Texture of Crystalline Solids, 1981, Vol. 4, pp. 159-169
0390-7951/81/0403-0159$06.50/0

Printed in the United States of America

A METHOD FOR OBTAINING THE COMPLETE POLE FIGURE WITH A SINGLE SAMPLE USING THE SCHULZ TECHNIQUE

M. ORTIZ and J. D. HERMIDA

Comisión Nacional de Energía Atómica,
Departamento de Materiales,
1429 Buenos Aires, Argentina

(Received August 18, 1980)

Abstract. A theoretical expression, which allows the diffracted intensities to be corrected for defocusing, is derived from the convolution over the geometrical defocusing width of a diffraction peak fitted by a Cauchy function. Alignment errors are taken into account, and a good agreement between theoretical curves and experimental points is established.

INTRODUCTION

The defocusing phenomenon in the Schulz technique has been extensively studied, among others by Schulz,[1] Chernock and Beck,[2] Tenckhoff,[3] etc. Basically, it can be described as a broadening of the diffraction peak, due to the irradiated zone displacement from the focusing plane, when the sample is tilted. In the receiving plane the maximum geometrical defocusing broadening is given by (Tenckhoff[3])

$$WD = 2h\,tg\alpha\,\cos\theta$$

where h is the beam height on the sample, α is the tilt angle and 2θ is the diffraction angle.

When WD becomes larger than the receiving slit width, which will certainly occur starting from a certain angle, there will be an intensity loss that should be taken into account for a correct evaluation of the results to be carried out.

The usual procedure is to make a reliable random sample and normalize the intensities by the random sample intensities for each inclination. However, it is not always possible to obtain a reliable random sample and, anyway, the necessity to make a new one for each material turns to be convenient the search of other solution.

Tenckhoff[3] proposed a Gaussian fitting of the defocused diffraction peaks, but this approximation is not possible starting from $\alpha = 75°$, when the peaks begin to show such a flat shape that cannot be fitted by Gaussian functions without introducing large errors (see Figure 8).

The correct way to solve the problem is to express the broadened peak as the convolution of the diffraction peak over the whole geometrical defocusing width WD (Segmüller[4]).

$$I(x,\alpha) = 1/WD \int_{-WD/2}^{WD/2} i(\tau-x)\ d\tau \qquad (1)$$

where $i(x)$ is the intensity profile and x is a distance defined in the receiving plane starting from the zero determined by the diffraction angle. The total intensity, collected by the receiving slit width WR, is given by

$$I_T(\alpha) = 1/WD \int_{-WR/2}^{WR/2} dx \int_{-WD/2}^{WD/2} i(\tau-x)\ d\tau \qquad (2)$$

Then, the intensity loss can be expressed as

$$R(\alpha) = I_T(\alpha)/I_T(\alpha=0) \qquad (3)$$

where

$$I_T(\alpha=0) = \int_{-WR/2}^{WR/2} i(x)\ dx$$

The double numerical integration of Equation (2) puts in evidence the necessity of dividing the peak into a large number of intervals to determine with sufficient accuracy the intensity profile, which undoubtedly is a very laborious task in a standard texture goniometer. If analytical functions were used to fit the diffraction peaks this experimental difficulty could be overcome.

THEORY

In normal focusing conditions of the diffractometer, the intensity profile of the diffraction peak results from the convolution of an instrumental profile with the profile arising from the microstructural state of the sample. In plastically deformed materials, this microstructural profile is in turn the convolution of the profiles corresponding to the two fundamental causes of broadening: smallness of coherent domains and microstrains. The instrumental profile, in standard conditions of the diffractometer, is approximately Cauchy, the profile corresponding to the small size of domains can be represented by a convolution of one or more Gaussian and Cauchy functions, whereas the profile arising from microstrains is more nearly Gaussian (Langford[5]). Then, it is evident that Cauchy and Gaussian functions are limiting cases for an experimental profile.

