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Multiple Laue Rocking Curves

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Abstract

Multiple Laue rocking curves of perfect crystals show a narrow central peak with a width of some 10^{-3} s arc and an additional oscillatory structure. The finite structure of these curves is analyzed for two- and three-crystal Laue arrangements. These profiles can be used for precise determinations of structure factors and for an extension of small-angle scattering experiments to the extreme small-angle regime where large objects and long-range particle correlations become visible. An extremely high angular resolution can be achieved without significant reduction of the intensity, owing to a decoupling of the angular resolution from angular width of the beam. The analytically calculated rocking curves are compared to numerical results and to experimental results and show good agreement with both.

Introduction

The dynamical diffraction of X-rays and neutrons on multiple-perfect-crystal arrangements has been studied extensively during the past years. Monolithic and polylithic, plane and bent, static and vibrating systems have been discussed. Dynamical focusing

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effects appearing in the spatial intensity profile have been studied for X-rays and neutrons (Indenbom, Suvorov & Slobodetskii, 1976; Bauspiess, Bonse & Graeff, 1976; Aladzhadzyan, Bezirganyan, Semerdzhyan & Vardanyan, 1977; Bonse, 1979; Petrascheck, 1979). Multiple Laue rocking curves also show a focusing effect on the angular scale in the form of a very narrow central peak (Bonse, Graeff, Teworte & Rauch, 1977; Bonse, Graeff & Rauch, 1979; Bonse & Teworte, 1980, 1982*a*, *b*; Cusatis, Hart & Siddons, 1983; Rauch, Kischko, Petrascheck & Bonse, 1983). The width of the central peak is of the order of d_{hkl}/t, where d_{hkl} is the lattice spacing and t is the thickness of the crystals. This peak can be resolved either by rotating a wedge-shaped material around the beam axis of a monolithically designed multiplate system (Bonse, Graeff & Rauch, 1979) or by a very precise mechanical rotation stage for a separated system (Bonse & Teworte, 1982*a*, *b*).

The resolution in momentum space lies in the range 10^{-7}–10^{-9} Å^{-1} and therefore small-angle scattering experiments can be extended to this regime where macroscopic objects, inhomogeneities or long-range interparticle interferences become observable. In a test experiment the Frauenhofer diffraction of slow neutrons ($\lambda = 1{\cdot}8$ Å) on macroscopic slits (2·5 and 5 mm) has been observed (Rauch, Kischko, Petrascheck & Bonse, 1983). There exists a decoupling between the intensity and the angular resolution and therefore one can use incident beams with a large geometrical cross section and quite broad distributions in wavelength and angle.

According to the physics of small-angle scattering (*e.g.* Glatter & Kratky, 1982) large objects and interparticle correlations over long distances become observable at a very low momentum transfer regime which can be measured very economically by the technique of multiple Laue reflections. The advantages of crystal collimation compared to slit collimation have been discussed for the Bonse–Hart camera (Bonse & Hart, 1965) by Kratky & Leopold (1970) and they are even more convincing for the multiple Laue camera if appropriately large objects have to be investigated. Because transmission geometry has to be used neutrons offer the additional advance of low absorption.

Here we present analytical results for multiple Laue rocking curves, which serve for a deeper understanding of the related phenomena. Various terms appear in these equations which have a quite different angular dependence and a different sensitivity to variations of the crystal thickness or of the wavelength spread.

Multiple Laue diffraction on parallel crystal plates

Successive Laue diffraction on parallel crystals is described by the well known dynamical diffraction profiles (Zachariasen, 1967; Rauch & Petrascheck, 1978; Sears, 1978). It is assumed that the diffracted and the transmitted beams are well separated, so one can consider only the diffracted waves, as is shown in Fig. 1(*a*). The reflectivity of a parallel crystal slab is given by

$$P(t, y) = \frac{\sin^2[A(1+y^2)^{1/2}]}{1+y^2} \quad (1)$$

for non-absorbing crystals. A is the reduced thickness, given by the thickness t and the *Pendellösung* length Δ_0:

$$A = \frac{\pi t}{\Delta_0} = \frac{\lambda |b_c F(\mathbf{G})| t}{v_c (\cos\gamma \cos\gamma_G)^{1/2}}$$

and y is the reduced angular deviation

$$\begin{aligned} y = {} & 2\pi v_c \cos\gamma \sin 2\theta_B(\theta_B - \theta) \\ & + \lambda^2 b_c F(0)(\cos\gamma - \cos\gamma_G) \\ & \times [2\lambda^2 |b_c F(\mathbf{G})| (\cos\gamma \cos\gamma_G)^{1/2}]^{-1}. \end{aligned}$$

b_c is the coherent scattering length, $F(\mathbf{G})$ is the geometrical structure factor, v_c is the volume of the unit cell, λ is the wavelength and γ, γ_G are the angles of incidence and of reflection, respectively. $(\theta - \theta_B)$ is the deviation of the incident wave from the exact Bragg angle.

