Textures and Microstructures, 1982, Vol. 4, pp. 201–210
0730–3300/82/0404–0201 $06.50/0

Printed in Great Britain

Defocusing in the Reflexion Technique for the Determination of Preferred Orientation Using EDXRD

J. KIVILÄ and E. LAINE

Department of Physical Sciences, University of Turku, 20500 Turku 50, Finland

(*Received December 15, 1980; in final form March 2, 1981*)

The defocusing effect in the reflexion technique for the determination of the preferred orientation using energy-dispersive x-ray diffraction (EDXRD) method is studied experimentally. The measurements show that the defocusing effect is dependent on the Bragg angle, the receiving slit width, the reflexion hkl and the metallurgical condition of the specimen. The defocusing correction or at least the optimizing of the measuring geometry is found to be necessary in quantitative texture measurements.

INTRODUCTION

The use of the energy-dispersive X-ray diffraction (EDXRD) method for determination of preferred orientations and its adaptability to different texture studies have been demonstrated in several papers (Laine and Lahteemaki, 1971; Szpunar *et al.*, 1974; de Ben and Broyde, 1973; Gerward *et al.*, 1976; Heller *et al.*, 1977; Laine *et al.*, 1977; Szpunar and Gerward, 1978, 1980; Kivila *et al.*, 1980). Many of these deal with the determination of inverse pole figures of the specimen by the Harris (1952) technique. Laine *et al.* (1977), Szpunar and Gerward (1978, 1980) and Kivila *et al.* (1980) have applied EDXRD to texture measurements using the Schulz reflexion technique (Schulz, 1949). Because in this reflexion technique the specimen is tilted about the axis located in the diffraction plane perpendicular to the goniometer axis, the correction has in most cases to be made for the occurrence of defocusing. Therefore, it is important to consider the defocusing effect in the case of EDXRD. The defocusing correction in the conventional angle-dispersive diffraction (ADXRD) has been studied by Gale and Griffiths (1960), Tenckhoff (1970), Huijser-Gerits and Rieck (1974) and Holt and Winegar (1977).

The purpose of the present work is to study experimentally the different factors which can effect defocusing in the energy-dispersive case. Such factors are the Bragg angle, the receiving slit width, the reflexion hkl and the metallurgical condition of the specimen.

THEORY

According to Tenckhoff (1970) the defocusing correction factor as a function of the polar angle α can be expressed by

$$C^{hkl}(\alpha) = \frac{I^{hkl}_{ran}(\alpha)}{I^{hkl}_{ran}(\alpha = 0)}, \tag{1}$$

where $I^{hkl}_{ran}(\alpha)$ is the measured intensity of the reflexion *hkl* from a specimen with no texture as a function of the polar angle α and $I^{hkl}_{ran}(\alpha = 0)$ is the corresponding intensity at $\alpha = 0$.

For the observed standard deviation of the diffracted beam profile in the defocused condition Holt and Winegar (1977) have represented the expansion

$$\sigma_o = (\sigma_s^2 + \sigma_f^2 + \sigma_{df}^2)^{\frac{1}{2}}, \tag{2}$$

where σ_s is the standard deviation of the profile representing broadening due to small grain size and lattice strain, σ_f is the standard deviation of the profile representing instrumental broadening in the focused condition ($\alpha = 0$) and σ_{df} is the standard deviation of the profile representing the broadening due to defocusing. For a given peak shape, the diffracted intensity observed at the receiving slit depends only on the ratio of the width of the receiving slit, W, to σ_o. When the defocusing increases also σ_o increases and W/σ_o decreases. Thus the diffracted intensity passing through the receiving slit decreases. Because the effect of σ_{df} on σ_o is greater for the annealed specimen (sharp diffraction line profile, $\sigma_s \approx 0$) than for the cold-worked specimen (broad diffraction line profile, $\sigma_s > 0$), so the defocusing corrections depend also on the metallurgical condition of the specimen.