In this work Equations (2) and (3) are used, but the intensity profiles are fitted by Gaussian and Cauchy functions. The latter has the advantage of allowing Equation (2) to be solved analytically. The half-widths of the diffractions peaks can be used to define both fittings.

The Cauchy fitting as a function of a distance x defined in the receiving plane can be expressed as

$$i(x) = i_m\{1/(1+K^2x^2) + 0.5/[1+K^2(x-\Delta x_{1,2})^2]\}$$

where $K = 1/x_{\frac{1}{2}}$, with $x_{\frac{1}{2}}$ being one half the half-width of the diffraction peak, and $\Delta x_{\frac{1}{2}}$ the distance between $K_{\alpha 1}$ and $K_{\alpha 2}$ peaks in the receiving plane.

According to Ortiz and Hermida[6] the tilt axis displacements from the sample surface Δd and from the diffraction plane Δh give rise to a shift of the centre of gravity of the broadened peak as a function of the tilt angle given by

$$X_0 = 2 \cos\theta\ [\Delta h \mathrm{tg}\alpha - \Delta d(1/\cos\alpha - 1)] \quad .$$

Therefore, it is necessary to include this effect in Equation (2) to take into account the alignment errors on evaluating the intensity loss. Then, both functions to be used in Equation (3) are expressed as

$$I_T(\alpha) = I(\alpha, x_i) + 0.5I(\alpha, x_i')$$

where

$$I(\alpha,x) = (i_m/2WDK^2)\{Kx_1\mathrm{tg}^{-1}(Kx_1/2) - Kx_3\mathrm{tg}^{-1}(Kx_3/2)$$

$$+ 2\ln\sqrt{(4+K^2x_3^2)/(4+K^2x_1^2)} + Kx_2\mathrm{tg}^{-1}(Kx_2/2)$$

$$- Kx_4\mathrm{tg}^{-1}(Kx_4/2) + 2\ln\sqrt{(4+K^2x_4^2)/(4+K^2x_2^2)}\}$$

with

$$x_1 = WD + WR - 2X_0, \qquad x_2 = WD + WR + 2X_0,$$

$$x_3 = WD - WR - 2X_0; \qquad x_4 = WD - WR + 2X_0 \quad \text{and}$$

$$X_0' = X_0 + \Delta x_{1,2} \text{ and}$$

$$I_T(\alpha=0) = (i_m/K)\{2\mathrm{tg}^{-1}(KWR/2) + 0.5[\mathrm{tg}^{-1}((K(WR/2-\Delta x_{1,2}))$$

$$+ \mathrm{tg}^{-1}(K(WR/2+\Delta x_{1,2}))]\} \quad .$$

When a Gaussian fitting is employed,

$$i(x) = i_m\{\exp(-K_g^2x^2) + 0.5\exp[-K_g^2(x-\Delta x_{1,2})^2]\} \quad .$$

Equation (2) has no longer an analytical solution, and numerical integration must be used. For this case the following equation is applicable

$$K_g = \ln 2/x_{\frac{1}{2}} = \ln 2K_c$$

where the subscripts correspond to Gaussian and Cauchy respectively.

EXPERIMENTAL

Several random samples with very fine powders (<10 μm) of copper and molybdenum were made. Filtered CuK_α radiation and a Siemens standard texture goniometer were used. The height and width of the aperture slit were: 1 mm, and the height of the receiving slit: 10 mm. Two different receiving slit widths were used for each case: 2 and 4 mm. The randomness of the samples was verified by the Schulz technique, using the (111) and (220) reflections for the copper and the (110), (211) and (310) reflections for the molybdenum. These materials were chosen because of their quite different absorption coefficients for copper radiation, which might be a significant factor affecting the intensity loss. These reflections were also used for analyzing the defocusing phenomenon. The peak maximum intensity was located with a 0.5 mm receiving slit width and the intensities for each inclination angle, in five-degree steps, were collected during the time necessary for the sample to rotate 360° around the axis normal to its surface. This procedure was chosen to compensate possible intensity losses due to localized density variations, in spite of an increment in the statistical error when diminishing the intensities. In order to obtain an estimation of this error, the intensities of the weaker reflections, (220) for copper and (310) for molybdenum, were collected twice for two different samples in each case. The average intensity spread was approximately 5%.

