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A Method of the Intensity Distribution Measurement of the Small-Angle Scattering of X-rays Eliminating the Influence of the Imperfect Collimation

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A new method is described for the intensity distribution measurement of the diffuse radially symmetrical small-angle scattering pattern distorted by the finite height of the direct beam of negligible width. This method consists in measuring the values of the intensities integrated along the lines parallel to the trace of the direct beam in the plane of observation. The true radial intensity distribution (corresponding to the use of the direct beam of point-like cross section) derived from the distribution of these integrated intensities is unaffected both by the intensity distribution along the height of the direct beam and by the finite height of the exploring slit of the detector used for the intensity measurements.

Introduction

The finite dimensions of the direct beam cross section in small-angle cameras imply a distortion of the true diffraction pattern which would be obtained with a beam defined by the pinhole system of infinitely small radius. By using the bent crystal monochromator, which focuses the beam into a line in the plane of observation, the distortion of the pattern due to the beam width is often sufficiently reduced (Guinier, 1939; Guinier & Fournet, 1948). Then there remains the distortion due to the beam height.

For the most common case of the radially symmetrical true intensity distribution, the intensity $J(x)$ measured in a point P_x on the equator line is related to the true intensity $I(x)$ by the equation

$$J(x) = \int_{-\infty}^{+\infty} i(t) I(\sqrt{(x^2+t^2)})\, dt\,, \tag{1}$$

which follows from Fig. 1. In this figure, the trace of the direct beam in the plane of observation lies in the t-axis and the intensity distribution of the direct beam is given by the function $i(t)$.

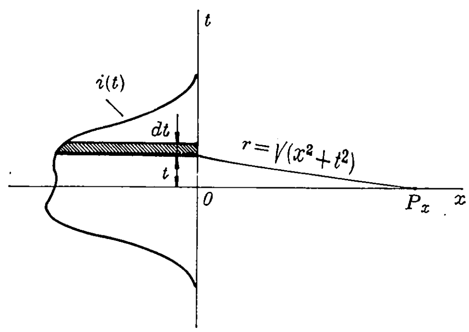

Fig. 1. Measurement of the intensity scattered into a point P_x on the equator line. The notation is explained in the text.

A rigorous solution of equation (1) for $I(x)$ was given only in one particular case of the direct beam of uniform intensity and infinite height by DuMond (1947) and by Guinier & Fournet (1947). In this case the equation (1) takes the form

$$J(x) = c\int_{-\infty}^{+\infty} I(\sqrt{(x^2+t^2)})\, dt\,, \tag{2}$$

since $i(t) = c$ for all values of t. The solution of (2) for $I(x)$ is then according to Guinier & Fournet given by

$$I(x) = -\frac{1}{\pi c}\int_0^{\infty} \frac{J'(\sqrt{(x^2+u^2)})}{\sqrt{(x^2+u^2)}}\, du\,, \tag{3}$$

where

$$J'(\sqrt{(x^2+u^2)}) = \frac{dJ(\sqrt{(x^2+u^2)})}{d\sqrt{(x^2+u^2)}} \tag{4}$$

and u represents an auxiliary variable of integration. This method can be applied only if the beam is of constant intensity along a distance at least twice that corresponding to the angle of diffraction, the diffracted intensity for which practically disappears.

For the case of a direct beam of uniform intensity and finite height, equation (1) was solved by Kratky, Porod & Kahovec (1951) to a good approximation. Other approximate correction methods were put forward by Guinier & Fournet (1947) and by Franklin (1950) for the beam of small height and uniform intensity.

The intensity distribution measured along the equator line must be corrected for the finite height of the slit of the counter or of the photometer used for the intensity measurements. This correction is negligible in the case of 'infinite' uniform direct beam when this height is sufficiently small (DuMond, 1947). For a certain ratio of the beam to the receiving slit height an approximate correction method was given by Gerold (1957).

