Simultaneous Measurement of Several X-ray Pole Figures

BY J. J. HEIZMANN AND C. LARUELLE

Laboratoire de Métallurgie Structurale, Metz University, Metz, France

(Received 6 November 1985; accepted 21 July 1986)

Abstract

At the moment, routine texture analysis is limited by the time required for data recording. A method allowing simultaneous acquisition of several pole figures with the use of several detectors or a position-sensitive detector is described. Because of the simultaneity, the movements of the texture goniometer used for one pole figure are also used at the same time to obtain the other pole figures. The complete geometric arrangement and the scanning of pole figures are explained for reflection and transmission measurements. The blind area, the absorption intensity correction and the defocusing phenomenon are discussed with respect to the sample. For some examples, the results obtained by this method are compared with those obtained by the usual method employed for the measurement of a single pole figure.

Introduction

Nowadays, most crystallographic texture measurements are performed using X-rays with a texture goniometer according to the method of Schultz (1949a, b). Microcomputer-controlled texture goniometers have recently appeared. The computer is meant not only to set the sample according to the X-ray incident beam, but also to analyse the X-ray beam diffracted by the \( (hkl) \) lattice planes. This leads to the \( (hkl) \) pole figure. The time required to obtain the figure is usually about an hour or more. To find the orientation distribution function of the crystallites, several \( (hkl) \) pole figures are needed (Bunge, 1969). Thus the time and cost of the texture measurement are widely increased. At present, the development of routine texture analysis is limited by the time required to record data. In this paper we are proposing a method which allows us to obtain simultaneously several \( (hkl) \) pole figures. This possibility had been anticipated, using neutron diffraction and a 2\( \theta \) position-sensitive detector, by Bunge, Wenk & Pan-netier (1982).

A. Method for the simultaneous recording of several pole figures

Because of the simultaneity, the different pole figures are obtained by the same goniometer movement and in the same time as required to obtain a single pole figure.

Let us consider a reference frame \( Oxyz \) bound to the sample, \( Oz \) being perpendicular to the sample. The normal \( [hkl]* \) to the \( (hkl) \) lattice planes of the sample is located by angles \( \psi \) and \( \varphi \) (Fig. 1). The incidence plane \( II \) is defined by the incident X-ray beam and several detectors, for example, the two detectors \( C_1 \) and \( C_2 \) in Fig. 2.

The sample is positioned with respect to detector \( C_1 \) in the Bragg-Brentano arrangement (incidence angle = reflection angle = \( \theta(hkl) \)). It follows that the second detector \( C_2 \) is not in the Bragg-Brentano arrangement.

On the goniometer the sample rotates about two perpendicular rotation axes: rotation axis \( \phi \) normal to

Fig. 1. A normal \( [hkl]* \) is located by two angles \((\psi, \varphi)\) with respect to the sample.

Fig. 2. Detector \( C_1 \), lying in the incidence plane \( II \), detects lattice planes whose normals \( [hkl]* \) are on a cone of aperture \( \psi \). Detector \( C_2 \), lying in the incidence plane \( II \), detects lattice planes whose normals \( [hkl]* \) are on a cone of aperture \( \psi_2 \).
the sample (azimuth angle $\phi$) and rotation axis $\psi$ (tilting angle) lying in the sample.

For a given tilting angle $\psi$ when the sample rotates about $\phi$ (Fig. 2): (a) the detector $C_1$ will record the $(h_1k_1l_1)$ lattice planes whose normals $[h_1k_1l_1]^*$ are on a cone of aperture $\psi$ which is tangent to the incidence plane $\Pi$. The whole stereographic set is scanned if the sample rotates by $2\pi$ about $\phi$ for each value of $0 < \psi < \pi/2$. (b) The detector $C_2$ will detect the lattice planes $(h_2k_2l_2)$ whose normals $[h_2k_2l_2]^*$ lie on a cone of aperture $\psi_2$.