The values for Δd and Δh were obtained by the method described by Ortiz and Hermida.[6]

Each peak was scanned in a Philips diffractometer, employing horizontal and vertical divergences similar to those used in the Siemens goniometer. The receiving slit width was 0.1 mm, to reduce to a negligible value the broadening produced by this factor. The intensities profiles were compared to Cauchy functions and equalities in the half-widths were chosen to define the values of K_c. In Table I all the parameters used in Equation (3) are shown.

To calculate the double integration of Equation (2), when using Gaussian functions, a computer program in FORTRAN IV was developed, which uses the Simpson's method. The number of integration intervals was chosen by means of a Cauchy function, to make an error smaller than 0.1%, in comparison to Equation (4).

RESULTS AND DISCUSSION

The curves of intensity loss for the given reflections with WR = 2, 4 mm and the experimental points as functions of the tilt angle are shown in Figures 1, 2, 3, 4 and 5. Solid lines correspond to Cauchy fittings and triangles to

TABLE I

Parameters Used in the Calculations for Intensity Losses

Material	hkl	θ (deg.)	K_C (mm^{-1})	$\Delta x_{1,2}$ (mm)	Δd (mm)	Δh (mm)
Copper	111	21.66	4.65	0.45		
	220	37.1	2.68	0.95		
					0.05	0.13
Molybdenum	110	20.27	5.57	0.36		
	211	36.87	4.13	0.94		
	310	50.79	2.75	2		

Values for: one half the diffraction angle, θ; the inverse of one half the half width of the diffraction peak in the Cauchy fitting, K_C; the doublet $K_{\alpha 1}$ - $K_{\alpha 2}$ separation in the receiving plane and the alignment errors Δd and Δh.

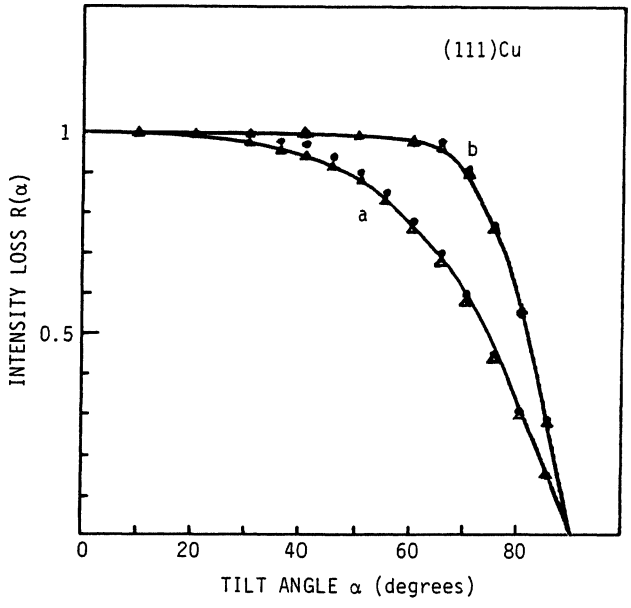

Figure 1. Intensity loss produced for defocusing as a function of the tilt angle for the (111) Cu reflection. a) Receiving slit width of 2 mm. b) Receiving slit width of 4 mm. Solid lines correspond to Cauchy fitting of the diffraction peak and triangles to Gaussian fitting. In both of them alignment errors are included. Dots represent experimental points.