The true intensity distribution $I(x)$ derived from the intensity measured according to the method described in this paper seems to be unaffected both by the form of the direct beam intensity distribution $i(t)$ and by the height of the receiving slit of the detector.

Description of the method

The basic idea of the proposed method is that the total intensity scattered into the line parallel to the direct beam height is proportional only to the total intensity of the direct beam and is unaffected by its intensity distribution.

This follows from the fact that the resulting small-angle pattern can be thought to be formed by superposing the true diffuse intensity haloes with their centers continually distributed along the trace of the direct beam in the plane of observation. The intensity of the individual haloes are proportional to the intensity of the direct beam in their centers. It is then obvious that by varying the intensity distribution of the direct beam with its total intensity kept constant, the profile of the scattered intensity along any line parallel to the trace of the direct beam changes too but the total intensity scattered into that line remains constant. Without changing the integrated intensity scattered into that line, the total intensity of the direct beam can be thus considered to be concentrated into an arbitrary point on the trace of the real direct beam. This corresponds to the use of a direct beam of a point-like cross section, the intensity of which equals to the total intensity of the real beam. By using this direct beam and by shifting uniformly the detector with a point aperture along a line parallel to the trace of the real direct beam, the same intensity contributions are recorded as by measuring the scattered intensity in an arbitrary point on the same line and by a parallel shift of this direct beam with the same velocity along the trace of the real direct beam. The second case is obviously analogous to the measurement of the scattered intensity in one point by using an infinite direct beam of uniform intensity.

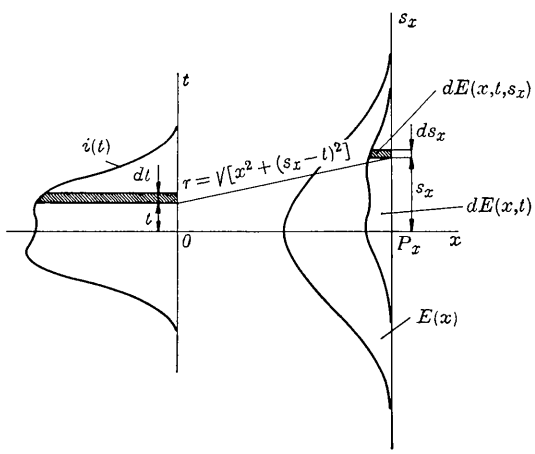

Fig. 2. Measurement of the intensity scattered into a line (s_x-axis) parallel to the trace of the direct beam (t-axis). The notation is explained in the text.

These arguments lead thus to the important result that by measuring the integrated scattered intensity along various lines parallel to the trace of the direct beam the obtained intensity distribution $E(x)$ is identical with the intensity distribution $J(x)$ measured by the usual technique in the points on the equator line by using the infinite direct beam of uniform intensity equal in each point to the total intensity of the real direct beam. Because of this equivalence the true intensity distribution $I(x)$ can be derived from $E(x)$ by the rigorous treatment of Guinier & Fournet (1947) mentioned in the introduction. This is then simply given by the equation (3) in which $E(x)$ is substituted for $J(x)$ and c is put equal to the total direct beam intensity.

This result obtained in an intuitive manner can be easily verified by the calculation. Let the distribution of direct beam intensity be given again by $i(t)$. The intensity of that part of the direct beam which after passing through the sample intersects the plane of observation in an element dt in height t is schematically represented in Fig. 2 by the corresponding shadowed region. Let us consider the contribution of this direct beam element to the intensity scattered into the element ds_x of the s_x-axis, the s_x-axis being situated in the plane of observation, parallel to and distanced by x from the t-axis. This intensity contribution is indicated in Fig. 2 by another shadowed region in the height s_x on that axis. Taking into consideration that the distance of the element ds_x from the element dt is $r=\sqrt{[x^2+(s_x-t)^2]}$, the amount of this diffracted intensity contribution expressed in terms of the true intensity function $I(r)$ and the direct beam intensity $i(t)$ will be given as follows:

$$dE(x, t, s_x)=i(t)I(\sqrt{[x^2+(s_x-t)^2]})\,dt\,ds_x\,.$$

The intensity contribution of that direct beam element after its passing through the sample into the whole length of the s_x-axis is then given by

$$dE(x, t) = i(t)\,dt\int_{-\infty}^{+\infty} I(\sqrt{[x^2+(s_x-t)^2]})\,ds_x\,.$$