For a constant shape of the profile, the standard deviation is

$$\sigma = A\beta, \tag{3}$$

where A is constant and β is the width of the profile measured at half-maximum-intensity. In terms of Eqs. (2) and (3) the expressions for the full width at half-maximum-intensity measured for the defocused and focused profiles from the cold-worked and annealed specimens are obtained:

$$\beta_{df}(\text{cold-worked}) = \frac{(\sigma_s^2 + \sigma_f^2(\text{cold-worked}) + \sigma_{df}^2)^{\frac{1}{2}}}{A} \tag{4}$$

$$\beta_f(\text{cold-worked}) = \frac{(\sigma_s^2 + \sigma_f^2(\text{cold-worked}))^{\frac{1}{2}}}{A} \tag{5}$$

$$\beta_{df}(\text{annealed}) = \frac{(\sigma_f^2(\text{annealed}) + \sigma_{df}^2)^{\frac{1}{2}}}{A} \tag{6}$$

$$\beta_f(\text{annealed}) = \frac{\sigma_f(\text{annealed})}{A}, \tag{7}$$

where the indices df and f refer to the defocused and focused condition of the specimen, respectively.

By dividing (6) by (7) and solving σ_{df}^2, we get

$$\sigma_{df}^2 = \left[\frac{\beta_{df}^2(\text{annealed})}{\beta_f^2(\text{annealed})} - 1\right] A^2\beta_f^2(\text{annealed}). \tag{8}$$

Then by dividing (4) by (5) and using (8), we get

$$\frac{\beta_{df}^2(\text{cold-worked})}{\beta_f^2(\text{cold-worked})} = 1 + \frac{\beta_{df}^2(\text{annealed}) - \beta_f^2(\text{annealed})}{\beta_f^2(\text{cold-worked})}. \tag{9}$$

For a constant shape of the profile the diffracted intensity observed by the receiving slit is proportional to W/σ. On the other hand $\sigma = A\beta$, so Eq. (1) can be written in the form

$$C^{hkl}(\alpha) = \frac{\beta_{ran}^{hkl}(\alpha = 0)}{\beta_{ran}^{hkl}(\alpha)}, \tag{10}$$

thus the change in the defocusing correction factor as a function of the tilting angle can be considered also as the ratio of the widths of the focused and defocused beam profiles.

The energy-dispersive x-ray diffraction is based on the Bragg law

$$E = \frac{hc}{2d\ \sin\theta}, \tag{11}$$

where 2θ is the fixed diffraction angle, h is Planck's constant and c is the velocity of light. Each lattice plane spacing d will select a corresponding energy E from the incident polychromatic x-ray beam. Differentiating with respect to 2θ gives the relation between the line breadths measured on the 2θ scale used in angle-dispersive diffraction and those measured on the energy scale used in energy-dispersive diffraction:

$$dE = \frac{hc}{2d}\frac{-\cos\theta}{\sin^2\theta}\mathrm{d}\theta = -\tfrac{1}{2}E\ \cot\theta\ d(2\theta). \tag{12}$$

Thus the diffraction peaks can easily be transformed into the energy scale by using Eqs. (11) and (12) and Eqs. (9) and (10) may be used also in the case of the energy-dispersive diffraction.

EXPERIMENTAL

To study the defocusing effect powder specimens with random texture must be prepared. As specimen materials we used aluminium and β-brass powders. The powders were pressed into disks using as low moulding pressures as possible. Specimen 1 was prepared from the aluminium powder using the pressure of 33.5 MPa. Specimen 2 was prepared from the cold-worked β-brass powder and specimen 3 from the annealed (1 hr. at 500°C) β-brass powder. In the preparation of both brass specimens a pressure of 46.8 MPa was applied. Thus the porosity and the surface roughness of the specimens were reduced and, furthermore, no significant effects of preferred orientation induced by preparation were discernible in the defocusing curves. The ratio of the full width at half-maximum of diffraction lines of the brass specimens prepared by the different methods was measured by the angle-dispersive method. For example the 111 lines of the cold-worked specimen were about two times as broad as the 111 lines of the annealed specimen.