By application of spherical trigonometry we can obtain this angle $\psi_2$:

$$\cos \psi_2 = \cos \psi \cos(\theta_2 - \theta_1).$$

(1)

The angular coordinate $\varphi_2$ of the normal $[h_2k_2l_2]^*$ is

$$\varphi_2 = \varphi + \Delta\varphi$$

(Fig. 3), with:

$$\sin \Delta\varphi = \sin(\theta_2 - \theta_1)/\sin \psi_2.$$  

(2)

So each measurement made at the $(\psi, \varphi)$ position of the goniometer must be transferred to the angular coordinates $(\psi_2, \varphi_2)$ of the pole figure. $(\psi_2, \varphi_2)$ are the angular coordinates of the normal $[h_2k_2l_2]^*$ with respect to the sample.

**Blind area**

When the goniometer's tilting angle $\psi = 0$, the normal $Oz$ of the sample lies in the incidence plane $\Pi$ and is the bisectrix of the $\pi - 2\theta_{(h_1k_1l_1)}$ angle. The normal $[h_2k_2l_2]^*$ of the $(h_2k_2l_2)$ lattice planes, detected by detector $C_2$, also lies in the incidence plane $\Pi$.

The angle between $[h_2k_2l_2]^*$ and $\phi$ is $\psi_2 = \theta_2 - \theta_1$; this implies that all the $(h_2k_2l_2)$ lattice planes whose normals are inside the cone of aperture $\psi_2$ cannot be detected by detector $C_2$. This leads to a blind area on the pole figure (Fig. 4).

**Stereographic projection in the Oxy plane of the sample**

In Fig. 4, the straight line $\Pi_0$ represents the incidence plane when $\psi = 0$. When the sample rotates only about $\psi$ the incidence plane becomes a great circle $\Pi_\psi$ whose position varies from $x'Ox$ to $x'yx$ when $\psi$ changes from 0 to $\pi/2$. Let us consider a value $\psi$ of the tilting angle. Poles $P_1$ and $P_2$ in the incidence plane are respectively the positions of the $[h_1k_1l_1]^*$ and $[h_2k_2l_2]^*$ normals of the $(h_1k_1l_1)$ and $(h_2k_2l_2)$ lattice planes seen by the detectors $C_1$ and $C_2$. When $\psi$ changes from 0 to $\pi/2$, $P_1$ moves on the straight line $O_1y$, and $P_2$ from $P'_2$ to $\psi$ on the great circle $P_2y$.

For a given value of $\psi$, a pole located on the circle of radius $\psi$ (which is the stereographic projection of the cone of aperture $\psi$) will, according to detector $C_1$, be in a diffraction position when the rotation $\phi$ brings it to $P_1$. In the same way, a pole located on the circle $\psi_2$ will be in a diffraction position when the rotation $\phi$ brings it to $P_2$.

Angle $\Delta\varphi$ is the azimuthal angular difference between the starting points of the two circles of radius $\psi$ and $\psi_2$.

**Scanning of the pole figure**

A regular scanning of the $(h_1k_1l_1)$ pole figure (5° step in tilting angle and 5° step in azimuth angle, for example) leads to an irregular scanning of the $(h_2k_2l_2)$ pole figure in the tilting angle, whereas the scanning remains regular in the azimuth angle. The measurements given by detector $C_2$ are easily transferred to the $(h_2k_2l_2)$ pole figure by using (1) and (2).

To find the information enclosed in the blind area we can use the Field & Merchant (1949) goniometric method. In this method the rotation $\psi$ is substituted by a rotation $\omega$ whose axis $\omega$ lies in the sample and is perpendicular to the incidence plane: $\omega \cdot \phi = 0$. The range of $\omega$ is $(\theta_1 - \theta_2)$, depending on the size of the blind area.

**Intensity corrections**

We must take into account two kinds of corrections: the absorption of X-rays by the sample and defocusing.

---

**Fig. 3.** Azimuthal difference between the normals $[h_1k_1l_1]^*$ and $[h_2k_2l_2]^*$. $O$, $P_1$, $P_2$: incidence plane, $yOx$: azimuth origin plane.

