The Resolution Function of a Triple-Crystal Diffractometer for High-Energy Synchrotron Radiation in Nondispersive Laue Geometry

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(Received 24 February 1994; accepted 24 June 1994)

Abstract
The k-space resolution function of a triple-crystal diffractometer is calculated for an arrangement of three perfect silicon single crystals Bragg diffracting in nondispersive Laue geometry. A comparison is made with the results of measurements using synchrotron radiation in the energy range from 80 to 150 keV. In this case, absorption is very weak and according to dynamical theory the width of the diffraction pattern of thick perfect single crystals is proportional to the wavelength $\lambda$, whereas its Lorentzian tails are proportional to $\lambda^2$. Together with the fact that the Bragg angles are only of the order of $2^\circ$, this leads to a concentration of the starlike k-space resolution function into a narrow band parallel to the reciprocal-lattice vector $G$. For diffraction of 80 keV synchrotron radiation at the silicon 111 reflection, the full width at half-maximum (FWHM) of the intensity distribution in the scattering plane is $1.1 \times 10^{-5} \text{ Å}^{-1}$ perpendicular to $G$ and $2.2 \times 10^{-4} \text{ Å}^{-1}$ parallel to $G$. The observed differences in the contributions from monochromator and analyzer crystal to the resolution function are explained by the finite width of the electronic window of the detector counting chain and the non-Bragg scattering contribution from the crystals. If annealed Czochralski-grown silicon single crystals with a mosaicity of $\sim 3^\circ$ are used as monochromator and analyzer, the resolution is reduced by one order of magnitude, but for studies of imperfect samples or of diffuse scattering large gains in intensity can be accomplished this way.

I. Introduction
The interaction of synchrotron radiation of energy of the order of 100 keV with matter is weak and diffraction experiments can be performed on large samples with dimensions typical for neutron scattering experiments. In fact, the same samples can be studied with neutrons and high-energy synchrotron radiation. Triple-crystal diffractometers (TCDs) for hard X-rays show very high momentum or k-space resolution in directions perpendicular to the reciprocal-lattice vector $G$. Parallel to $G$, the resolution is comparable to the resolution of TCD using X-rays in the 1 Å range.

The starlike resolution function of X-ray TCDs was first observed by Pick, Bickmann, Pofahl, Zwoll & Wenzl (1977) and Iida & Kohra (1979) and has been discussed since then by several authors for X-rays with wavelengths in the 1 Å range. Zaumseil & Winter (1982) calculated the TCD resolution for a non-dispersive arrangement of three perfect crystals; they also discussed the advantage of using multiple-reflection channel-cut crystals as monochromator and analyzer, in order to reduce the contribution of the long tails of the intrinsic diffraction patterns of perfect crystals. A more general discussion of the resolution of a TCD has been given by Pynn, Fujii & Shirane (1983), who assumed a sample with a Gaussian-shaped diffraction pattern, which is appropriate for mosaic crystals. Cowley (1987) gives a complete description of the central part of the TCD resolution function for perfect as well as for mosaic crystals. Werner & Arif (1988) introduced another aspect to the discussion of TCD resolution. They reconsidered the resolution of a high-resolution high-sensitivity neutron triple-axis diffractometer and demonstrated that two intensity streaks intersecting at the Bragg point should always be expected in a diffraction experiment because both the detector acceptance function and the angular intensity distribution of the beam incident on the sample are composed of a narrow and a wide distribution, the latter being due to the transmission characteristics of real Soller collimators, small-angle scattering and air scattering. Lucas, Gartstein & Cowley (1989) discuss three different experimental configurations using perfect germanium, distorted germanium and pyrolytic graphite as monochromator and analyzer crystals, respectively. In a further paper, Gartstein & Cowley...
(1990) present general theoretical expressions for the k-space intensity distribution of Bragg reflections for both Bragg and Laue geometry for the sample with the assumption that monochromator, sample and analyzer are perfect crystals. They include the spectral distribution of an X-ray tube and obtain good agreement between their calculations and experimental data. In the present paper, the k-space resolution function of a triple-crystal diffractometer for synchrotron radiation with energies in the range from 80 to 150 keV is calculated for an arrangement of three perfect silicon single crystals as well as mosaic crystals as monochromator and analyzer in non-dispersive, symmetric and asymmetric Laue geometry and again comparison is made with experimental data.

