular reflection, absorption and surface roughness effects reduce the Bragg intensity.

The effect of specular reflection (Kishino & Kohra, 1971) is to remove intensity from the Bragg reflected beam at very low glancing angles (near the critical angle for reflection). This effect is ignored here as much larger glancing angles were used.

The effect of absorption can in principle be significant at these larger glancing angles. This has previously been illustrated for a Ge(111) monochromator operating at 1.54 Å wavelength (Deutsch, 1980), where a maximum gain was obtained at a glancing angle of approximately 0.5°. At smaller glancing angles the increased absorption counteracts the increased gain due to the asymmetry factor and the intensity falls off even before specular reflection occurs. We have repeated these calculations using the same method and obtained the same results as Deutsch (1980). The theoretical curves (allowing for absorption but not specular reflection) for an Si(111), 8.4° asymmetrically cut double-crystal monochromator as a function of energy are shown in Fig. 1. Similar results were also obtained using the program *Fhkl* (Soyer, 1995) to calculate the integrated reflectivity as a function of glancing angle and wavelength.

The effect of crystal imperfections on the intensity and crystal rocking widths is rather complex. Surface and subsurface damage will be caused during the construction of channel-cut monochromators. A simple mosaic block model of the damaged layers gives some indication of the effects that might occur. If the angular distribution of mosaic blocks is larger than the Darwin width, the effect would be to increase the range of wavelengths selected from a white beam incident on the crystal, giving increased flux. Another factor, which might give an opposite effect, relates to the fact that two reflections are used, one from each channel. The incident monochromatic beam on the second surface might have to traverse through a considerable distance before it met a block with the correct orientation for reflection. Consequently, a decrease in intensity of the reflected beam due to absorption might occur. The small glancing angles and the narrowing of the Darwin width between the grooves (see below) would increase these effects in asymmetric monochromators.

Some indication of the effect of surface imperfections can in practice be obtained from the asymmetric single-crystal monochromators at the SRS. These are used in compression geometry (Lemonnier, Fourme, Rousseaux & Kahn, 1978) at grazing angles giving asymmetry factors of around 9. At lower exit grazing angles, there is no longer a uniform intensity distribution across the reflected beam, presumably due to surface imperfections. For a perfect two-crystal system, the surfaces must be parallel to within a fraction of their rocking widths in order to be matched for reflection. As the rocking width between the two crystal surfaces is decreased with respect to a symmetric

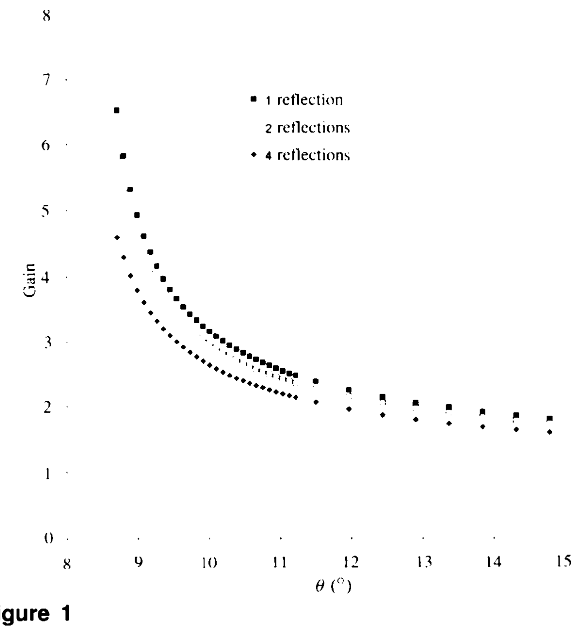

Figure 1
Theoretical curves for the gain of an 8.4° cut Si(111) monochromator compared with a symmetric reflection. The calculation takes into account the variation of f' and f'' with wavelength and the consequent effects of absorption. The reduction in intensity due to the specularly reflected beam is negligible at glancing angles greater than 0.5° and is therefore ignored. The calculation also assumes a perfect crystal with no subsurface damage. Curves are shown for one reflection and four reflections in addition to the two-reflection case relevant to the monochromators used here.

monochromator (by $b^{-1/2}$), the Bragg planes have to be parallel to within a few microradians. This means that a strain-free mounting has to be devised and any thermal effects on the first reflection surface minimized. It is possible that failure to take these factors into account has meant that such devices are not in routine operation at synchrotron sources. The experience with the asymmetric single-crystal monochromators indicates that gains of around 3 (at an asymmetry factor of 9) should be achievable, without any special surface preparation, provided the monochromator is well constructed.