The integrated intensity of n successive Laue diffractions on crystal slabs of thickness t is given by

$$R^{(n)} = \int_{-\infty}^{\infty} \mathrm{d}y [P(t, y)]^n. \quad (2)$$

For reflection on a single-crystal plate one obtains Waller's formula. For thick crystals the Bessel function J_0 may be replaced by its asymptotic expansion (DeMarco & Weiss, 1965)

$$\begin{aligned} R^{(1)} = {} & \int_0^{2A} \mathrm{d}t\, J_0(t) \\ \simeq {} & \frac{\pi}{2}\left\{1 - \frac{1}{(\pi A)^{1/2}}\left[\cos\left(2A + \frac{\pi}{4}\right) \right.\right. \\ & \left.\left. + \frac{5}{8}\,\frac{\sin(2A + \pi/4)}{2A}\right]\right\}, \end{aligned} \quad (3)$$

which holds for $t/\Delta_0 > 1$.

In a two-crystal arrangement we obtain approximately

$$\begin{aligned} R^{(2)} \simeq {} & \frac{3\pi}{16} - \frac{1}{2}\left(\frac{\pi}{A}\right)^{1/2} \\ & \times \left[\cos\left(2A + \frac{\pi}{4}\right) + \frac{13}{8}\,\frac{\sin(2A + \pi/4)}{2A}\right] \\ & + \frac{1}{8}\left(\frac{\pi}{2A}\right)^{1/2}\left[\cos\left(4A + \frac{\pi}{4}\right) + \frac{13}{8}\,\frac{\sin(4A + \pi/4)}{4A}\right]; \end{aligned} \quad (4)$$

and for $n=3$

$$R^{(3)} \simeq \frac{15\pi}{128} - \frac{15}{32}\left(\frac{\pi}{A}\right)^{1/2} \times \left[\cos\left(2A+\frac{\pi}{4}\right) + \frac{21}{8}\frac{\sin(2A+\pi/4)}{2A}\right] + \frac{6}{32}\left(\frac{\pi}{2A}\right)^{1/2}\left[\cos\left(4A+\frac{\pi}{4}\right)+\frac{21}{8}\frac{\sin(4A+\pi/4)}{4A}\right] - \frac{1}{32}\left(\frac{\pi}{3A}\right)^{1/2}\left[\cos\left(6A+\frac{\pi}{4}\right)+\frac{21}{8}\frac{\sin(6A+\pi/4)}{6A}\right]. \tag{5}$$

$R^{(1)}$, $R^{(2)}$, $R^{(3)}$ and $R^{(5)}$ are plotted in Fig. 1(*b*) *vs* t/Δ_0. The intensity oscillates around an average value given by the first term of (3)–(5). The averaged intensity decreases whereas the height of the oscillations remains nearly unchanged. Also, the frequency is essentially determined by $2A = 2\pi t/\Delta_0$, as for one single crystal. The oscillations of higher frequency modify only the form of the oscillations. Thus the dependence on the thickness becomes more important for increasing the number of reflections and has to be considered in the calculation of the rocking curves for two- or three-crystal spectrometers.

Two-crystal spectrometer

The fine structure of the Laue-case rocking curves have been investigated during the last few years. First, Bonse, Graeff, Teworte & Rauch (1977) pointed out that the intrinsic rocking curves of a perfect-crystal spectrometer in a non-dispersive arrangement should have an oscillatory structure

$$R^{(2)}(v,t) = \int_{-\infty}^{\infty} \mathrm{d}y\, P(t,y)P(t,y+v) = R_a + R_p + R_t + R_0 \tag{6}$$

with its maximum value for $v=0$. The oscillatory structure of $P(t,y)$ and $P(t,y+v)$ becomes different for increasing v which reduces the contributions of large y values. Fig. 2 shows the numerical calculations for two thicknesses which are near the values of minimum and maximum intensity. We may rewrite (6) by replacing the powers of the trigonometric functions with the multiple of their arguments.