The paper is organized as follows. After an outline of the layout of the triple-crystal diffractometer for high-energy synchrotron radiation and a presentation of the general formula for the TCD intensity distribution, the results of calculations for perfect and mosaic-type crystals are given for symmetric Laue geometry. A discussion of the results obtained for an asymmetrically cut perfect sample crystal is included. The comparison with experimental data is followed by a discussion of observed differences in the contributions from monochromator and analyzer crystal to the resolution function at distances far from the reciprocal-lattice point. Finally, the special aspects of TCD for high-energy synchrotron radiation are summarized.

II. Calculation of the resolution function

Fig. 1 shows the principle of the TCD. Once the photon energy is chosen, the monochromator is fixed and the k-space distribution of the Bragg-scattered intensity is scanned by tilting the sample and the analyzer.

The TCD resolution function is best discussed in k-space and a local coordinate system with its origin at a reciprocal-lattice point is introduced. \( q_x \) and \( q_y \) are the coordinates in the scattering plane parallel and perpendicular to the reciprocal-lattice vector \( G \), respectively. The coordinate perpendicular to the scattering plane is not considered explicitly, because along this direction the length of the resolution element is of the order of \( 8 \times 10^{-2} \text{ Å}^{-1} \), so that full integration of the intensity distribution occurs, i.e., in the following, only the projection of the three-dimensional intensity distribution onto the scattering plane is considered.

If \( \mathbf{k}_l(0) \) and \( \mathbf{k}_f(0) \) denote the wave vectors of the incident and the diffracted beams, respectively, Bragg scattering from the sample occurs if \( \mathbf{G} = \mathbf{k}_f(0) - \mathbf{k}_l(0) \). The corresponding angular positions of the sample and analyzer crystals are denoted \( \omega_2(0) \) and \( \omega_3(0) \) and represent the reference setting of the triple-crystal diffractometer. As shown in Fig. 2, deviations \( \omega_2 \) and \( \omega_3 \) from this reference setting define the k-space coordinates \( q_x \) and \( q_y \) according to

\[
q_x = |\mathbf{k}| \cos \theta_B \omega_3 \\
q_y = |\mathbf{k}| \sin \theta_B (\omega_3 - 2\omega_2)
\]

\( \theta_B \) is the Bragg angle.

If \( J(\Delta\lambda) \) denotes the spectral and \( F(\varphi) \) the angular distribution function of the primary synchrotron-radiation beam, the intensity distribution measured with a TCD in a nondispersive setting is calculated on a relative scale by expanding the formula for a double-crystal diffractometer (Zachariasen, 1945) to a TCD as

\[
I(\omega_2, \omega_3) = \int \int d(\Delta\lambda) F(\varphi) J(\Delta\lambda) R_M(\varphi) R_S(\varphi) R_A(\varphi) \\
\times \left[ R_M(\omega_2 - b_1(\varphi)) \right] \\
\times \left[ R_S(\omega_3 + \omega_2 - b_2(\varphi)) \right] \\
\times \left[ R_A(\omega_3 - 2\omega_2 - b_3(\varphi)) \right]
\]

(2.2)

with

\[
b_i = \sin (\theta_B - \varphi_i)/\sin (\theta_B + \varphi_i).
\]

In the present case, \( J(\Delta\lambda) \) can be assumed to be constant over the narrow wavelength band involved in the diffraction. \( R_M, R_S \) and \( R_A \) denote the diffraction patterns from monochromator, sample and analyzer crystal. \( \delta = (\Delta\lambda/\lambda) \tan (\theta_B) \) represents the change in the wavelength.