**Fig. 4.** Stereographic projection on the $Oxy$ plane of the sample.

$\Pi_0$ is the incidence plane when $\psi = 0$; $\Pi_\psi$ is the incidence plane for a given tilting angle $\psi$; the shaded disk is the blind area; $\Delta\varphi$ is the azimuth difference between the circles of radius $\psi$ and $\psi_2$. 
Absorption. In the geometric Bragg–Brentano arrangement used for the measurements of the \((h, k, l)\) pole figure, intensity corrections for absorption are unnecessary when the sample rotates about \(\psi\) and \(\varphi\). On the other hand, detector \(C_2\) is used outside the Bragg–Brentano arrangement. In this case the X-ray intensity caught by detector \(C_2\) is not the same as it would be if the sample were subject to Bragg–Brentano conditions.

Let us consider a parallel X-ray beam of cross section \(S_0 = l \times h\), reaching the sample with an incidence angle \((\theta + \omega)\). The angle of reflection is therefore \((\theta - \omega)\) (Fig. 5). The irradiated area on the sample is

\[
S = S_0 / \sin(\theta + \omega).
\]

(3)

If \(i\) is the intensity diffracted by unit volume located at the surface, the intensity diffracted by an elementary volume \(dv = Sdx\) located at depth \(x\) is

\[
dI = \frac{iS_0 dx}{\sin(\theta + \omega)} \exp\left\{-\mu \left[ \frac{x}{\sin(\theta + \omega)} + \frac{x}{\sin(\theta - \omega)} \right]\right\}.
\]

(4)

where \(\mu\) = linear absorption coefficient. The total intensity is

\[
I_{+\omega} = \int_{0}^{\infty} dI = iS_0 \left[ \frac{1}{\mu} + \frac{\sin(\theta + \omega)}{\sin(\theta - \omega)} \right].
\]

(5)

In the same way, when the incidence angle is \((\theta - \omega)\),

\[
I_{-\omega} = iS_0 \left[ \frac{1}{\mu} + \frac{\sin(\theta - \omega)}{\sin(\theta + \omega)} \right].
\]

(6)

In the Bragg–Brentano arrangement \(\omega = 0\); therefore the total intensity diffracted is

\[
I_0 = iS_0 / 2\mu.
\]

(7)

So the intensity detected must be corrected by a factor \(A\):

\[
A = I_{\omega} / I_0 = 2 \left[ 1 + \frac{\sin(\theta + \omega)}{\sin(\theta - \omega)} \right].
\]

(8)

When \(\omega > 0\) there is an attenuation; when \(\omega < 0\) an amplification. One can show that \(A\) is independent of the rotation \(\psi\) and \(\varphi\) of the sample but depends only on the angle which characterizes the gap between the Bragg–Brentano arrangement and that used to make the measurement.

Defocusing

This question has already been discussed by Couterne & Cizeron (1971) in the case of the Schulz (1949a) goniometric method, with \(\omega = 0\). If \(\omega \neq 0\) the results are quite different. Let us first summarize the results given by Couterne & Cizeron (1971).

\(\omega = 0\). A parallel X-ray beam \(ABCD\) (of width \(l\) and height \(h\)) reaches the sample at incidence angle \(\theta\) (Fig. 6). The irradiated area on the sample \(ABCD\) changes its shape as the tilting angle increases. The projection on the incidence plane \(\Pi\) of the extreme apices of the parallelogram \(A'B'C'D'\) drawn with X-ray beams parallel to the incidence plane give the width \(L\) of the Debye cone. This width is

\[
L = l + 2h \tan(\psi) \cos \theta
\]

(9)

Above some value \(\psi_D\) of the tilting angle, the width \(L\) of the reflected beam becomes larger than the width \(L\) of the detector slits. This leads to a decrease in the intensity recorded by the detector which detects only part of the reflected beam.

\(\omega \neq 0\). The same phenomenon occurs. The Debye cone enlarges as the tilting angle increases. However, the angle \(\omega\) plays an important part: when \(\omega > 0\) the reflected beam is focused whereas when \(\omega < 0\) the reflected beam is spread as we can see in Fig. 5 drawn for \(\psi = 0\).

The width \(L_\omega\) is easily calculated from the geometric pathway of the beams parallel to the incidence
plane as was done when $\omega = 0$. The width is

$$\mathcal{L}_\omega = \frac{\sin(\theta - \omega)}{\sin(\theta + \omega)} + 2h \tan \frac{\theta \cos \theta}{\sin(\theta + \omega)}$$

(10)

So defocusing will occur for an angle $\psi$ larger than $\psi_D$ if we choose $\omega > 0$.