Thus, the different contributions to the integral of (6) read:

$$R_a = \frac{1}{4}\int_{-\infty}^{\infty} \mathrm{d}y \frac{1}{(1+y^2)[1+(y+v)^2]} = \frac{\pi}{8(1+v^2/4)}, \tag{7}$$

which is the convolution of the two average (Fig. 2(*a*)) reflection curves, the Lorentzian of (7). The next term

$$R_p = \frac{1}{8}\int_{-\infty}^{\infty} \mathrm{d}y \frac{\cos\{2A[1+(y+v)^2]^{1/2} - 2A(1+y^2)^{1/2}\}}{(1+y^2)[1+(y+v)^2]} \simeq \frac{\pi}{8}\frac{J_1(2Av)}{2Av} \tag{8}$$

describes the central peak (*b*) of Fig. 2.

The half width of the central resonance is $v_H = 2{\cdot}215/A$. A comparison with the experiments of Bonse, Graeff & Rauch (1979) gives a good agreement between the experimental value of $v_H = 7\times10^{-3}$ s arc and the theoretical value of $6{\cdot}6\times10^{-3}$ s arc.

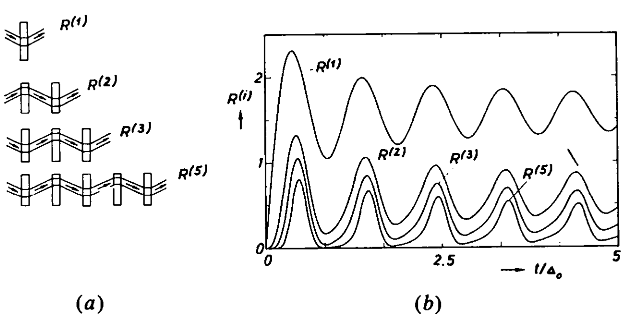

Fig. 1. (*a*) Scheme of the experimental situation and (*b*) integral reflectivities for multiple Laue rocking curves as a function of the crystal thickness.

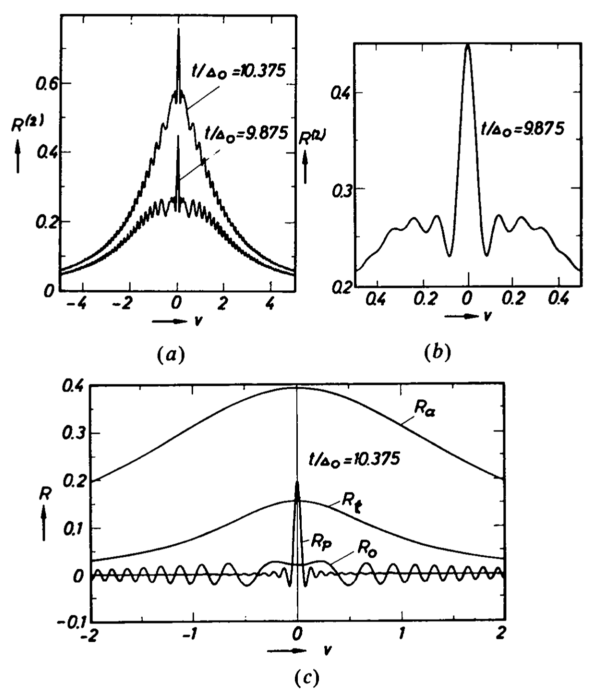

Fig. 2. (*a*) Analytically and numerically calculated double Laue rocking curves for two different crystal thicknesses. The central part (*b*) and the various contributions (*c*) are shown separately.

In a more detailed study of the reflection curve one may calculate further terms:

$$R_t = -\frac{1}{2}\int_{-\infty}^{\infty} dy \frac{\cos[2A(1+y^2)^{1/2}]}{(1+y^2)[1+(y+v)^2]}$$
$$\simeq -\frac{1}{2}\left(\frac{\pi}{A}\right)^{1/2}\frac{\cos(2A+\pi/4)}{1+v^2}$$
$$-\frac{1}{2A}\left(\frac{\pi}{A}\right)^{1/2}\frac{\sin(2A+\pi/4)}{1+v^2}$$
$$\times\left[\frac{5}{16}+\frac{1}{2(1+v^2)}-\frac{2v^2}{(1+v^2)^2}\right], \quad (9)$$

where R_t is a thickness-dependent (t) contribution which lowers or raises the broad background. Equation (9) is evaluated by the method of the stationary phase – as are most of the following integrals. This method is appropriate for oscillating integrals of the type of (9) and one obtains very accurate results for thick crystals.