![Fig. 1. Schematic drawing of a TCD in nondispersive setting. \( \beta \) is the aperture of the solid-state detector.](image)

![Fig. 2. k-space diagram of the sample crystal. \( q_x \) and \( q_y \) denote the local coordinate system with its origin at the reciprocal-lattice point \( H \). \( \theta_B \) is the Bragg angle. By tilting the sample crystal by an angle \( \omega_2 \), one scans reciprocal space almost perpendicular to \( G \), while, with the analyzer crystal tilted by an angle \( \omega_3 \), the scan is perpendicular to \( k_f(0) \).](image)
Bragg angle if the wavelength is changed by $\Delta \lambda$ with respect to the value $\lambda^{(0)}$ for the reference setting. $b_i$ is the asymmetry parameter, with $\phi_i$ being the angle between the net planes and the surface normal $n$ of the crystal. In practice, the limits of the integration over $\phi$ are determined by the first collimator and the limits of the integration over $\Delta \lambda$ are determined from the width of the electronic window in the counting chain of the solid-state detector.

II.1. Perfect crystals

For high-energy synchrotron radiation, the Bragg angles are small, absorption can be neglected in most cases and from dynamical theory the diffraction pattern of a thick plane-parallel perfect crystal in Laue scattering geometry with averaging over Pendellösung oscillations is given by (Zachariasen, 1945)

$$R^2 = 1/2(1 + y^2);$$

$$y = \frac{[(1 - b)/2] \psi_0 + b(\theta_B - \theta)}{|b|^{1/2} |\kappa \psi_H|};$$

$$\psi_H = - \frac{(r_0^{2/3} \pi V)}{F_H};$$

$$\omega_{dyn} = 2 |\kappa \psi_H| / b \sin 2\theta_B. \quad (2.3)$$

$\theta$ is the angle of incidence. $\kappa$ is the polarization factor, which is very close to unity for the small Bragg angles involved in diffraction of high-energy synchrotron radiation at low-order Bragg reflections. $V$ represents the unit-cell volume and $F_H$ is the structure factor including the thermal Debye-Waller factor. $r_0 = 2.82 \times 10^{-5}$ Å is the classical electron radius. $\omega_{dyn}$ is the FWHM of the diffraction pattern.

The diffraction pattern is of Lorentzian shape and Fig. 3 shows examples calculated for Mo $K\alpha$, as well as for 150 and 300 keV X-rays, which are available at modern synchrotron-radiation facilities. It is important to note that the FWHM of the diffraction pattern decreases in proportion to the wavelength $\lambda$, whereas the Lorentzian tails decrease in proportion to $\lambda^2$, i.e., in angle space, the scattering power is highly localized at the Bragg peak.

Fig. 4(a) shows the k-space intensity distribution calculated for silicon 111 assuming perfect crystals as monochromator, sample and analyzer with thicknesses much larger than the extinction length $t_{ext} \approx 0.1$ mm. It is confined in a very narrow band parallel to the reciprocal-lattice vector $G_{111}$. In Fig. 4(b), the scale for the component $q_y$ perpendicular to $G_{111}$ is enlarged by a factor of 10 and the starlike shape of the TCD resolution function becomes visible. There are three streaks, the peaks of which correspond to settings of the TCD for which two of three diffraction patterns $R_i$ in formula (2) have their peak values. For example, the so-called sample streak is obtained for a setting where monochromator and analyzer crystals show maximum reflectivity and the analyzer streak corresponds to a setting where sample and monochromator crystals have their maximum reflectivity. In Fig. 4(c), the monochromator, sample and analyzer streaks are indicated.

