Quantitative Determinations and Descriptions
of Preferred Orientation*

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MASTER

INTRODUCTION

Pole figures are an unprejudiced graphical description of preferred orientation. However, such graphical descriptions may only be qualitatively useful, even if determined using quantitative methods of measuring diffraction intensity. The problem is particularly acute if one desires a quantitative numerical description of the preferred orientation, i.e., relative volume of material associated with a given set of intensity maxima. Without such quantitative descriptions, any attempt to predict material anisotropy based on preferred orientation data is at best only a qualitative prediction. Present x-ray techniques such as reflection methods can be employed to yield representative data for complete descriptions of the preferred orientation. These data can be treated by computer and/or graphical methods to determine relative volumes in a given orientation. This paper describes a proposed approach to this problem.

DATA COLLECTION

A single method for compiling all the data necessary for complete description of a preferred orientation is desired.

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One reason for this is that difficulty is usually encountered in mating x-ray data in the annulus of overlap if reflection and transmission data are used in preferred orientation studies. This mating would be difficult to handle in subsequent data treatment by a digital computer. A reflection method such as the modified Schulz method using orthogonal reflection surfaces (1,2,3)* yields data in a form that can be readily handled by a computer program. The offset reflection method described by Meieran (4) or Lopata and Kula (5) even further simplifies subsequent data handling. Specimen preparation in the offset reflection method consists of making a cube from the specimen material. The cube is approximately 3/4 inch on edge. A corner is cut off the cube exposing a reflection surface whose normal is 54.74° from the orthogonal axes of the cube. If the cube is laminated from sheet material such as rolled metal, the exposed plane represents an average preferred orientation over the cross-section of each lamellar sheet; hence, the texture gradient that frequently occurs in rolled metal sheet is averaged, and the surface is representative of the entire cross-section. Alternatively, the sheets can be prepared and thinned prior to lamination so that the reflection surface is representative of a selected layer in the sheet.

* Denotes references included at the end of the paper as a list of references.

A Schulz goniometer (1) may be used for both the "normal" and "offset" reflection methods, see Fig. 1, and the diffracted x-ray intensity detected using an electronic counter.* Rather than continuously rotate the specimen simultaneously through both ϕ' and α' at some fixed rate, the data may be recorded in a more convenient form if ϕ' is held constant while α' rotates. The angle, ϕ' is measured in degrees of latitude from the starting position and α' is degrees of longitude from the starting position. The angles ϕ' and α' may or may not be identical to the usual ϕ and α stereographic coordinates depending on the starting position. After α' has rotated from 0 to 360 degrees, the goniometer is programmed to advance through a fixed increment, e.g., 2.5° , in the angle ϕ' .

Ratemeter counting is often used to determine the diffracted intensity as a function of lapsed time which can be related to spatial position of the specimen reflection surface. These data are usually recorded on strip charts; however, if the data are to be numerically integrated, it is advantageous to use scaler counting at preselected spatial positions. Fixed intervals of

*The goniometer alignment may be accurately checked using a randomly oriented standard. If properly aligned, the Bragg angle will not change as the standard is moved through ϕ' and α' rotations. Changing Bragg angles indicate that the ϕ' rotation axis is not coincident with the specimen surface or the specimen reflecting surface is not normal to the α' axis.

2.5 to 5° in the angle α' are suitable counting positions. Fixed counts for each spatial position of the specimen insure that the data are equally reliable in a statistical sense. This, however, is usually impractical because of the long times required to attain a reasonable fixed count at positions near distribution minima.* As a result, fixed time scaler counting is the more practical approach. Subsequent data treatment can be done with greater facility if the diffraction data for each position are recorded in a print-out form.**

In order to achieve clear definition of diffracted intensity distribution, the diffractometer slit system should be selected for good resolution. This also decreases the chance of encountering spurious diffraction peaks (6). Spurious diffraction can also be avoided by using pulse height analysis to discriminate diffraction due to longer or shorter wavelengths than the $K\alpha$ radiation used. The masking effects due to low peak to noise ratios, spurious diffraction, and near characteristic wavelengths that pass the monochromating filter are reduced by selecting the more intense diffraction lines.

*The distribution of material at a given spatial position, (ϕ_i', α_i') , is directly related to the diffraction intensity at that position.

**If intensities are counted and recorded in 2.5° intervals of both ϕ' and α' , then approximately 3169 ($[22 \times 144] + 1$) datum points are involved.