The corrections in (9) of the order of $1/A$ are obtained also by the method of the stationary phase, similar to the expansion of Gaussian integrals. The series have only semiconvergent character. For most cases we may neglect these corrections. The oscillating structure of Fig. 2 is determined by

$$R_0 = \frac{1}{8}\int_{-\infty}^{\infty} dy \frac{\cos(2A\{[1+(y+v)^2]^{1/2}+(1+y^2)^{1/2}\})}{(1+y^2)[1+(y+v)^2]}$$
$$\simeq \frac{1}{8}\left(\frac{\pi}{2A}\right)^{1/2}\frac{\cos[4A(1+v^2/4)^{1/2}+\pi/4]}{(1+v^2/4)^{5/4}}$$
$$+\frac{1}{8\cdot 32A}\left(\frac{\pi}{2A}\right)^{1/2}(13-v^2)$$
$$\times\frac{\sin[4A(1+v^2/4)^{1/2}+\pi/4]}{(1+v^2/4)^{7/4}}. \quad (10)$$

The different contributions to the intensity, (7)–(10), are displayed in Fig. 2 for $t = 10{\cdot}375\ \Delta_0$. The corrections in (9) and (10) are very small. Thus, our analytical results are, within the accuracy of the drawings, in agreement with the numerical calculated intensity profiles.

Absorbing crystals

Although absorption is less important for neutrons than for X-rays its influence can be included. In the symmetric case we have to replace (1) by (Zachariasen, 1967; Rauch & Petrascheck, 1978)

$$P(t, y) = e^{-\Sigma t_e}\left|\frac{\sin[A(\nu^2+y^2)^{1/2}]}{(\nu^2+y^2)^{1/2}}\right|^2, \quad (11)$$

where $t_e = t/\cos\theta_B$ is the effective thickness. Σ is the macroscopic attenuation cross section and $\nu^2 = V(-\mathbf{G})V(\mathbf{G})/|V(-\mathbf{G})V(\mathbf{G})| \simeq 1-2i\kappa$, where $V(\mathbf{G})$ is the Fourier transform of the interaction potential. As in the non-absorbing case one can separate $R^{(2)}(v)$ into the different contributions and one obtains

$$R^{(1)} \simeq e^{-\Sigma t_e}\left\{\frac{\pi}{16(1+v^2/4)}\left\{1+I_0\left(\frac{4\kappa A}{(1+v^2/4)^{1/2}}\right)\right.\right.$$
$$\left.+\left(1+\frac{v^2}{4}\right)I_2\left[\frac{4\kappa A}{(1+v^2/4)^{1/2}}\right]\right\}$$
$$+\frac{\pi}{8}\frac{J_1(2Av)}{2Av}-\frac{1}{2}\left(\frac{\pi}{A}\right)^{1/2}\frac{\cos(2A+\pi/4)}{1+v^2}$$
$$\times\cosh\left[\frac{2\kappa A}{(1+v^2/4)^{1/2}}\right]+\frac{1}{8}\left(\frac{\pi}{2A}\right)^{1/2}$$
$$\left.\times\frac{\cos[4A(1+v^2/4)^{1/2}+\pi/4]}{(1+v^2/4)^{5/4}}\right\}. \quad (12)$$

I_0 and I_2 are the modified Bessel functions. The first term contains the anomalous weak absorbed parts and remains in thick crystals, whereas the central peak is attenuated by the normal absorption.

It is already known from the spatial intensity profiles in two- or three-crystal arrangements (Petrascheck, 1979) that focusing phenomena are strongly affected by the absorption because such effects are related to the interference of strong and weak absorbed waves. Thus it is necessary for the observation of the rocking curves to keep $\Sigma t_e < 1$. Very accurate measurements of such rocking curves have been done (Bonse & Teworte, 1980, 1982*a*, *b*).

Triple Laue case

For the triple Laue rocking curves the rotation of the crystals (v, w) or the beam deflection between the crystals can be in the same or in the opposite direction. For such arrangements the intensity is

$$R^{(3)}(t, v, w) = \int_{-\infty}^{\infty} dy\, P(t, y)P(t, y-v)P(t, y-v-w)$$
$$= R_a + R_p + R_t + R_0. \quad (13)$$

Two different cases are discussed in the following: $v = -w$ and $v = w$.