All three streaks appear because the diffraction patterns of perfect single crystals are not $\delta$ functions, instead, in Laue geometry, their shape is Lorentzian. Qualitatively, the k-space distribution of the scattering power of the sample along the $q_y$ coordinate is obtained by multiplying the angle coordinate $\omega_2$ in Fig. 2 by the magnitude of the reciprocal-lattice vector $G$. The k-space distribution of monochromator and analyzer streaks, inclined by the Bragg angle from the direction of $G$, is obtained by multiplying the angle coordinate $\omega_3$ in Fig. 2 with the magnitude of the wave vector $k^{(0)}$, which, for

![Fig. 3. Lorentzian-shaped reflectivity curves calculated by means of dynamical theory for the 220 reflection of a thick plane-parallel silicon crystal for three different energies. The intensity in the wings of the rocking curve for 150 keV synchrotron radiation is two orders of magnitude weaker than for Mo $K\alpha$ radiation at 17.7 keV.](image)

![Fig. 4. (a) Intensity distribution of a TCD in the vicinity of the Si 111 reciprocal-lattice point calculated for 80 keV synchrotron radiation diffracted from three perfect crystals in symmetric Laue geometry. The peak intensity is normalized to 1, the contour levels are 0.8, 0.5, 0.2, 0.1, 0.05, 0.03, 0.02 and 0.0125. (b) The scale $q_y$ perpendicular to $G_{111}$ is stretched by a factor of ten. (c) Schematic representation of the directions of the monochromator (M), sample (S) and analyzer (A) streaks. Monochromator and analyzer streaks are inclined by $2\theta_B$.](image)
80 keV synchrotron radiation, is 20.22 times larger than \(|G_{111}|\). This large difference between \(|k^0|\) and \(|G|\) leading to the small Bragg angles causes the strong asymmetry in the resolution function. Because the FWHM of the diffraction pattern is proportional to \(|k^0|^{-1}\), to a first approximation, the extension of the central part of the TCD resolution function in the direction parallel to \(G\) is independent of wavelength. However, the distribution of the scattering power of the sample in the direction perpendicular to \(G\) is proportional to \(\lambda\).

For the symmetrical Laue geometry discussed so far, in \(k\)-space the sample streak extends in the direction perpendicular to \(G\), whereas in symmetrical Bragg geometry it would extend in the direction parallel to \(G\) (Zaumseil & Winter, 1982). This is because the wave-vector components parallel to the crystal surface are conserved when the vacuum waves are coupled to the waves inside the crystal [for a detailed discussion, see Batterman & Cole (1964)]. Thus, when a crystal is rocked through the angular range where Bragg scattering occurs, only wave-vector components parallel to the surface normal can be modified. In symmetrical Laue geometry, the surface normal is perpendicular to the reciprocal-lattice vector \(G\); in symmetrical Bragg geometry, it is parallel \(G\).

Fig. 5 shows the intensity distribution calculated for an asymmetrically cut plane-parallel silicon sample where the 220 net planes form an angle of \(\varphi = 45^\circ\) with the sample surfaces, i.e. the asymmetry parameter in (2.2) for the sample is \(b_1 = -1.0067\), which results in a tilt of the sample streak in \(k\)-space by \(45^\circ\). Qualitatively, this feature can be understood with the help of Fig. 6. It shows one branch of the dispersion surface for the wave vectors of the waves propagating inside the crystal together with sections of the two Ewald spheres for the incident and diffracted vacuum waves crossing in the Laue point \(L\). The diffracting net planes form the angle \(\varphi\) with the surface normal. The angle of incidence of the primary X-ray beam may differ by a small angle \(\omega\) from the Bragg angle, the corresponding wave vector is given by the line between \(P\) and \(O\). According to the \(k\)-vector conservation mentioned above, a line along the surface normal \(n\) through \(P\) fixes the tie point \(A\) on the dispersion surface and thus the wave vectors of the wave fields inside the crystal. The same argument holds for the coupling of the diffracted wave inside the crystal to the corresponding vacuum wave. Its wave vector is then given by the line between \(P\) and \(H\). If this vector for the diffracted vacuum wave is displaced parallel so that it starts in \(P\), it ends in a point near \(H\) on a line forming the angle \(\varphi\) with the diffracting net planes.