DATA STANDARDIZATION

It is necessary to correct the data for background radiation. The method employed in many preferred orientation studies is to average the background intensity on both sides of a Bragg diffraction maxima. This background intensity, I_{back} , is then subtracted from the intensity observed, $I_{\text{obs}}(\phi'_i, \alpha'_i)$, at a given spatial position, (ϕ'_i, α'_i) , as follows:

$$I(\phi'_i, \alpha'_i) = I_{\text{obs}}(\phi'_i, \alpha'_i) - I_{\text{back}} \quad (1)$$

In both the standard Schulz and offset reflection techniques, defocusing of the diffracted beam occurs (3,7) as the goniometer ring is rotated from the zero position to angles further removed from the starting position, i.e., for increasing ϕ' .^{*} This defocusing can, and usually does, result in appreciable loss of diffracted intensity; the loss, $-\Delta I_{\text{obs}}$, increases with increasing ϕ' . This decrease in intensity varies with the slit assemblies, alignment, and diffraction line. It can, however, be determined experimentally by observing the change in diffracted intensity from a randomly oriented standard as ϕ' changes. The experimental data are then corrected for each ϕ' position of the specimen by a factor equivalent to the change in random intensity, I_R , i.e., $[I_R(0, \alpha'_i)] / [I_R(\phi'_i, \alpha'_i)]$.

^{*}If one considers the first quadrant of a stereographic projection, the starting position in the offset reflection method is centered in that quadrant. Hence, the relative ϕ position, ϕ' , is zero at the starting position whereas the absolute ϕ position, ϕ_0 , is 54.74° .

After the data are corrected for defocusing and background radiation, the data are standardized. A direct method of standardization is to divide the corrected observed intensity, $I_E(\phi'_i, \alpha'_i)$, at a given spatial position, (ϕ'_i, α'_i) , by the random intensity (corrected for defocusing and background), i.e., $[I_E(\phi'_i, \alpha'_i)] / [I_R(o, \alpha'_i)]$.

The following expression may be used to obtain I_E ,

$$I_E(\phi'_i, \alpha'_i) = \left[I(\phi'_i, \alpha'_i) \right] \left[I_R(o, \alpha'_i) / I_R(\phi'_i, \alpha'_i) \right] \quad (2)$$

The standardized intensity is related to I_E by;

$$I_S(\phi'_i, \alpha'_i) = I_E(\phi'_i, \alpha'_i) / I_R(o, \alpha'_i) \quad (3)$$

Then

$$I_S(\phi'_i, \alpha'_i) = I(\phi'_i, \alpha'_i) / I_R(\phi'_i, \alpha'_i) \quad , \quad (4)$$

where I is the observed intensity corrected for background (see equation (3)); I_E is the experimental intensity corrected for defocusing; I_S is the standardized intensity; $I_R(o, \alpha'_i)$ is the intensity (corrected for background) of a random sample at $\phi' = 0^\circ$; $I_R(\phi'_i, \alpha'_i)$ is the intensity (corrected for background) of a random sample for arbitrary ϕ'_i . From this it follows that the correction for defocusing and the standardization can be done in a single step provided $I_R(\phi'_i, \alpha'_i)$ is known for each spatial position, (ϕ'_i, α'_i) . Since I_R does not depend on α'_i , twenty-three independent values of $I_R(\phi'_i, \alpha'_i)$ are required for the offset reflection method and a $\Delta\phi'$ of 2.5° .

The background values and random values at the various ϕ' settings can be used in a computer routine to correct and standardize the raw data, I_{obs} . The values of $I_{\text{obs}}(\phi'_i, \alpha'_i)$ are part of the computer input.

DATA TREATMENT

The standardized intensity data can be plotted using a rectilinear plotter (3) if one transforms the ϕ, α coordinates for each I_S reading to equivalent Cartesian coordinates. Due to symmetry in pole figures only one quadrant of a stereographic projection need be plotted in most cases. For a polar stereographic projection oriented with the pole at the origin of the total projection, the stereographic coordinates, ϕ and α , can be converted to rectilinear coordinates (3) by the following expressions (see Fig. 2).

$$x = \frac{2 \sin \phi}{\cos \phi + 1} \cos \alpha \quad (5)^*$$

$$y = \frac{2 \sin \phi}{\cos \phi + 1} \sin \alpha \quad (6)$$

where ϕ is the degrees of latitude of the position from the origin, and α is the degrees of longitude relative to the origin.

*The angle α is the radial azimuthal angle and ϕ is the polar azimuthal angle in spherical coordinates.

The starting position in the offset reflection method (4,5) does not coincide with the origin, but is $\phi_0 = 54.74^\circ$, and $\alpha_0 = 45^\circ$ from the origin. Since angles are measured from the starting position, this necessitates a conversion of ϕ' and α' positions relative to the starting position, i.e., the measured angles, to the angles ϕ and α which are relative to the origin. The situation is described in Fig. 3 where x and y axes are as indicated and the positive z direction is into the plane of the paper. The angles ϕ and α are functions of both ϕ' and α' . By using vector calculus, one can find the i, j, and k vectors of the starting position and equate i, j, and k components for the starting position and the origin. The resulting expressions relate ϕ and α to ϕ_0 , α_0 , ϕ' and α' where ϕ_0 and α_0 are the coordinates relative to the origin of the starting position; ϕ' and α' are the angles measured from the starting position. The expressions are:

