BACK-SCATTERING CHANNEL-CUT HIGH RESOLUTION MONOCHROMATOR FOR INELASTIC X-RAY SCATTERING*

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Backscattering channel-cut high-resolution monochromator for inelastic x-ray scattering

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ABSTRACT

We report on a design and on some experimental results for the performance of a new high energy resolution monochromator. It is a large channel-cut Si crystal with a 197 mm separation between the two faces designed to operate in a near-backscattering regime. The device was tested as a second monochromator on Sector 3 of the Synchrotron Radiation Instrumentation Collaborative Access Team (SRI-CAT) at the Advanced Photon Source using the Si(777) reflection at a photon energy of 13.84 keV. The same monochromator can be used for other energies with reflections of the type (hhh). Special care has been taken to equalize the temperature of the two faces by employing a Peltier heat pump. A Si(111) double-crystal pre-monochromator designed to withstand the high heat load of the undulator radiation was used upstream on the beamline. The measured throughput efficiency of the Si(777) channel-cut monochromator was less than ideal by a factor of 1.9. Dynamical diffraction theory was used to calculate the throughput of an ideally perfect crystal.

Keywords: inelastic x-ray scattering, high energy resolution, x-ray optics, synchrotron radiation instrumentation

1. INTRODUCTION

Inelastic x-ray scattering (IXS) spectra are a source of information on the dynamics of atoms.[1] However, progress in the field has been limited due to the low scattering cross sections of elementary excitations (e.g., phonons). Very high incident beam fluxes with bandpasses of the order of $\Delta E/E = 10^{-7}$ are needed to study such excitations. Access to such beams is becoming increasingly available at both the European Synchrotron Radiation Facility (ESRF)[2] and at the Advance Photon Source (APS). Both the pioneering instrument called INELAX (see ref. 1) at the synchrotron in Hamburg (HASYLAB) and the more recent instrument at ESRF[3] have employed a backscattering geometry from one crystal to reach the requisite incident bandpass. Energy scanning with such a monochromator is achieved by tuning the temperature difference between the monochromator and a high-resolution analyzer.[4] Beamline layouts employing a single backscattering reflection to set the incident bandpass are made complicated because of the need to transport the beam close to a sample before diffracting it from the backscattering crystal. The inelastic scattering beamline (3-ID) in the Synchrotron Radiation Instrumentation Collaborative Access Team (SRI-CAT) of the APS has instead employed in-line monochromators to set a narrow bandpass. Other types of monochromators that are commonly referred to as nested and are made up of two channel-cut crystals have been used at beamline 3-ID to set a narrow bandpass.[5] The double backscattering monochromator (DBSM) presented here has the following advantages over the nested design: i) it uses only two bounces as opposed to four in the nested design, and ii) it accommodates (hhh) reflections and can therefore be used at several energies (i.e., those corresponding to backscattering from (hhh) reflections). Scanning for the DBSM is possible by rocking the crystal so that the Bragg angle is changed but must be done in conjunction with temperature control to establish a temperature difference between the DBSM and an analyzer in order to capture an elastic peak in IXS experiments.

2. BACKSCATTERING MONOCHROMATOR

The design of the double backscattering monochromator (DBSM) is shown in Fig. 1. The monochromator is made of a single crystal of silicon and measures 197 mm between faces. An x-ray beam coming from the pre-monochromator is first

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backscattered from the downstream surface of the DBSM. A beam reflected from this surface is again backscattered towards a specimen. As shown in Fig. 1, the overlap between the two faces is 1 mm.