II.2. Mosaic crystals

Highly perfect samples are needed in order to make full use of the high \(k\)-space resolution of a TCD for high-energy synchrotron radiation as discussed above. However, very often, only imperfect single crystals are available as samples, or one is interested in studies of diffuse scattering, which often is rather widely spread in \(k\)-space. In these cases, one may use mosaic crystals as monochromator and analyzer to gain intensity at the expense of resolution. For this purpose, pyrolitic graphite crystals are often used in TCDs for X-rays in the usual wavelength range. The FWHM of the diffraction pattern of these crystals is of the order of \(0.3^\circ\), which in general is too wide in the case of 0.1 Å synchrotron radiation, where the Bragg angles are only of the order of \(2^\circ\). It has been shown that mosaic crystals of high reflectivity with diffraction patterns of FWHM of the order of \(10^\circ\) can be produced by proper thermal annealing of Czochralski-grown silicon.
crystals, in which typically $10^6$ O atoms are dissolved (Schneider, Gonçalves, Rollason, Bonse, Lauer & Zulehner, 1988). The diffraction patterns can be calculated from the mosaic distribution as measured with a double-crystal $\gamma$-ray diffractometer (Schneider & Graf, 1986), on the basis of Darwin's theory of secondary extinction; primary extinction is taken into account via an experimentally determined factor that reduces the effective sample thickness to a value smaller than the geometrical one (Schneider, Nagasawa, Berman, Hastings, Siddons & Zulehner, 1989). The diffraction pattern of such annealed silicon crystals can then be calculated for various sample thicknesses and results are shown in Fig. 7. The peak reflectivity has been normalized to unity and one sees that for 144.7 keV synchrotron radiation the FWHM of the diffraction patterns can be changed by more than a factor of 2 by reducing the geometrical crystal thickness from 10 to 1 mm. Such changes in thickness and thus in FWHM of the diffraction pattern can be realized continuously if a thin mosaic crystal is rotated around the scattering vector. With these mosaic crystals as monochromator and analyzer, the resolution of the TCD can be adapted to the problem under study.

In order to calculate the resolution of a TCD using mosaic crystals as monochromator and analyzer, the Lorentzian distributions $R_M$ and $R_A$ used so far in formula (2) are replaced by the diffraction patterns calculated for the mosaic crystals; results for crystal thicknesses of 10 and 1 mm are shown in Figs. 8(a) and (b). The sample was assumed to be a perfect crystal. For comparison, the resolution function calculated for perfect monochromator and analyzer crystals is shown in Fig. 8(c).

III. Comparison with experimental data

The measurements have been performed with a TCD for high-energy synchrotron radiation, which was built at the NSLS at Brookhaven National Laboratory. First measurements had been performed at CHESS at Cornell (Hastings, Siddons, Schneider & Berman, 1989). The instrument was then transferred to HASYLAB and after some modifications was installed at a dipole and later at a wiggler beam line. All three silicon crystals diffract in nondispersive ($-, +, -$) Laue geometry with a vertical scattering plane. The measurements of the resolution function with three perfect Si 111 crystals in symmetric Laue case have been performed with a new TCD, built at HASYLAB (Bouchard, Kracht, Neumann, Rütt, Schmidt, Poulsen & Schneider, 1994), where the scattering plane is horizontal. The storage ring DORIS was run at an electron energy of either 4.5 or 5.3 GeV. The synchrotron-radiation beam was filtered by a 2 mm-thick water-cooled copper absorber, effectively suppressing radiation with energies below 30 keV. This way, background radiation as well as the heat load on the monochromator crystal was greatly reduced. The intensity of the beam incident on the sample was monitored by means of a scintillation counter measuring the fluorescence from a thin tantalum foil. The scattered photons are measured with an intrinsic germanium solid-state detector with energy resolution