The measurements were carried out by the energy-dispersive method and a Siemens texture goniometer. The measuring equipment has been described

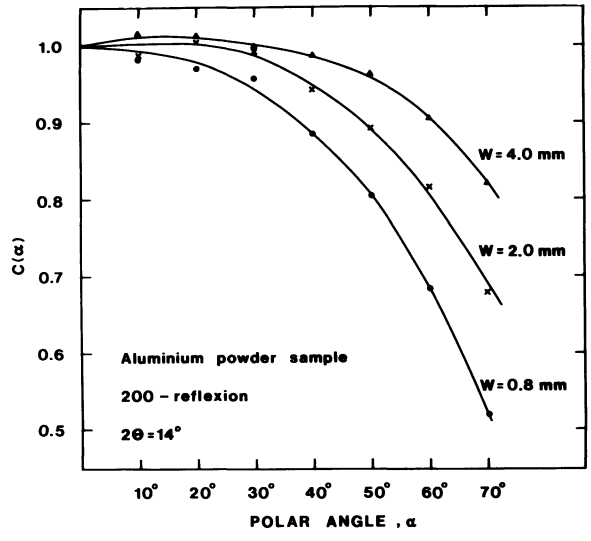

FIGURE 1 Effect of the width of the receiving slit on the defocusing correction factor.

previously elsewhere (Laine *et al.*, 1977). A flatfaced specimen was placed in the sample holder of the texture goniometer and the specimen was radiated by polychromatic radiation from a Cu-tube. The diffraction peaks were obtained in the energy range between 10 and 30 keV. The specimen was rotated in its own plane and it was inclined in regard to the vertical plane by up to $\pm$ 70°. In the measurements three different receiving slit widths (0.8, 2.0 and 4.0 mm corresponding 0.2°, 0.5° and 1.0°, respectively) and two different Bragg angles (14° and 28°) were used. The diffraction spectra

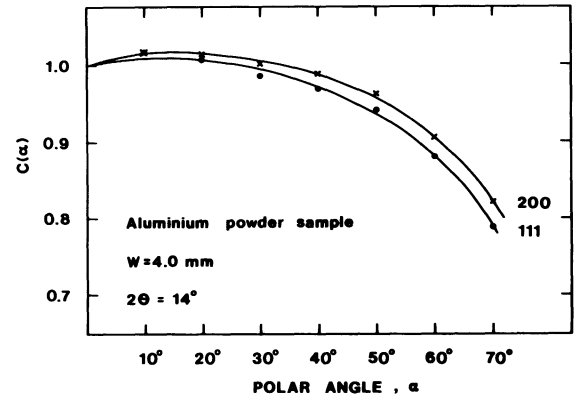

FIGURE 2 Effect of different *hkl* reflexions on the defocusing correction factor.

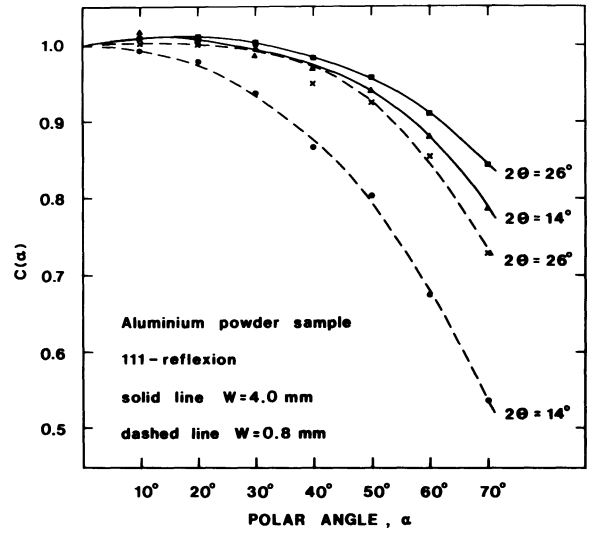

FIGURE 3 Effect of the Bragg angle on the defocusing correction factor.

obtained were recorded on punched tape and analysed by a computer program, which calculated the integrated intensities and the full widths at half-maximum of the 111 and 200 reflexions.