The purpose of the tests was to investigate which of the various effects was limiting in an asymmetrically channel-cut monochromator and to demonstrate that increased intensity could be usefully exploited for protein crystallography.

3. Design of the monochromator crystal

The monochromators described in this paper were constructed for use on beamline 9.5 at the SRS Daresbury, where a total heat loading of 9 W is to be expected on the monochromator first crystal. The first crystal is therefore cooled to prevent any distortion of the crystal face or change in Bragg spacing due to thermal effects. For the very small angles of incidence at which the monochromators are used, the footprint of the X-rays is long, typically 4 × 70 mm. Thus, the thermal load per mm^2 is relatively low and direct jet cooling of the first crystal surface (Hart, 1990), as used with the symmetric monochromator, is not necessary. The silicon crystal is attached to a water-cooled copper block and good thermal contact is ensured by a layer of liquid gallium between the copper and the silicon. The monochromator crystal is glued onto the surface of the copper block by a strain-free mounting. A monochromator of this type is shown in Fig. 2.

4. Testing of the monochromator

Beamline 9.5 is equipped with a toroidal mirror providing horizontal and vertical focusing of the X-ray beam from the wiggler. The beamline accepts 1.2 mrad horizontally and 0.1 mrad vertically. The monochromator is situated near the focus of this mirror, where the vertical size of the beam is small. The full beam can be accepted by the asymmetric monochromator at narrow incident angles to the crystal surface. The non-uniform distribution of intensity at the monochromator position, due to the toroidal mirror, introduces complications when testing the intrinsic performance of the monochromators. The tests on station 9.5 were therefore confined to evaluating the gain which could be achieved on this beamline and that satisfactory data could be collected for protein crystallography.

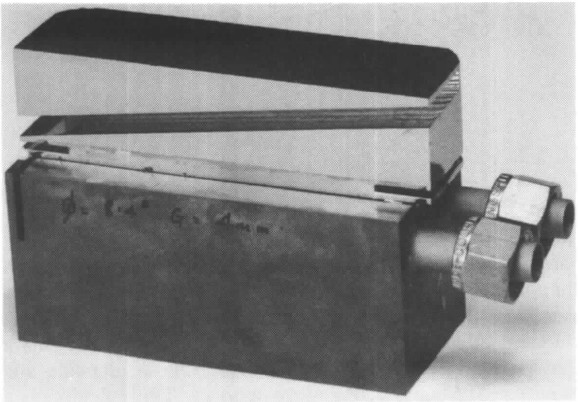

Figure 2

A channel-cut monochromator with an asymmetry of 8.4°. The cooling block and strain relief mounting can be seen.

In order to investigate the intrinsic performance of the monochromators, additional testing was carried out on station 7.6, a bending-magnet beamline on the SRS. The experimental station is 80 m away from the X-ray source and, with the small (0.3 mm FWHM) vertical dimensions of the source, excellent angular collimation is achieved. The tests were performed with a small (1 mm horizontal by 0.1 mm vertical) beam in order to examine the behaviour of the monochromator at defined positions along its length. An ionization chamber before the monochromator was used to normalize for incident flux and a second one was used to measure the flux in the 111 reflection as a function of monochromator angle. The experiment was repeated with a symmetric monochromator in order to obtain the gain factor as a function of wavelength.