![Fig. 7. Diffraction patterns of annealed Czochralski-grown silicon crystals calculated from a measured mosaic distribution function for the 220 reflection and various sample thicknesses. The photon energy is 144.7 keV. The maximum reflectivity is set equal to 1. The broad curve corresponds to a crystal thickness of 10 mm.](image1)

![Fig. 8. TCD intensity distribution in the vicinity of the 220 reciprocal-lattice point calculated for 144.7 keV synchrotron radiation. Monochromator and analyzer are mosaic crystals of (a) 10 mm and (b) 3 mm thickness. For comparison, the intensity distribution for three perfect crystals is shown in (c). The horizontal scale is stretched by a factor of 20. Contour levels are 1, 0.8, 0.5, 0.2, 0.1, 0.08, 0.05, 0.01, 0.006, 0.004, 0.002 and 0.001.](image2)
$\Delta E/E$ of better than 1% at 100 keV. In order to reduce background due to fluorescence radiation and higher harmonics, a single-channel analyzer electronic window of $\pm 1$ keV was set at the energy of the monochromatic beam.

III.1. Perfect crystals

After alignment of the three perfect silicon crystals on the TCD, the center of the resolution function was determined by iterative scans of the sample and the analyzer crystal. Fig. 9 shows sample and analyzer scans for reflection 111 at 80 keV together with the theoretical distributions calculated with (2.2) for three perfect crystals in symmetric Laue geometry. In order to determine the resolution function, a series of sample scans was performed for different settings of the analyzer crystal. The individual sample scans have been scaled by comparing the analyzer peak with the corresponding value of the analyzer scan presented in Fig. 9(b). With a spline-function interpolation scheme, the resolution function of the TCD has been calculated from the series of scaled sample scans. The result is shown in Fig. 10 and good agreement with the calculated distribution of Fig. 4(b) is obtained. The central resolution is described by the FWHM of the intensity distribution and for 80 keV synchrotron radiation one obtains $\Delta q_z = 1.1 \times 10^{-5} \text{Å}^{-1}$ in a direction perpendicular to $G_{111}$ and $\Delta q_x = 2.2 \times 10^{-4} \text{Å}^{-1}$ parallel to $G_{111}$.

The same procedure has been applied in order to determine the intensity distribution of an asymmetrically cut plane-parallel silicon sample, where the 220 net planes form an angle of $\varphi = 45^\circ$ with the sample surfaces. The result is presented in Fig. 11 and again good agreement with the calculated distribution (Fig. 5) is obtained over an intensity range of five orders of magnitude.

III.2. Mosaic crystals

Fig. 12 shows the k-space intensity distribution measured with 144.7 keV synchrotron radiation in the case where annealed silicon crystals are used as monochromator and analyzer. The sample is a perfect silicon crystal in symmetrical Laue geometry. The

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Fig. 9. (a) Sample and (b) analyzer scan for three symmetrically cut Si 111 crystals and 80 keV synchrotron radiation. The experimental data (dots) are well reproduced by the calculation.

Fig. 10. Intensity distribution of a TCD in the vicinity of the Si 111 reciprocal-lattice point measured with 80 keV synchrotron radiation with three perfect crystals in symmetric Laue geometry. The contour levels are identical to those in Fig. 4(b).

Fig. 11. Measured intensity distribution around the Si 220 reciprocal-lattice point for an asymmetrically cut sample crystal. All parameters are the same as in Fig. 5.
agreement with the calculations presented in Fig. 8(a) is excellent. The FWHM of the intensity distribution is 
\[ \Delta q_y = 1 \times 10^{-4} \text{ Å}^{-1} \] in a direction perpendicular to \( G_{220} \) and 
\[ \Delta q_x = 4 \times 10^{-3} \text{ Å}^{-1} \] parallel to \( G_{220} \), i.e.
the size of the distribution increased by about one order of magnitude compared to the TCD working at the same energy but with a perfect crystal as monochromator and analyzer.