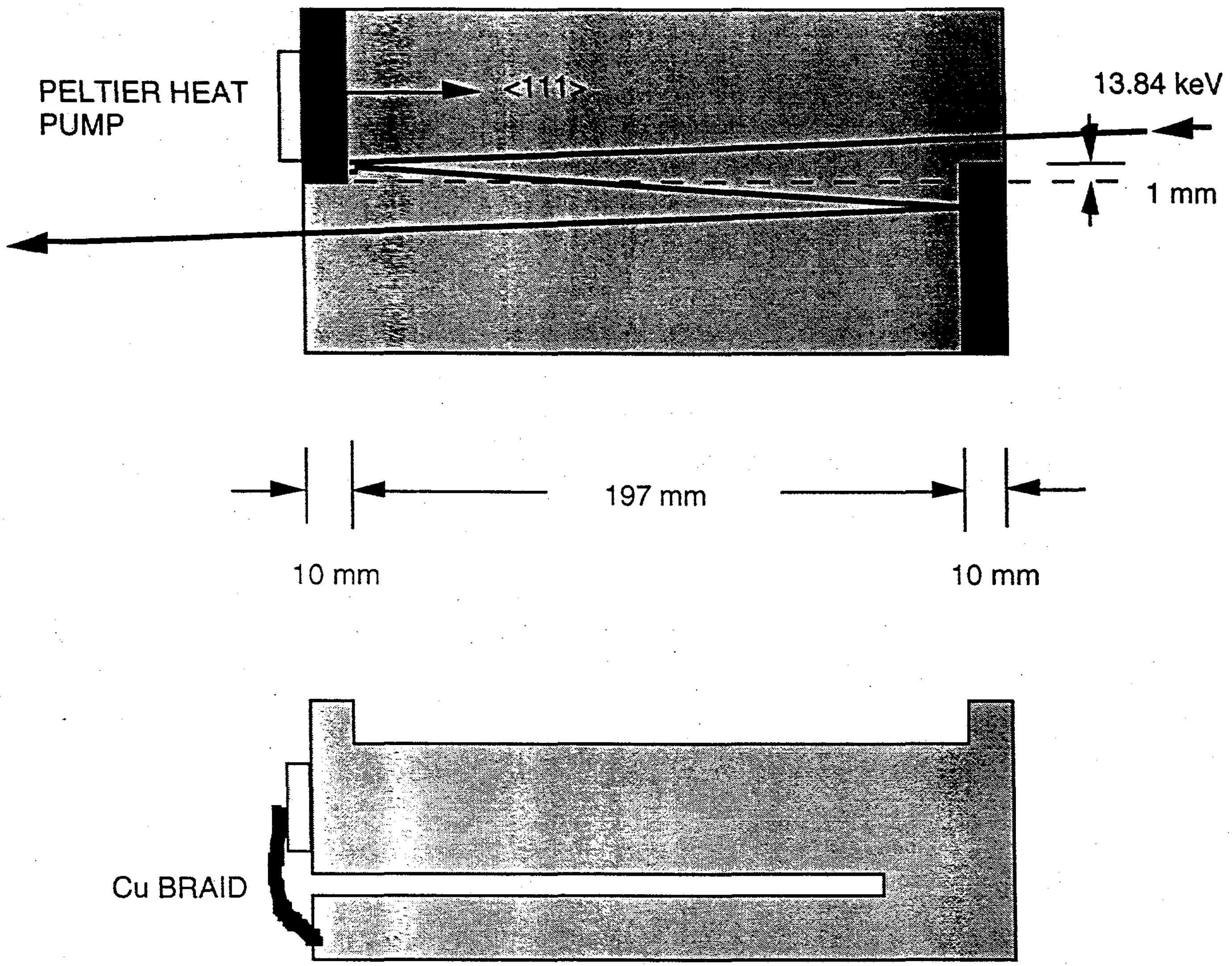

Fig. 1. Double backscattering monochromator (DBSM).

The overlap, together with the 197 mm value for the channel-cut opening, sets an upper limit for the Bragg angle. For the (777) reflection, the upper limit is 89.9°. For IXS experiments with a backscattering analyzer designed to function with a Bragg angle larger than 89.9°, the elastic peak is not measurable if the temperature of the DBSM and the analyzer are identical. Only a small temperature difference between the two optics (0.5 deg for the (777) reflection) is needed to overcome this difficulty, however.

Energy can be scanned to higher values by decreasing the Bragg angle, i.e., by rotating the DBSM away from backscattering. An effective limit for this is reached when the Darwin width decreases to a value equal to the incident beam divergence. For higher energies than this limit, throughput will be diminished because not all of the incident beam is accepted. For the (777) reflection, scanning over an energy range in excess of 1.0 eV is possible according to this criteria. In an IXS experiment for which energy analysis of the scattered radiation is done at a fixed energy, the energy loss spectrum (i.e., Stokes shifted) can be obtained by scanning the monochromator to higher energies. This means the present monochromator design is suitable. The energy bandpass, however, changes as one scans due to a changing geometrical contribution (i.e., the contribution arising from the derivative of Bragg's law and the incident divergence). However, this effect is not large. The geometrical term contributing to the bandpass is only 50% of the intrinsic term at an energy shift of 1 eV.

A full calculation of the reflectivity for 4 reflections (2 from the pre-monochromator and 2 from the high-resolution monochromator) is shown in Fig. 2 for ideally performing crystals. These calculations are at the high Bragg angle limit of the DBSM for which the geometrical contribution is negligible (~10% of the intrinsic contribution). Here Darwin curves[6] at different energies were obtained by shifting them according to Bragg's law for each reflection. Explicit inclusion of refraction was not necessary because all crystals were aligned to be at y=0. The structure-factor-related polarization terms in the dielectric constant of the crystals are listed in Table I.

Table I. Crystal polarization terms (proportional to the structure factor).