The latter expression can be rewritten by substituting w for s_x-t in the integral in the form

$$dE(x, t) = i(t)\,dt\int_{-\infty}^{+\infty} I(\sqrt{(x^2+w^2)})\,dw\,.$$

The total intensity scattered by the whole irradiated volume of the sample into the s_x-axis is then simply

$$E(x) = \int_{-\infty}^{+\infty} i(t)\,dt\int_{-\infty}^{+\infty} I(\sqrt{(x^2+s_x^2)})\,ds_x\,. \qquad (5)$$

In (5) the notation w for the integration variable in the second integral was altered for s_x. By comparing (5) with (2) a similar expression to (3) can be immediately written for $I(x)$:

$$I(x) = -\frac{1}{\pi\int_{-\infty}^{+\infty} i(t)\,dt}\int_0^\infty \frac{E'(\sqrt{(x^2+u^2)})}{\sqrt{(x^2+u^2)}}\,du\,, \qquad (6)$$

where analogous definition (4) holds for $E'(\sqrt{(x^2+u^2)})$. The total direct-beam intensity given by the first integral on the right hand side of equation (5) need not be known for relative measurements of small-angle scattered intensity.

It is furthermore obvious that the true intensity distribution $I(x)$ derived from $E(x)$ will not be influenced by the height of the receiving slit of the counter used for the intensity measurements. This follows from the fact that the values of $E(x)$ are obtained by integrating the scattered intensity in the direction of the larger dimension of the counter slit. This height thus represents only a factor enlarging or reducing all measured intensity values in the same proportion. By measuring the diffuse intensity from small-angle diffraction photographs, the height of the photometer exploring slit must be chosen small enough for the density to be considered approximately constant in all points along the slit height for each setting.

It should be noted that for small scattering angles the variation of the sample thickness effects the values of $E(x)$ approximately in the same way as the modulation of $i(t)$ so that it introduces only a little error into the derivation of $I(x)$.

For practical measurement of the small-angle scattered intensity by this method, the counter assembly should be provided with the automatic translatory movement of constant velocity perpendicular to the counter axis and parallel to the large dimension of the slit. In this way, the scattered intensity is integrated along a particular s_x-axis. After every integration of intensity along the s_x-axis the counter is turned about an axis passing through the sample and parallel to the large dimension of the counter slit to perform the same measurement with x altered. The interval scanned by the counter along the s_x-axis must be chosen sufficiently large for the scattered intensity to achieve its background value in the whole length of the entrance slit in the upper and lower positions of the counter. The background intensity should be subtracted in the usual manner. In special cases when the vertical extension of the measurable small-angle scattered intensity does not exceed the allowed height of the entrance-slit opening, the counter itself integrates the scattered intensity along the s_x-axis, the translatory movement being not necessary. The background intensity will be subtracted simply after shifting the counter into the background intensity region.

Another kind of comparison of the measured and calculated angular intensity distribution of small-angle X-ray scattering should be mentioned in connection with the method described. The most easy theoretical calculations of the angular intensity distribution for various systems giving the small-angle scattering are claimed to be those which can be compared with the true scattering function $I(x)$. As it was shown by Schmidt (1955), the intensity distribution corresponding to the small-angle pattern, distorted by using the infinite uniform line-like direct beam, is almost as easy to calculate as the perfect collimation function. These 'slit-corrected functions' can thus be immediately compared to the measured values of $E(x)$ without the necessity of the somewhat tedious conversion of $E(x)$ to $I(x)$ by the relation (6).

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