IV. Comparison of monochromator and analyzer streaks at high values of \( q_x \)

Recently, double-crystal rocking curves were measured with 40 and 80 keV synchrotron radiation in dispersion-free Laue-Laue, Laue-Bragg and Bragg-Bragg settings and compared with the results from dynamical theory (Chapman, Hastings, Moulin, Siddons, Garrett, Nachaliel & Dilmanian, 1992). Excess scattering was found in the wings of the diffraction patterns that could not be explained as a sample volume effect alone, although this effect is more important at higher X-ray energies because of beam penetration. It was also found that the excess scattering in the far wings does not completely follow a \( 1/q^2 \) power law, which would indicate thermal diffuse scattering as its origin (Kazimirov, Kovalchuk & Kohn, 1990). The additional scattering which is needed to explain the experimental data could be non-Bragg scattering from the second crystal collected by the rather large aperture of the detector.

For the TCD for high-energy synchrotron radiation, the white synchrotron-radiation beam produces non-Bragg scattering in the first collimator, which can be taken into account by adding a constant background to the intensity distribution \( F(q) \). Model calculations show that this background leads to a slight increase of the intensity of both monochromator and analyzer peaks in the wings of the two streaks (Fig. 13).

Fig. 14 shows experimental sample scans for \( q_x \) values \( \geq 1.2 \times 10^{-2} \text{ Å}^{-1} \) measured with 100 keV synchrotron radiation at the silicon 220 reflection. The analyzer peaks are normalized to unity. In contrast to expectation, the analyzer peak is significantly higher than the monochromator peak and the difference increases with increasing \( q_x \) values. This observation can be explained by considering the non-Bragg scattering from monochromator and analyzer crystals and the finite electronic window limiting the energy range of the photons measured with the solid-state detector.

In addition to Bragg diffraction, the three crystals produce Compton, Rayleigh and thermal diffuse scattering, which are distributed over a much wider angular range limited only by the corresponding collimators of the TCD. For this reason, the sample crystal will transmit an additional wider-spread energy band of low intensity, which, however, affects the shape of the resolution function only slightly in the case of nondispersive TCD settings. Because of its narrow diffraction profile, the analyzer crystal accepts

Fig. 12. Measured intensity distribution near the Si 220 reciprocal-lattice point for 10 mm-thick annealed Czochralski-grown silicon crystals as monochromator and analyzer and a perfect sample crystal. All parameters are the same as in Fig. 8(a).

Fig. 13. Calculation of a sample scan at \( q_x = 6.1 \times 10^{-3} \text{ Å}^{-1} \) with and without a constant background of 5\% in the intensity distribution \( F(q) \) of the incident synchrotron-radiation beam.

Fig. 14. Sample scans for \( q_x = (1.2, 2.4, 3.6) \times 10^{-2} \text{ Å}^{-1} \) measured with 100 keV synchrotron radiation using three perfect silicon crystals reflecting at 220. The analyzer peaks are normalized to 1. The monochromator peak decreases strongly for large-\( q_x \) settings.
only a negligible small part of the non-Bragg scattering from the sample crystal. On the other hand, a large fraction of the non-Bragg scattering from the analyzer is accepted by the aperture $\beta$ of the detector. In the simulation for the given scattering geometry, the additional scattering is a factor of $\sim 10^{-3}$ smaller than the intensity of the analyzer Bragg peak. One thus has to add a constant contribution to the diffraction pattern of the analyzer crystal. This explains the difference between monochromator and analyzer peaks observed in the experimental data for the case where a wide wavelength band is accepted by the counting system. In addition to an increase of the analyzer peak, the monochromator peak decreases if the electronic window of the counting chain of the solid-state detector is reduced. This effect can be explained qualitatively using DuMond (1937) diagrams.