RESULTS AND DISCUSSION

The effect of the width of the receiving slit on the defocusing correction was studied using the aluminium specimen. The defocusing correction factors $C(\alpha)$ of the 200 reflexion of aluminium as a function of the polar angle α, when the 2θ angle is 14°, are presented in Figure 1. The measurements were carried out using three different receiving slit widths. It was found that the sharper the slit the greater is the defocusing correction. The difference between the correction curves is greater at the small Bragg angle than at the large one (see Figure 3).

Also the defocusing curves measured for different *hkl* reflexions differ from each other. In Figure 2 the defocusing correction factors $C(\alpha)$ of the 111 and 200 reflexions of aluminium are presented as a function of the polar angle when 2θ is 14°. The curve of the 111 reflexion runs lower than the one of the 200 reflexion, so the loss of intensity is greater for the reflexions of low index.

The effect of the Bragg angle on the correction curves can be seen in

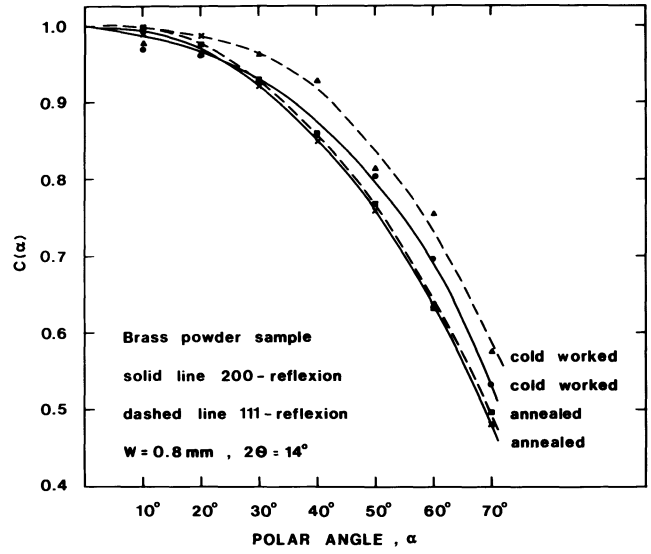

FIGURE 4 Effect of the metallurgical condition of the specimen on the defocusing correction factor.

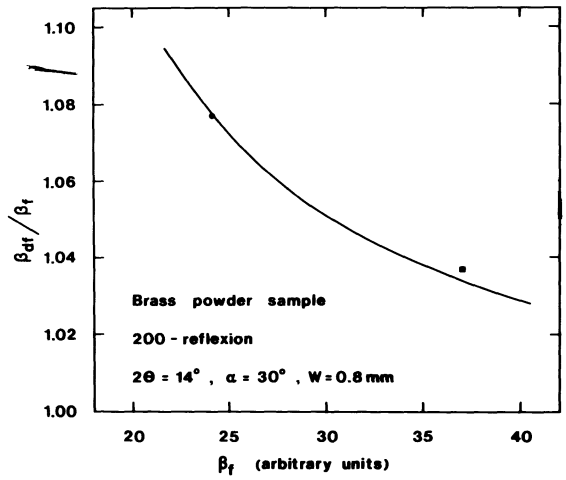

FIGURE 5 Change of the ratio of the full widths at half-maximum-intensity due to defocusing measured from the annealed (●) and cold-worked (■) β-brass specimen. The solid line is calculated from Eq. (9).