**Transmission case**

(a) Measurement of the $(h_1 k_1 l_1)$ pole figure by the usual method. The sample moves about two rotation axes $\phi$ and $\omega$ which are perpendicular (Fig. 7); $\phi$ lies in the incident plane and is perpendicular to the plane of the sample. The rotation $0 < \phi < 2\pi$ is the azimuthal rotation. $\omega$ is perpendicular to the incidence plane, and is the new tilting angle.

When $\omega = 0$ the $Oxy$ plane of the sample is the bisectrix of $-2\theta_{(h_1 k_1 l_1)}$. The $(h_1 k_1 l_1)$ lattice plane seen by detector $C_1$ has its normal $[h_1 k_1 l_1]^*$ in the plane of the sample and in the incidence plane. When the sample rotates about $\phi$ we scan the border of the pole figure.

When the tilting angle $\omega \neq 0$, detector $C_1$ will see the $(h_1 k_1 l_1)$ lattice plane whose normals are on a cone of aperture:

$$\psi = (\pi/2) - \omega.$$

(11)

Absorption corrections are necessary because of the variation of the thickness of the sample crossed by the X-ray beam and because of the variation of the irradiated area during the movement $\omega$.

(b) Simultaneous measurement of several pole figures.

As in the reflection case a second detector $C_2$ is fixed in the incidence plane at the $2\theta_{(h_2 k_2 l_2)}$ Bragg angle. All the geometric positions the sample can take in relation to detector $C_2$ are those occurring when the sample moves in relation to detector $C_1$. Nevertheless we must be careful with the starting point of the angle $\psi$.

For example, in Fig. 7, if we choose a clockwise rotation $\omega$ starting when $[h_1 k_1 l_1]^*$ is in the plane of the sample, the $(h_1 k_1 l_1)$ pole figure is scanned from its border towards the centre until $\omega = \theta_2 - \theta_1$, whereas the $(h_2 k_2 l_2)$ pole figure is simultaneously scanned from $(\pi/2) - (\theta_2 - \theta_1)$ to the border.

Then (when $\omega > \theta_2 - \theta_1$) the scanning of the $(h_1 k_1 l_1)$ pole figure proceeds normally whereas the same section $[\pi < \psi < (\pi/2) - (\theta_2 - \theta_1)]$ of the $(h_2 k_2 l_2)$ pole figure is scanned again until $\omega = 2(\theta_2 - \theta_1)$.

Then the two scans proceed normally towards the centre. If we stop the scanning with $\omega$ at the final angle $\omega_f$ the $(h_1 k_1 l_1)$ pole figure has been scanned from the border $\psi = \pi/2$ to $\psi = \pi/2 - \omega_f$ and the $(h_2 k_2 l_2)$ pole figure from the border $\psi = \pi/2$ to $\psi = (\pi/2 - (\theta_2 - \theta_1)) - \omega_f$.

**B. Experimental results**

To verify all we have explained up to now, we have used two kinds of samples: a sodium chloride compressed powder very close to an isotropic sample, and a copper sheet coming from a tube, whose texture is not too flat.

(a) Verification of the absorption law

We have seen before that the absorption factor $A$ depends only on the angle $\omega = \theta_2 - \theta_1$. As the position of the $[h_1 k_1 l_1]^*$ normal with respect to the detector depends also on the angle $\omega$ we have chosen for the measurement the isotropic sample. Thus we can avoid intensity variations due to the texture of the sample.

The sample was placed in the Bragg–Brentano arrangement $(\omega = 0)$. The diffracted intensity $I_0$ has been measured. Then the diffracted intensity $I_w$ was measured for different values of $\omega$, characteristic of the gap between the new geometric arrangement and the Bragg–Brentano arrangement.

The results shown in Fig. 8 confirm the absorption law; the slight differences between the theoretical and experimental curves may come from a slight anisotropy of the sample resulting from the uniaxial compression of the powder.