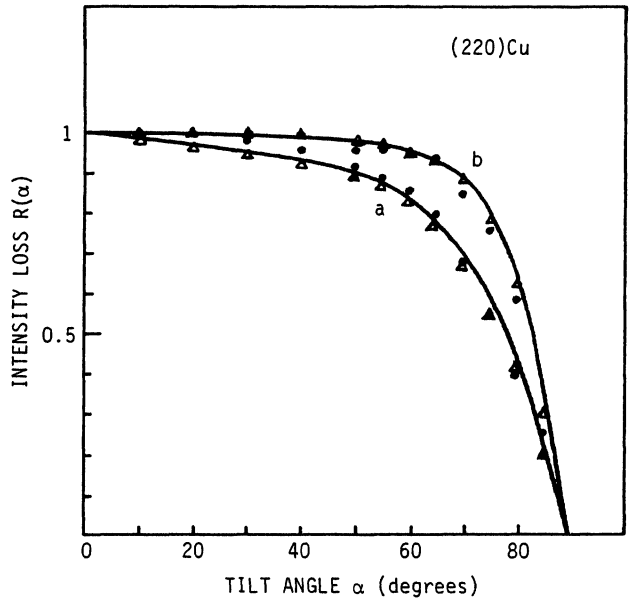

Figure 2. As for Figure 1, but for the (220) Cu reflection.

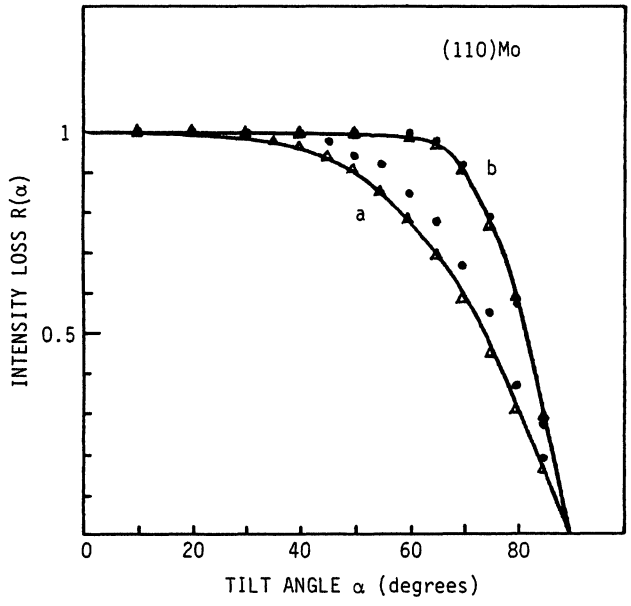

Figure 3. As for Figure 1, but for the (110) Mo reflection.

Gaussian. Differences in the values of R(α) corresponding to both fittings are very small, which means that the intensity loss is quite independent of the peak shape.

The agreement between theoretical curves and experimental points is quite good and is always within a 5% error, except for the (110) for Mo with WR = 2 mm. Possible explanation for this case will be given later.

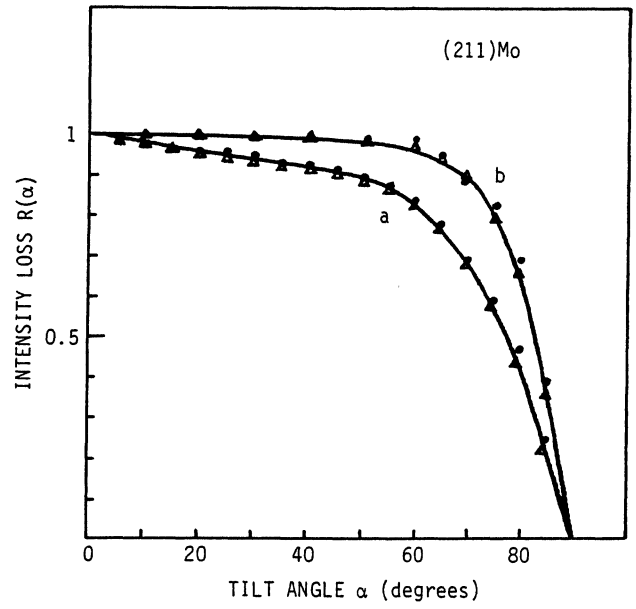

Figure 4. As for Figure 1, but for the (211) Mo reflection.