The DuMond diagram is a visualization of Bragg’s law: it is a plot of wavelength $\lambda$ versus diffracting angle $\theta$. Fig. 15(a) shows the DuMond diagram for the nondispersive reference setting of the TCD, i.e. sample and analyzer are set at $\omega(0)_{\text{syn}}$ and $\omega(0)_{\text{syn}}$, respectively. All three crystals are represented by a bar $\Delta \omega_{\text{syn}} = 0.385^\circ$ wide, which corresponds to the FWHM of their individual diffraction patterns for 100 keV synchrotron radiation and silicon 220 shown in Fig. 3. The solid lines at $\lambda_1/\lambda_2$ and $\theta_1/\theta_2$ correspond to the limits of integration of (2.2). The wavelength limitation is due to the electronic window of the detector counting chain, which accepts only a narrow wavelength band of $\Delta \lambda/\lambda \approx \pm 1\%$. The angle limitation is due to the finite angular spread of the intensity distribution transmitted by the first collimator. Fig. 15(b) shows the DuMond diagram for the analyzer peak where the diffraction patterns of monochromator and sample crystals overlap with each other and with the tail of the analyzer diffraction pattern over the full wavelength range $\lambda_2 - \lambda_1$. Fig. 15(c) shows the DuMond diagram for the monochromator peak where the diffraction patterns of the analyzer and the sample crystal overlap with each other and with the tail of the monochromator crystal, however, only over the smaller wavelength range $\lambda_2 - \lambda_1$. Therefore, the monochromator peak is expected to be smaller than the analyzer peak and its center is shifted to smaller wavelengths, which has been observed in the experiment. Fig. 16 shows sample scans performed for $q_x = 6.1 \times 10^{-2} \text{Å}^{-1}$ for two different settings of the electronic window of the counting chain together with the results of model calculations. The agreement between calculation and experimental data is good.

V. Summary

TCD for high-energy synchrotron radiation shows very high $k$-space resolution in nondispersive settings and large samples can be studied because of the high penetration power of the radiation. This diffractometer combines the best resolution in directions perpendicular to the scattering vector obtained by means of double-crystal $\gamma$-ray diffractometry (Schneider & Graf, 1986) with the best resolution parallel to the scattering vector obtained with neutron backscattering spectrometers for large single crystals (Birr,
Heidemann & Alefeld, 1971). It is therefore very well suited to high-resolution studies of structural phase transitions, incommensurate structures and diffuse scattering in general. It has also been applied to characterize the perfection of large imperfect single crystals like Si–Ge gradient crystals (Bouchard, Kouptsidis, Neumann, Schmidt & Schneider, 1993). It is interesting to note that the resolution of the TCD for high-energy synchrotron radiation in directions parallel to the reciprocal-lattice vector of the sample is almost independent of the sample mosaicity, i.e. of the width of the angular distribution of mosaic-block orientations in a real crystal.

Thanks are given to Dr W. Zulehner of Wacker-Chemitronic GmbH, Burghausen, Germany, for kindly providing the silicon single crystals.

References


Fig. 16. (a) Sample scans measured at \( q_x = 6.1 \times 10^{-3} \, \text{Å}^{-1} \) with an electronic window of the counting chain accepting an energy band of 1.5 and 20 keV, respectively. The analyzer peaks (A) have been normalized to the same peak value. The smaller value of the monochromator peak (M) corresponds to the smaller energy band. The simulation in (b) describes the peak heights of the experimental data reasonably well if the non-Bragg-scattering contributions of the first collimator and the analyzer crystal are taken into account. This additional scattering is of the order of \( 10^{-3} \) of the Bragg scattering at \( q_x = q_y = 0 \). Because of the non-Bragg-scattering contributions, the analyzer peak is always larger than the monochromator peak.