For $v = -w$ the diffraction pattern of a Laue two-crystal spectrometer is folded with the diffraction pattern of one crystal plate. As in the case of two crystals we separate (13) into the different contributions. First we consider the rocking curve which is obtained from the averaged rocking curves

$$R_a = \frac{3\pi}{128}\frac{3+v^2/4}{(1+v^2/4)^2}. \quad (14)$$

The central peak is given by

$$R_p = \frac{1}{8} \int_{-\infty}^{\infty} dy \frac{\cos(2A\{[1+(y+v)^2]^{1/2} - (1+y^2)^{1/2}\})}{(1+y^2)^2[1+(y+v)^2]}$$

$$\simeq \frac{3\pi}{8} \frac{J_2(2Av)}{(2Av)^2}. \qquad (15)$$

For thick crystals the height of this peak can be estimated from the difference of the maximum of $R^{(3)}$ which appears for $v = w = 0$ as $15\pi/128$ [see (5)] and the maximum of the rocking curve between a monolithic double crystal and a third crystal plate which is $9\pi/128$ resulting from (14). The difference of $6\pi/128$ belongs to the central peak (Rauch, Kischko, Petrascheck & Bonse, 1983).

If the incident waves have a sufficiently broad wavelength distribution, the thickness-dependent terms R_t and the oscillating terms R_0 vanish by averaging and the rocking curve is obtained from (14) and (15). The width of the central peak is given by $v_H = 2{\cdot}755/A$.

For higher-resolution work the thickness-dependent terms may appear and they are treated within the stationary-phase approximation.* In Fig. 3 the different contributions to the intensity are displayed.

* An Appendix containing this detailed information has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39215 (3 pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

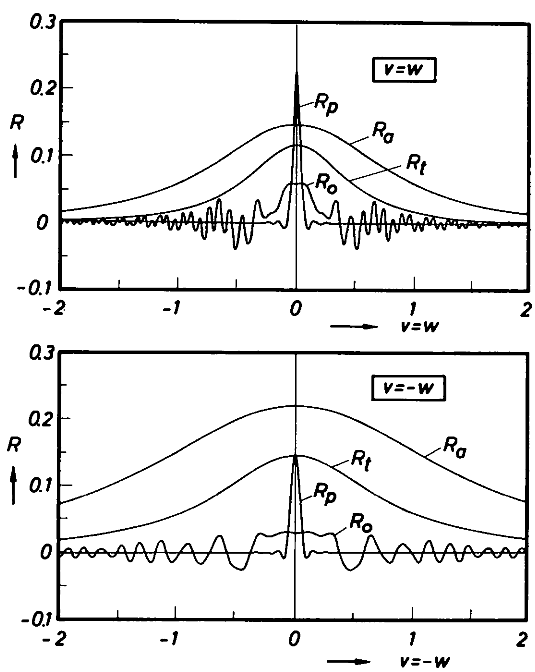

Fig. 3. The different contributions to the triple Laue rocking curves for $t/\Delta_0 = 10{\cdot}375$.

The numerical integrated intensites are again in agreement with our analytical results within the accuracy of drawings.

Now we draw our attention to the other case $v = w$. R_a is obtained by averaging each crystal plate separately

$$R_a = \frac{3\pi}{64} \frac{1}{(1+v^2/4)(1+v^2)}. \qquad (16)$$

The central peak is now given by

$$R_p = \frac{1}{16} \int_{-\infty}^{\infty} dy \, \{2\cos\{2A[[1+(y+v)^2]^{1/2} - (1+y^2)^{1/2}]\}$$
$$+ \cos(2A\{[1+(y+v)^2]^{1/2} - [1+(y-v)^2)]^{1/2}\})\}$$
$$\times \{[1+(y+v)^2](1+y^2)[1+(y-v)^2]\}^{-1}$$
$$\simeq \frac{3\pi}{16}\left[2\frac{J_2(2Av)}{(2Av)^2} + \frac{J_2(4Av)}{(4Av)^2}\right]. \qquad (17)$$

As in the former case the height of this peak is given by the difference of the value of $R^{(3)}$ [(5)] and the value of (16) for $v = 0$. Here it is $9\pi/128$. Thus, the height of the peak dominates the background by a factor of $\frac{3}{2}$. The half width is given by $v_H = 2{\cdot}10/A$. As in the former case the thickness-dependent terms R_t and R_0 have been treated in the same manner.

For practical application one has to include the spread of the wavelength distribution. Assuming a Gaussian distribution this causes for R_t and R_0 an attenuation factor proportional to $(A\Delta\lambda/\lambda)^2$ which strongly reduces these contributions, whereas the attenuation factor for R_p is to first order proportional to $(\Delta\lambda/\lambda)^2$ only and therefore this term remains nearly unchanged. This is in agreement with numerical calculations (Bonse, Graeff, Teworte & Rauch, 1977).

