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SHORT COMMUNICATION

A METHOD FOR TESTING THE TEXTURE GONIOMETER ALIGNMENT OF THE SCHULZ REFLECTION TECHNIQUE

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INTRODUCTION

In this widely used technique there is an intensity loss due to the broadening of the diffraction peak as a consequence of the defocusing of the irradiated area when the specimen is tilted. An inadequate alignment of the goniometer produces an additional intensity drop which can significantly affect the results. There are two factors responsible for this phenomenon: a) the tilt axis is not in the surface plane of the specimen; b) the tilt axis is not in the diffraction plane. The first factor was referred to by Chernock and Beck[1] and by Feng.[2] Both factors produce a peak shift towards lower or higher diffraction angles as the sample is tilted. If a random sample is used, when correcting the intensities produced by the textured sample as a result of defocusing, both factors are included in the random intensities. But, if a theoretical expression is used (Tenckhoff,[3] Feng,[2] Segmuller[4]), these misalignment factors should be detected and taken into account.

THEORY

In Figure 1 the top view of the experimental conditions is shown schematically. The beam diverges from S and focuses at F_1. For a displacement of the sample d, normal to the tilt axis and parallel to the diffraction plane, the peak shift W in the receiving plane is given by

$$W = 2d \cos \theta$$

(In fact, there is also a little broadening due to the position of the new foci F_2 and F_2', which do not lie in the receiving plane, but this effect is negligible compared to W.)

In Figure 2 a side view is shown. The sample has been tilted at an angle α around the y axis, which lies in the diffraction plane.

The beam has a height h, so the displacement d is a function of z and α. The maximum geometrical defocusing broadening for a tilt angle α is

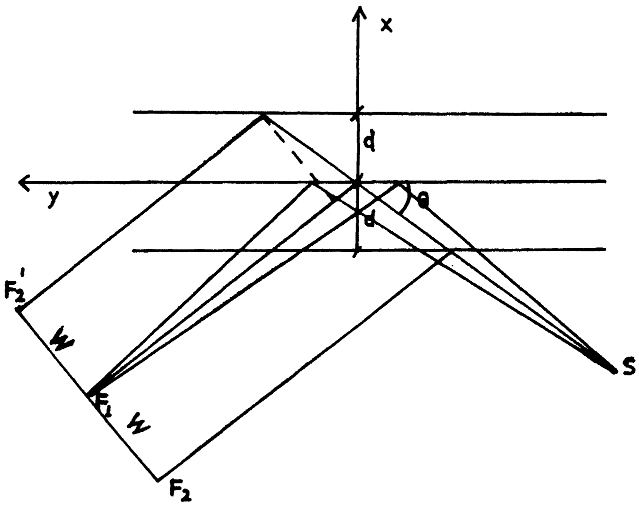

Figure 1. Schematic top view of the experimental conditions in the Schulz technique.

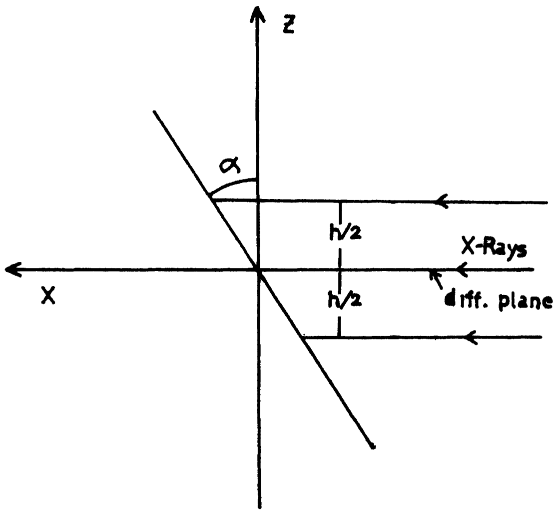

Figure 2. Schematic side view of the experimental conditions in the Schulz technique.

$$W_D = 2htg\alpha \cos\theta$$

which is the expression derived by Tenckhoff.[3] In the receiving plane there are contributions by the diffracted beam up to a distance $htg\alpha \cos\theta$ on each side of the centre, F_1.