(b) Simultaneous pole figures

(1) The Schulz (1949a, b) classical method (Bragg–Brentano geometric arrangement) was used to obtain...
Table 1. Different parameters for the three experimental arrangements

<table>
<thead>
<tr>
<th>Experiment</th>
<th>1</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\omega = \theta_2 - \theta_1$</td>
<td>$-11.86^\circ$</td>
<td>$+11.86^\circ$</td>
<td></td>
</tr>
<tr>
<td>Absorption factor $A$</td>
<td>1</td>
<td>1.28</td>
<td>0.72</td>
</tr>
<tr>
<td>Beginning of defocusing</td>
<td>$\psi_B$</td>
<td>$\psi &lt; \psi_B$</td>
<td>$\psi &gt; \psi_B$</td>
</tr>
<tr>
<td>Size of the blind area</td>
<td>0</td>
<td>$</td>
<td>\omega</td>
</tr>
</tbody>
</table>

the (220) pole figure of the copper sample. The pole figure was recorded by detector C2 and will be the reference pole figure (Fig. 9a). The experimental conditions were: wavelength = Cu $K\alpha$, $\theta_1 = 37.1^\circ$, $2\theta_2 = 74.20^\circ$, collimator $0.8$ mm diameter, detector slit $H = 8$, $L = 4$ mm, tilting step $\Delta \psi = 2.5^\circ$, azimuth step $\Delta \phi = 5^\circ$, X-ray power $40$ kV, $18$ mA.

(2) Then, with the same experimental conditions other than the position of the sample ($\theta_1 = 25.24^\circ$), we recorded the (220) pole figure with detector $C_2$ ($2\theta_2 = 74.20^\circ$) and at the same time recorded the (200) pole figure with detector $C_1$ ($2\theta_1 = 50.48^\circ$).

Detector $C_1$ ($2\theta_1 = 50.48^\circ$) and the sample ($\theta_1 = 25.24^\circ$) were in the Bragg–Brentano geometric arrangement. If we refer to the first experiment and to detector $C_2$, the sample was rotated by $\omega$ with $\omega = \theta_2 - \theta_1 = -11.86^\circ$; detector $C_2$ ($2\theta_2 = 74.20^\circ$) and the sample ($\theta_1 = 25.24^\circ$) were not in the Bragg–Brentano arrangement. Thus we obtained with $C_2$ a new (220) pole figure (Fig. 9b).

(3) We have also recorded the (220) pole figure with detector $C_2$, the sample being placed in a symmetrical position in relation to the Bragg–Brentano arrangement. In this experiment $\omega = +11.86^\circ$. We thus obtained Fig. 9(d).

Discussion of the results

All the pole figures have been drawn with the same intensity level. In Table 1 we point out the differences expected for the three (220) pole figures.

Looking at the pole figures obtained, we notice the blind areas at the centres of Figs. 9(b) and (d). Their angular radii are $\omega$ as expected. The intensities of Fig. 9(b) are higher than those of (a) whereas those of (d) are lower. This is normal if we take absorption into account.

Figs. 9(c) and (e) show (b) and (d) corrected by the absorption factor. We notice the very good intensity agreement of these figures with the reference pole figure (a).

One can see in (c) and (e) the influence of defocusing. In (c) the two small peaks on the border have disappeared because the defocusing begins at a smaller tilting angle.

C. Concluding remarks

We have shown that it is possible to obtain simultaneously and without any loss of precision several pole figures. Intensity variations due mainly to a geometric arrangement different from the Schulz (1949a, b) arrangement are easily corrected. Even defocusing could be limited if we choose a geometry with $\omega > 0$.

With new position-sensitive X-ray detectors (Ballon, Comparat & Pouxe, 1983), it is possible to record simultaneously the whole 2$\theta$ spectrum, i.e. the beams diffracted by several $(h,k,l)$ lattice planes. We can see how interesting such detectors are for texture.
analysis. Moreover, most intensity problems could be solved by this kind of detector because it could be considered as a variable-slit detector; for example, in the defocusing phenomenon the widening of the diffracted beam during the tilting movement of the sample can be taken into account.

As a large amount of information is brought simultaneously from this kind of detector to the microcomputer one needs a high data-transfer rate to preserve the time saved by this kind of detector. We shall shortly describe the use of a position-sensitive X-ray detector connected to an X-ray texture goniometer.

References