$\Psi'_{111} = -2.678 \times 10^{-6}$

$\Psi''_{111} = -2.865 \times 10^{-8}$

$\Psi'_{777} = -3.933 \times 10^{-7}$

$\Psi''_{777} = -1.856 \times 10^{-8}$

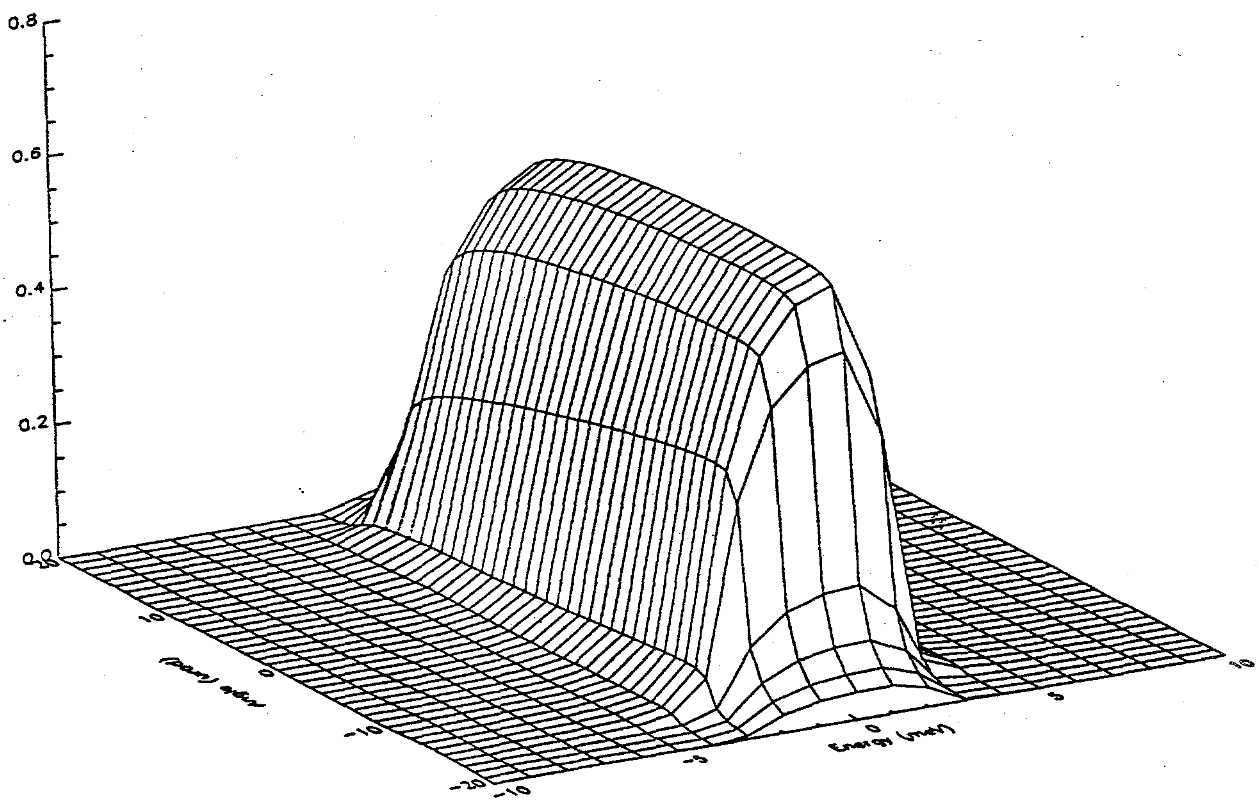

Fig. 2. The calculated reflectivity function for the DBSM and including the pre-monochromator. The surface represents the distribution in angle and energy for a beam exiting the high-resolution monochromator.

We incorporated the Hönl corrections for anomalous dispersion (Δf' and Δf") according to data by Cromer and Liberman[7] (as made available by Brennan and Cowan[8]) and Debye-Waller factors according to Deutsch et al.[9] A cross cut at constant angle through the surface shown in Fig. 2 is shown in Fig. 3. The FWHM of this cross cut is 4.4 meV.