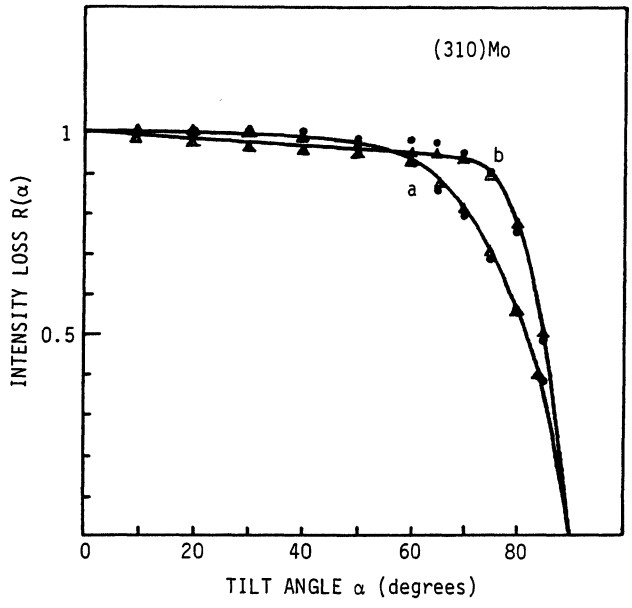

Figure 5. As for Figure 1, but for the (310) Mo reflection.

As expected, the intensity loss is more pronounced for the smaller slit width for each reflection and for both curves it is less pronounced as the diffraction angle increases.

Coincidences between the curves corresponding to the reflections (111) Cu with (110) Mo and (220) Cu with (211) Mo are very significant. The differences in absorption coefficients and in shape of the peaks do not seem to influence

the curves. These coincidences may be interpreted considering the diffraction angle, by means of the geometrical defocusing width WD, as the principal magnitude affecting the curves of intensity loss. This fact may be verified by analyzing, separately, shape and absorption effects. For both pairs of reflection the values of K were interchanged maintaining the same value for the rest of the parameters. No significant variations were detected. Different absorption coefficients produce different depth penetration, which may be represented by variations in the values of Δd. For this reason the influence of Δd on the intensity loss was analyzed. In Figure 6 the curves of intensity loss for the (111) Cu

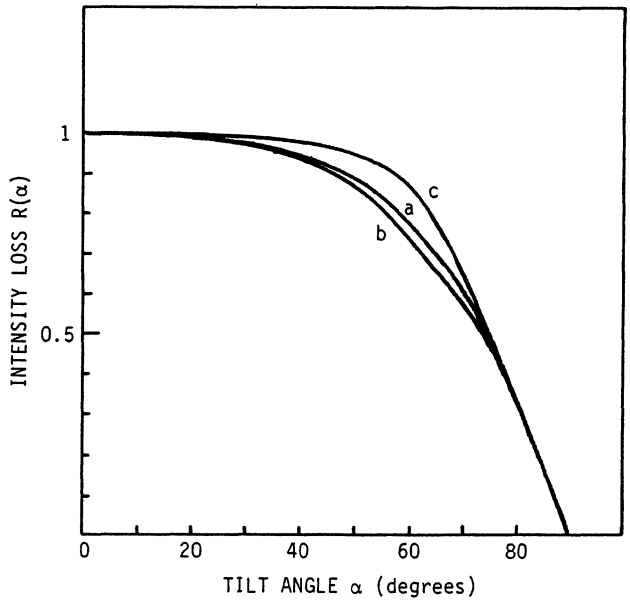

Figure 6. Curve of intensity loss for the (111) Cu reflection with a receiving slit width of 2 mm. a) with both alignment errors Δd and Δh included; b) with $\Delta d = 0$; c) with $\Delta d = \Delta h = 0$.