Figure 3. There are presented the defocusing correction curves measured at two different Bragg angles using two widths of the receiving slit. The loss of the intensity at the greater Bragg angle is significantly less than at the smaller one. The difference between the curves comes out more clearly when the narrow receiving slit is used. Furthermore, when the Bragg angle increases the arching of the curves begins at slightly larger values of the polar angle.

The metallurgical condition of the specimen has considerable affects on the magnitude of the defocusing correction, as one would expect on the basis of Eq. (2). In Figure 4 the defocusing correction curves are seen for the 111 and 200 reflexions of the cold-worked and annealed β-brass specimens. It was found, that the loss of the intensity from the annealed specimen, whose diffraction peaks were sharp, is greater than for the cold-worked specimen whose diffraction peaks are markedly broader. The difference is greatest when the receiving slit was as narrow as possible and the Bragg angle was small. Furthermore, the effect of the metallurgical condition of the specimen on the 200 reflexion or in general on the high index reflexions was stronger than on the low index reflexions (e.g. the 111 reflexion).

The change of the ratio of the full widths at half-maximum in tilting the specimen is presented in Figure 5 as a function of the line breadth of the 200 reflexion at the polar angle $\alpha = 0$. The solid curve has been calculated from Eq. (9). Two experimental observation points correspond to the cold-worked and the annealed β-brass specimen when $2\theta = 14°$, $\alpha = 30°$ and $W =$ 0.8 mm. The measured points agree well with the calculated curve, so the assumption that the shape of the profile is constant in defocusing also in

TABLE I
Effect of the width of the receiving slit and the metallurgical condition on the defocusing correction factor C^{111} (60°) measured for the 111 reflexion of β-brass specimen at two different Bragg angles.

		$C^{111}(60°)$	
2θ	Specimen	Slit 0.2°	1.0°
14°	Annealed	0.64	0.86
14°	Cold-worked	0.70	0.88
26°	Annealed	0.85	0.92
26°	Cold-worked	0.89	0.93

the case of the EDXRD does not lead to a disagreement between the theory and the experiments.

From Table I it is seen the effect of the width of the receiving slit and the metallurgical condition of the specimen on the defocusing correction factor for the 111 reflexion of β-brass, when the polar angle of the specimen is 60°.

It was found that when the receiving slit was made wider the difference of the defocusing correction factors due to the metallurgical condition of the specimen decreased. Furthermore, the effect on the broad peak profile was smaller than on the sharp peak shape.

The defocusing correction curve at small polar angles tended to rise over a value of 1.0 when the width of the receiving slit was wide. Thus in the normal diffraction position the whole intensity falls on the detector. When the specimen was tilted the peak broadened and the increase of the diffracting volume raised the intensity. When the receiving slit was wide enough, it did not, however, at small tilting angles reduce the broadened intensity distribution but greater intensity was obtained than at the tilting angle $\alpha = 0$. The effect appeared at its strongest when the diffraction peak was narrow, as in the aluminium powder specimen. Cold worked β-brass behaved differently. Its peaks, although initially wide enough, decreased when the tilting angle increased.

We calculated also the defocusing correction functions according to an equation proposed by Gale and Griffiths (1960). The results indicated that the equation may be adapted in the angle range $14° \leqslant 2\theta \leqslant 26°$ only if the width of the receiving slit was $W \geqslant 3$ mm (0.8°). When the width of the slit was smaller the calculated correction curve decreases more rapidly than the experimental one. The use of Gales's calibration function in the case of EDXRD calls for more detailed examination. It would appear to be best to determine the defocusing correction curve with the aid of specimens with a random texture, if possible. Also the experimentally determined functions best correspond to the measuring conditions.