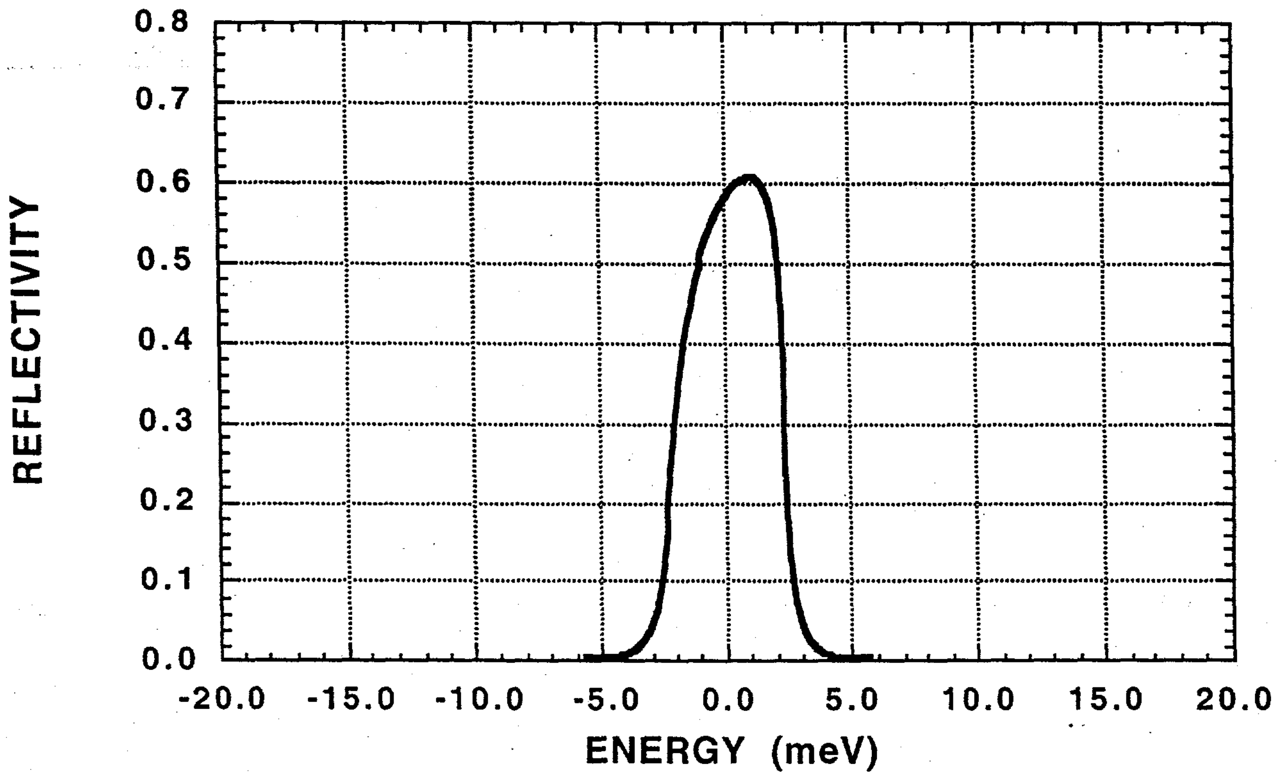

Fig. 3 Cross cut at zero angle through the monochromator reflectivity function. The FWHM is 4.4 meV.

The crystal was cut from a (111) oriented boule that was 100 mm in diameter grown by the float zone technique. A Meyer-Burger slicing saw with a diamond-impregnated bronze blade suitable for rapid cutting, i.e., with large diamond particles, was used to cut both crystals. Long diffraction faces for both channel cuts were present after sawing, and these were ground by hand to a fine finish using 9 micron aluminum oxide grit. The long diffracting faces of the inner crystal were then shortened using a grinding wheel. Polishing of the diffraction surfaces was then performed by hand. Lastly, acid etching of the entire crystal for strain removal was then performed in a mixture of nitric acid and hydrofluoric acid.

3. EXPERIMENT

A main concern for such monochromators is the efficiency. Measurement of the efficiency was conducted at the Sector 3 undulator beamline of Advanced Photon Source (APS). We employed a high-heat-load pre-monochromator with two separate asymmetrically cut silicon crystals set for the (111) reflection, the first one being liquid gallium cooled.[10] The incoming beam power from this pre-monochromator is about 40 milliwatt, and almost all this energy is dissipated in the first reflecting surface of the DBSM. This power heats the first reflecting surface just enough to change its bandpass by one Darwin width, and, therefore, the beam will not be reflected from the second surface. In order to prevent this "heat load" problem, a Peltier cooling chip (1 cm x 1 cm) was glued behind the first reflecting surface and connected via the strain relief portion to the other side of the crystal with copper mesh (see Fig. 1).

Ideally, one desires to reduce the throughput by a factor proportional only to the bandpass reduction. Values of up to ~32 microrad FWHM (with a shoulder-peak structure) were measured for the vertical angular profile of the beam exiting the pre-monochromator.[11] For radiation distributions with this vertical divergence, the ideal throughput reduction ratio for the DBSM is 1200 as calculated by dynamical diffraction. A value for the loss ratio was measured and was found to be 2300. The loss ratio was measured with ion chambers placed immediately upstream and immediately downstream of the high-resolution monochromator. The downstream ion chamber had been calibrated with respect to the upstream ion chamber by letting the beam pass directly from one to the other, i.e., without intervening optics. The reason for the loss in throughput beyond that expected from an ideal performance has yet to be clarified.

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