reflection and WR = 2 mm with both misalignment factors Δd and Δh (curve "a"), with $\Delta d = 0$ (curve "b") and with $\Delta d = \Delta h = 0$ (curve "c") shown. As can be seen, the difference between curves "a" and "b" is small and consequently it seems to be improbable that any variation in the absorption coefficients will significantly modify the curve of intensity loss when the misalignment factors are included. Curve "c" was included to show the influence of both misalignment factors together. If they had not been considered the agreement between theoretical curves and experimental points would have been very poor. In Figure 7 the curves for the (220) Cu reflection and WR = 4 mm, with Δd and Δh included (curve "a") and omitted (curve "b") are shown. The difference between both curves is smaller than before due to the increments in the diffraction angle and in the slit width but, however, is still significant.

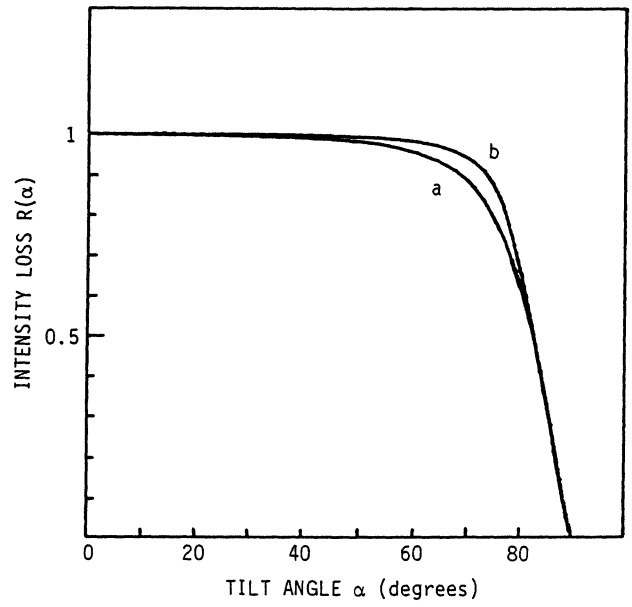

Figure 7. Curve of intensity loss for the (220) Cu reflection with a receiving slit width of 4 mm. a) with Δd and Δh included; b) with $\Delta d = \Delta h = 0$.

In Figure 8 a general sequence of the broadening of the diffraction peak is a function of the tilt angle with misalignment factors included is shown. The plane of the paper corresponds to the receiving plane. As can be seen, the centre

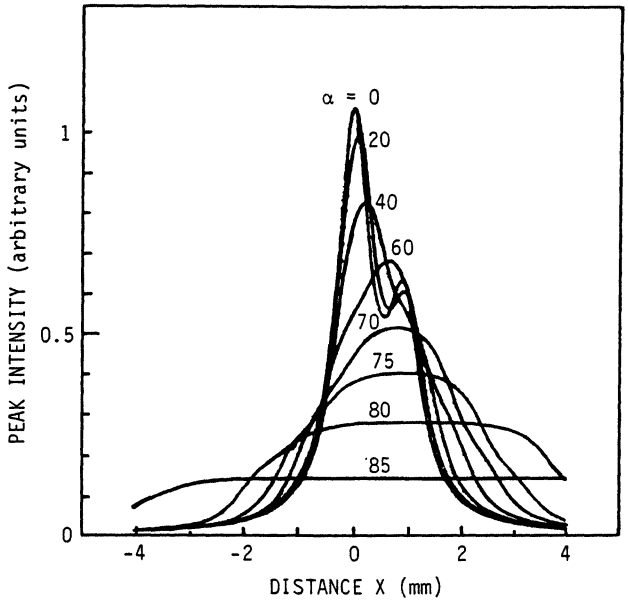

Figure 8. Sequence of the broadening of a diffraction peak for some values of the tilt angle. Alignment errors are included.

of gravity of the peak is shifted towards the right as α increases. At the beginning this shifting is so small that there is no extra intensity loss, but starting from a certain angle, as part of the peak has moved away from the slit width, there is an increment in the intensity loss and the corresponding curve begins to separate from the one in which no alignment errors are included (see, for example, Figure 6). This curve displacement reaches a maximum and then begins to diminish until both curves are superposed again, because the peak has become so flat shaped that the position of the centre of gravity no longer has any influence.