However, if both misalignment factors are taken into account, the maximum defocusing broadening remains unchanged, but the peak is no longer centered at F_1. Figure 3 shows the same view as Figure 2 with the misalignment factors included. The sample is displaced a distance d from the tilt axis in a direction parallel to the diffraction plane which is also

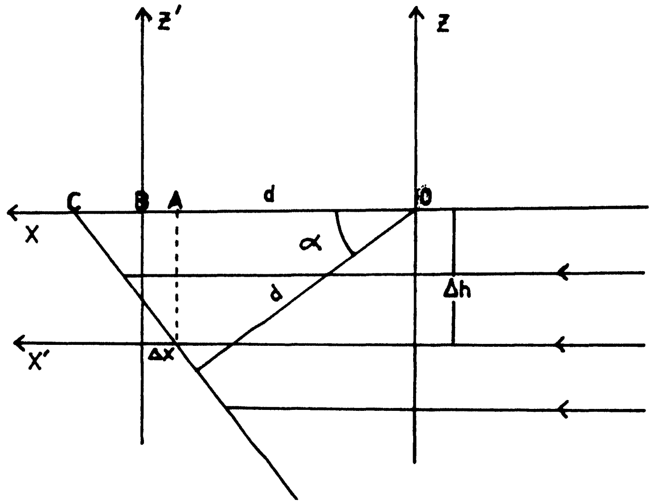

Figure 3. Same view as Figure 2 with the misalignment factors included.

displaced downward by Δh from its original position. Accordingly, for $\alpha = 0$ the peak is shifted to $2d \cos \theta$ towards lower angles. If the detector is placed in this new position, when the sample is tilted counterclockwise, the center of the peak will now be shifted to higher angles by the amount $2\Delta x \cos \theta$. This displacement Δx can be related to d, Δh and α by means of the following equations:

$$OC = d/\cos \alpha \qquad BC = OC - d = d(1 - \cos \alpha)/\cos \alpha$$

$$AC = \Delta htg \qquad \Delta x = AB = AC - BC$$

and consequently

$$\Delta x = \Delta htg\alpha - d(1 - \cos \alpha)/\cos \alpha. \tag{1}$$

This expression, Equation (1), has a maximum positive value for $\alpha m = \sin^{-1}(\Delta h/d)$, and the value becomes smaller for higher angles until a certain angle, given by the relation

$$d = \Delta hctg(\alpha_0/2) \tag{2}$$

is reached, when $\Delta x = 0$. For higher angles Δx becomes negative, which means that the peak shifts to lower angles, because Δx is situated to the left of the z' axis.

Then, for $\alpha = \alpha_0$, which is the angle corresponding to $\Delta x = 0$, the peak is again centered at F_1 and, if W_D is still smaller than the receiving slit, the same intensity as for $\alpha = 0$ should be received by the detector, because no defocusing has been yet detected.

The counterclockwise tilt is defined as negative. If Δh is positive, Equation (1) transforms into

$$\Delta x = -[\Delta htg\alpha + d(1 = \cos \alpha)/\cos \alpha]$$

so the sample must be tilted clockwise to repeat the aforementioned procedure. Otherwise the peak would continuously shift towards lower angles.

Equation (2) gives the value of α_0 for certain values of d and Δh. The distance d can be expressed by

$$d = d_e + \Delta d,$$

that is, a known displacement from the zero obtained in the alignment plus an unknown displacement from the true zero. Then, Equation (2) can be rewritten in a general way as

$$d_e = \Delta d + \Delta h \mathrm{ctg}(\alpha_0/2) \qquad (3)$$

and if a plot of d_e vs. $\mathrm{ctg}(\alpha_0/2)$ is made it will be possible to obtain Δd and Δh, the two misalignment factors.

EXPERIMENTAL

A random sample of copper was prepared with a very fine power ($<10\mu$) mixed with glue and gently pressed into a circular cavity. The randomness of the sample was tested in a Siemens goniometer by means of the Schulz technique. The (111) reflection was analyzed with filtered copper radiation. The receiving slit width was 2 mm. Variations in intensity were no larger than 5% and for up to a 30 degree change in the tilt angle there was no significant intensity drop.