5. Results of monochromator tests

Fig. 3(*b*) shows a plot of the intensity for the 8.4° cut monochromator against diffraction angle compared with the intensity given by a symmetric monochromator (Fig. 3*a*). A maximum gain of approximately 3 is obtained. The intensity for the asymmetric monochromator peaks at an angle

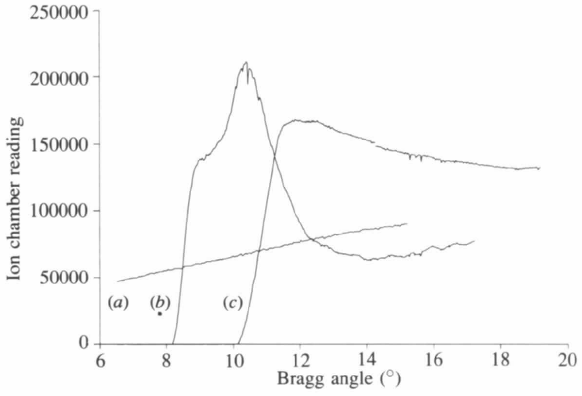

Figure 3

The relative intensities given by a two-crystal asymmetric monochromator compared with a symmetric monochromator. The asymmetric monochromator has an angle α = –8.4° for the first reflection and α = 8.4° for the second reflection. These curves were measured on station 7.6 at the SRS as described in the text. (*a*) Symmetric monochromator; (*b*) 8.4° cut asymmetric monochromator, position one; (*c*) 8.4° cut asymmetric monochromator, position two.

of just under 2° to the surface and then decreases again. White-beam topographs were taken using a beam reflected from only one surface of the monochromator at an incident angle of 1.5°. These showed the characteristic 'orange peel' effect due to subsurface damage and/or roughness. The crystal surfaces had been sawn then chemically polished to relieve surface strain. However, subsurface damage may remain in the crystal. The channel width is too small to syton polish the diffracting surfaces. The presence of subsurface damage would explain the reduction in intensity at low grazing angles as discussed in §2. Taking this into account, the gain shown in Fig. 3 matches the expected value reasonably well. However, different behaviour was observed from different parts of the monochromator. Both higher and lower intensities could be observed at some angles. An example is given in Fig. 3(*c*). A possible explanation for this is a variation in surface quality along the crystal with an increased gain occurring under some conditions as described in §2. Roughening of the crystal surfaces is in fact an alternative way of providing increased bandpass. However, the peak intensity in Fig. 3(*c*) is less than that in Fig. 3(*b*). In addition, a highly mosaic surface on the crystal can have a detrimental effect on the focusing in the beamline.

On station 9.5, when tuned to give the maximum intensity (as measured on an ion chamber), gains of 3 were obtained in comparison to the standard symmetric cut monochromator on this station. Test data sets from a standard lysozyme crystal were of similar quality ($R_{\rm symmetric} = 0.037$, $R_{\rm asymmetric} = 0.041$) and scaled together with an $R_{\rm merge} = 0.047$. This indicates that the increased intrinsic bandpass did not introduce significant additional errors in the data. This is as expected because the overall bandpass under these conditions is similar, due to the 0.1 mrad angular convergence of the vertically focused X-rays.

One problem that did occur with these monochromators was due to the additional Laue reflections. As the beam on station 9.5 almost completely fills the gap between the crystals, the reflections are rather close to each other. When the beamline was well focused, it was possible to slit out unwanted reflections. However, if the toroidal mirror becomes defocused (due either to source or optics instabilities) the various reflections can overlap leading to contamination of the monochromatic beam. Accurate alignment of the focusing optics is therefore crucial when using this type of monochromator.

6. Discussion

The monochromators as tested give results in agreement with the principles outlined in §2. This does not mean to say that they give predictable results. The precise nature of the surface and subsurface layers appears to be crucial in determining the overall reflectivity of double-crystal monochromators at low glancing angles. This is difficult to control, especially with a channel-cut system containing a narrow groove.

The tests have suggested that a monochromator with two separate crystals (rather than a channel-cut crystal) would have certain advantages. Firstly, it should be possible to prepare the surfaces optimally. This might allow operation at smaller incident angles and lead to higher gains. Secondly, it would also be possible to have different orientations of the two crystals with respect to one another (about an axis normal to the Bragg planes) in order to decrease the number of additional Laue spots near the 111 reflection. Some tuning of the Bragg angle of the two crystals with respect to one another would be necessary.

Nevertheless, we have demonstrated that useful gains in intensity can be obtained with these monochromators both under the test conditions of station 7.6 and during data collection on station 9.5. As the incident beam on the first crystal is spread out over a large area, monochromators of this type are particularly suitable for operation on a high-powered wiggler or undulator source. However, at the highest thermal loads, jet cooling of the monochromators (Hart, 1990) would eventually become necessary.

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