If the centre of the slit is originally shifted towards the right, which means that an error in the peak location has been committed, the subsequent movement of the peak will produce a diminution in the intensity loss, related to the curve in which alignment errors have been included. In Figure 9

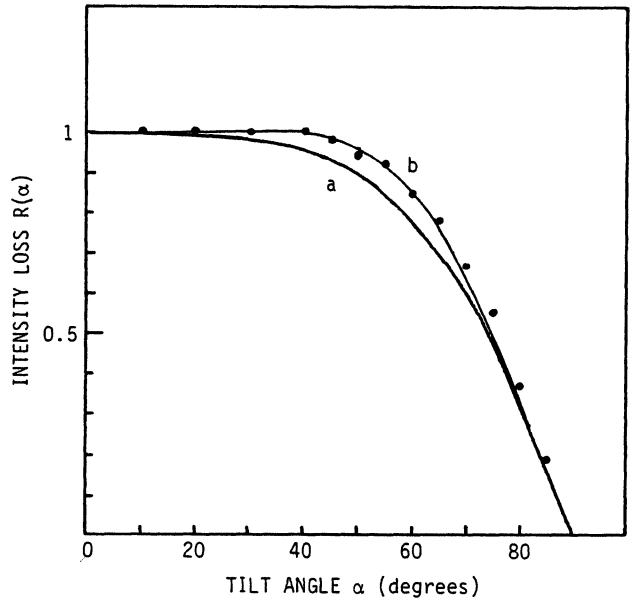

Figure 9. Curve of intensity loss for the (110) Mo reflection with a receiving slit width of 2 mm. a) as for Figure 3a; b) a displacement error in the peak location of 0.3 mm has been included.

two curves corresponding to the reflection (110) Mo with WR = 2 mm and the experimental points are shown. Curve "a" is the same previously shown in Figure 3 and in curve "b" an error of 0.3 mm in the peak location has been included. For this curve the fitting has markedly improved, so, an accidental displacement of the detector might have been responsible for this anomalous distribution of experimental points.

CONCLUSIONS

As the agreement between theoretical curves and experimental points has been so good, and the Gaussian fittings generate curves quite close to Cauchy fittings, it is possible to conclude that a Cauchy fitting of the experimental peak and its subsequent convolution provide a reliable method to correct the intensities for defocusing from $\alpha = 0$ up to $\alpha = 90°$. This is equivalent to saying that, by using this method, complete pole figures can be obtained from a single sample by means of the Schulz technique.

For a given beam height h, the diffraction angle and the slit width are the principal parameters that rule the shape of the curves of intensity loss. Metallurgical state is represented by K, but no precise measurement is required. Intensities coming from reflections of different materials with similar diffraction angle and not very different metallurgical state can be corrected by the same curve.

For every set of experiences a careful test of the alignment conditions is necessary to be carried out and the use of the method described by Ortiz and Hermida is recommended. In particular, it must be pointed out that a small displacement of the tilt axis from the diffraction plane can be the source of large errors.

Finally, it is interesting to note that approximately the first and last 15 degrees define the more reliable zones of the curves of intensity loss when unknown errors are possible to exist.

REFERENCES

1. L. G. Schulz, *J. Appl. Phys.*, 20, 1030 (1949).
2. W. P. Chernock and P. A. Beck, *J. Appl. Phys.*, 23, 344 (1952).
3. E. Tenckhoff, *J. Appl. Phys.*, 41, 3944 (1970).
4. A. Segmüller, *J. Appl. Phys.*, 43, 2907 (1972).
5. V. I. Langford, *J. Appl. Cryst.*, 11, 10 (1978).
6. M. Ortiz and J. D. Hermida, *Texture*, 4, 57 (1980).