CONCLUSIONS

When texture studies are performed by the ADXRD or EDXRD method using the Schulz technique the defocusing causes an error by diminishing the integrated intensities. So it is important to perform the defocusing correction of the integrated intensities. The loss of intensity can be decreased markedly by choosing as favourable a measuring geometry as possible for the measurements. In the EDXRD method the Bragg angle is fixed and therefore the texture goniometer is easier to adjust exactly. In practice the optimum angle, 2θ, is found to be in the range of 10°–40° depending on the anode material of the X-ray tube and the specimen studied. Because the loss of the intensity at a large angle is smaller than at a small one, the angle, 2θ, should be selected as close to the final end of the angle range as the fluorescence peaks allow. The most suitable width of the receiving slit is 0.5°–1.0°. When the slit width is decreased the resolution will improve but on the other hand it increases the defocusing correction so that the slit width below 0.5° is not worth using. When using the slit width over 1.0° it will become difficult to separate the reflexions from each other especially at large values of the polar angle. Also the loss in intensity for different reflexions is dissimilar. For the high index reflexions greater defocusing corrections must be made. The magnitude of the defocusing correction also depends greatly on the width of the diffracted beam profile or, that is to say, on the metallurgical condition of the specimen. The loss of intensity is relatively greater for a sharp than for a broad profile. In general from the texture specimens fairly broad diffracted linewidths are obtained compared with the linewidths of the annealed powder specimen. When the defocusing correction is determined experimentally the full width at half-maximum of the reflexion obtained from the textureless specimen should also correspond to that of the textured specimen. Thus by optimizing the values of the different parameters in each case the measurements of the intensities can be carried out reliably in tilting the specimen up to $\pm 60°$. After that it is still necessary to perform the defocusing correction. In determining the direct pole figure the inner parts of the figure must be measured using the reflexion technique and the outer parts using the transmission technique. So performing the defocusing correction improves the accuracy of the pole figure data, which is essential in deriving the properties of textured materials. The EDXRD method is suitable for the determination of the whole direct pole figures (Szpunar and Gerward, 1980) and for the study of the texture with the aid of polar axis density distributions (Laine *et al.*, 1977; Kivila *et al.*, 1980). The advantages of the EDXRD method compared with the conventional ADXRD method have been discussed previously elsewhere (Szpunar *et al.*, 1974; Laine *et al.*, 1977).

However, in spite of the many advantages we think that the EDXRD method will, in the future, be complementary to, rather than a substitute for ADXRD methods in the texture measurements.

Acknowledgement

We want to express our thanks to A. Elomaa for his assistance during the experiments.

References

De Ben, H. S. and Broyde, B. *Appl. Spectrosc.* **27**, 99 (1973).
Gale, B. and Griffiths, D. *Brit. J. Appl. Phys.* **11**, 96 (1960).
Gerward, L., Lehn, S. and Christiansen, G. *Texture* **2**, 95 (1976).
Harris, G. B. *Phil. Mag.* **43**, 113 (1952).
Heller, H., Slakhorst, J. and Verbraak, T. *Z. Metallkde.* **68**, 31 (1977).
Holt, R. A. and Winegar, J. E. *J. Appl. Phys.* **48**, 3557 (1977).
Huijser-Gerits, E. M. C. and Rieck, G. D. *J. Appl. Cryst.* **7**, 286 (1974).
Kivila, J., Laine, E. and Parviainen, S. *Texture* **4**, 63 (1980).
Laine, E. and Lahteenmaki, I. *J. Mater. Sci.* **6**, 1418 (1971).
Laine, E., Kivila, J. and Lahteenmaki, I. *Texture* **2**, 243 (1977).
Schulz, L. G. *J. Appl. Phys.* **20**, 1030 (1949).
Szpunar, J., Ojanen, M. and Laine, E. *Z. Metallkde.* **65**, 221 (1974).
Szpunar, J. and Gerward, L. Texture of Materials, *Proc. of the Fifth Int. Conf.* on Textures of Materials, Aachen, Germany, Vol. I, Springer-Verlag, Berlin (1978).
Szpunar, J. and Gerward, L. *J. Mater. Sci.* **15**, 469 (1980).
Tenckhoff, E. *J. Appl. Phys.* **41**, 3944 (1970).