The sample was then placed at a distance d_e = 1 mm from the zero alignment, according to Figure 3. The detector was moved to the new peak position which was found with a 0.5 mm receiving slit width, and the sample was tilted clockwise in 2 degree steps. For each position a fixed counting of 6 minutes was employed which was the time necessary for the sample to rotate 360 degrees around the axis normal to its surface. The intensity continuously decreased from the beginning. When a counterclockwise tilt was made the intensity first decreased, but after 10 degrees it began to increase until at 17 degrees the same intensity as for $\alpha = 0$ was detected. The same procedure was repeated with d_e = 0.6, 0.8 and 1.5 mm. These values were chosen so as to give α_0 angles below 30 degrees, to ensure that there were no defocusing contributions. To increase the range of d_e, negative values were also used, that is to say that the sample was placed at different positions to the right of the z axis (Figure 3). Figure 4 shows the results.

A linear regression curve gave the following values:

$$\Delta d = -0.05 \text{ mm} \quad ; \quad \Delta h = -0.13 \text{ mm}$$

and a correlation factor of $\rho = 0.96$.

DISCUSSION

The experimental spread is low enough to assume a good agreement between theory and experiment. The value of d is quite close to the penetration depth for this powder sample

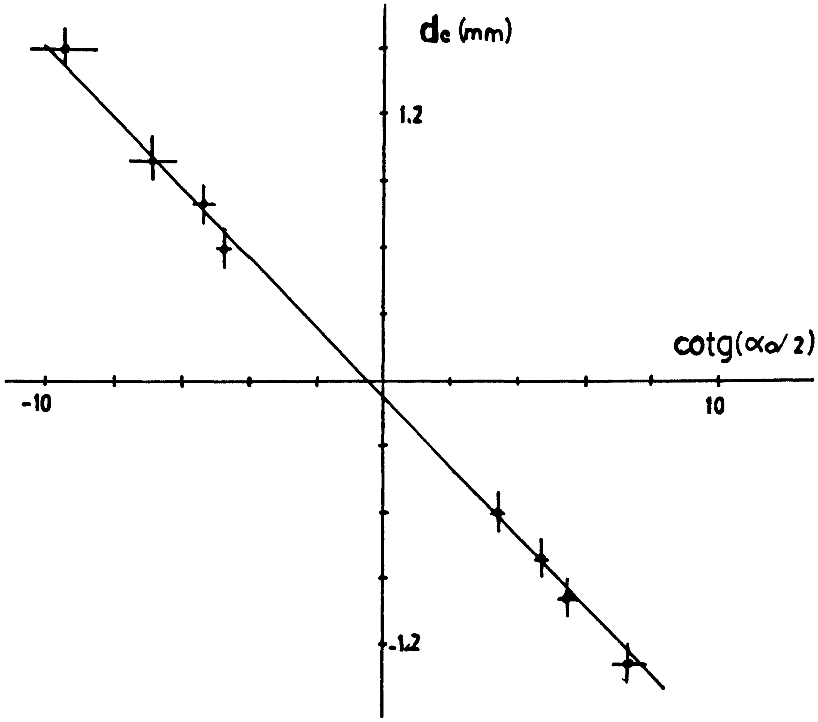

Figure 4. Experimental points and linear regression curve of the d_e vs. ctg(α_0/2) plot.

and will not significantly affect the defocusing intensity drop. This is not the case for Δh, which will play a dominant role, as can be derived from Equation (1), continuously shifting the peak towards higher angles as the sample is tilted counterclockwise, or vice versa.

The positioning of a flat surface sample will rarely introduce an error much larger than 0.05 mm but an error of 0.1 mm in the position of the diffraction plane is not surprising, because of the visual method employed in aligning the height of the beam.

CONCLUSION

A method for detecting misalignment factors in the Schulz reflection technique has been derived. Its principal importance is related to the use of a theoretical expression to correct the intensities for defocusing. However, even in the case when a random sample is used, it is convenient to check that these factors, especially the displacement of the diffraction plane, are not large enough to produce an excessive intensity drop.

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