Related measurements have been reported (Rauch, Kischko, Petrascheck & Bonse, 1983). The spectrometer is based on the monolithically designed neutron interferometer (Rauch, Treimer & Bonse, 1974) and the deflection of the beams between the perfect-crystal plates is achieved by wedges rotated around the beam axis (Fig. 4(a)). The beam deflection within the horizontal plane is given in this case as

$$\delta = \delta_0 \sin\alpha = \frac{Nb_c\lambda^2}{h} \tan\frac{\beta}{2} \sin\alpha,$$

where α is the rotation angle around the beam axis, β is the wedge angle and N and b_c are the particle density and the coherent scattering length of the wedge material. Characteristic results for the case $v = -w$ and an aluminum wedge having $\beta = 13°$ and for $v = w$ measured with an aluminum wedge with $\beta = 22°$ are shown in Figs. 4(b) and (c), respectively. The measurements have been made at a neutron wavelength $\lambda = 1{\cdot}835$Å, a symmetrical 220 reflection on perfect silicon crystals with thicknesses of $t/\Delta_0 = 60{\cdot}60$. The experiments have been performed using an entrance slit wider than the width of the Borrmann fan to justify a comparison with the results of the plane-wave theory.

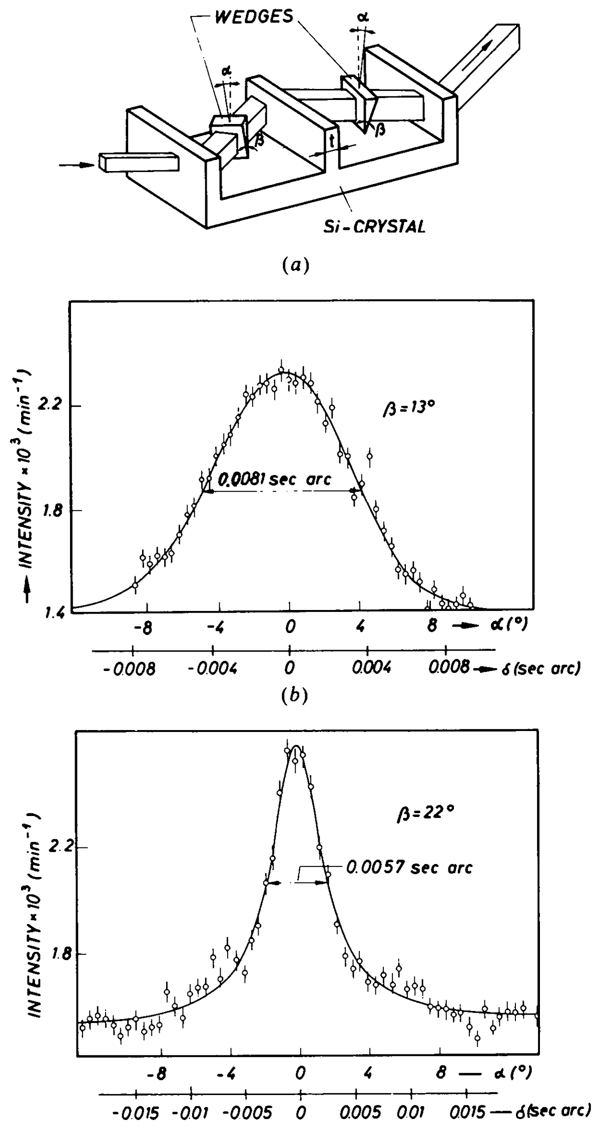

Fig. 4. (a) Sketch of the experimental set-up and characteristic results for the cases (b) $v = -w$ and (c) $v = w$ measured with different aluminum wedges.

Before one compares the experimental rocking curves with calculated results one has to consider the influence of the wavelength spread, variations of the crystal thicknesses *etc.*, which cause a variation of the reduced thickness of about $\Delta A/A \sim 1\%$. Thus, the thickness-dependent terms R_t and R_0 become strongly damped owing to the wavelength spread of about $\Delta\lambda/\lambda \sim 1\%$ and one can compare with R_a and R_p directly. The experimental full widths at half maximum for the case $v = -w$ is 0·0081 s of arc and for $v = w$ it is 0·0057 s of arc, which has to be compared to the calculated values of 0·0079 and 0·0060 s of arc, respectively.

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