$$\begin{aligned} \sin\phi \cos\alpha &= \sin\phi' \cos\alpha' \cos\phi_0 \cos\alpha_0 \\ &\quad - \sin\phi' \sin\alpha' \sin\alpha_0 \\ &\quad + \cos\phi' \sin\phi_0 \cos\alpha_0, \end{aligned} \tag{7}$$

$$\begin{aligned} \sin\phi \sin\alpha &= \sin\phi' \cos\alpha' \cos\phi_0 \sin\alpha_0 \\ &\quad + \sin\phi' \sin\alpha' \cos\alpha_0 \\ &\quad + \cos\phi' \sin\phi_0 \sin\alpha_0, \end{aligned} \tag{8}$$

$$\cos\phi = - \sin\phi' \cos\alpha' \sin\phi_0 + \cos\phi' \cos\phi_0. \tag{9}$$

Substituting equations (7), (8) and (9) into equations (5) and (6), the following are obtained:

$$x = \frac{2(\sin\phi' \cos\alpha' \cos\phi_0 \cos\alpha_0 - \sin\phi' \sin\alpha' \sin\alpha_0 + \cos\phi' \sin\phi_0 \cos\alpha_0)}{-\sin\phi' \cos\alpha' \sin\phi_0 + \cos\phi' \cos\phi_0 + 1} \quad (10)$$

$$y = \frac{2(\sin\phi' \cos\alpha' \cos\phi_0 \sin\alpha_0 + \sin\phi' \sin\alpha' \cos\alpha_0 + \cos\phi' \sin\phi_0 \sin\alpha_0)}{-\sin\phi' \cos\alpha' \sin\phi_0 + \cos\phi' \cos\phi_0 + 1} \quad (11)$$

The angles ϕ_0 , α_0 , ϕ' and α' are all part of the data input into a digital computer. The computer then can convert the ϕ' , α' coordinates of each spatial position to x, y coordinates which represent these spatial positions in stereographic projection.

The standardized intensity, I_S , (see equation (4)) for each specimen position and the x, y coordinates of these positions can be obtained as computer output. In turn, this output can be used in a rectilinear plotter to plot the stereographic projection of the preferred orientation data which is similar to a method previously described by Holland et al (3). Because of the great number of $I_S(\phi'_i, \alpha'_i)$ values generated in the data collection, it may be less cumbersome to plot the pole figure using selected values. A reasonable mode of selection is to plot those values that lie on or very near whole number multiples of I_R . These then can be used to fix iso-intensity (standardized intensity) contours of the pole figure.

After the datum points for the pole figure are plotted and iso-intensity contours are established, the pole figure is analyzed in terms of the crystallographic components of the preferred orientation. This can be done in the usual manner of comparing the positions of intensity maxima with pole positions in a standard projection.*

After obtaining a satisfactory crystallographic solution of the preferred orientation, the problem remains to determine a quantitative index of the texture strength and the relative volumes associated with texture components. Essentially this is a surface integration problem, and numerical integration by a computer program can be used to obtain the desired result. A scan of the corrected (but unstandardized) intensity data $I_E(\phi'_i, \alpha'_i)$ for maxima and minima can be made on a computer, and the data integrated over the first quadrant of a stereographic projection. The integration over the interval from $\phi = 0$ to $\phi = \frac{\pi}{2}$ and $\alpha = 0$ to $\alpha = \frac{\pi}{2}$, where ϕ and α are the spherical coordinates previously noted will give the pole concentration per unit area, i.e., the corrected intensity, for an ideally randomly oriented specimen for an octant of the reference sphere.

*An improvement on this would be to use a computer to do the matching and in this way obtain crystallographic solutions. This is a more difficult problem than correcting and plotting the data, and the best manner to accomplish this is not obvious.

That is,

$$I_{R'} = \frac{2}{\pi} \int_{\phi=0}^{\phi=\frac{\pi}{2}} \int_{\alpha=0}^{\alpha=\frac{\pi}{2}} I_E(\phi, \alpha) \sin \phi \, d\phi \, d\alpha \quad , \quad (12a)$$

where $\frac{\pi}{2}$ is the area of an octant of a sphere of unit radius. This is equivalent to a summation of the experimental intensities, $I_E(\phi_i, \alpha_i)$, at N given positions, (ϕ_i, α_i) , over the range from $\phi=0$ to $\phi=\frac{\pi}{2}$ and $\alpha=0$ to $\alpha=\frac{\pi}{2}$ as follows:

$$I_{R'} = \frac{2}{\pi} \sum_{i=1}^N I_E(\phi_i, \alpha_i) \sin \phi_i \Delta \phi_i \Delta \alpha_i \quad , \quad (12b)$$

or

$$I_{R'} = \frac{1}{N} \sum_{i=1}^N I_E(\phi_i, \alpha_i) \quad , \quad (12c)$$

where $0 \leq \phi_i \leq \frac{\pi}{2}$ and $0 \leq \alpha_i \leq \frac{\pi}{2}$. This value of random intensity should agree with the corrected experimentally determined value, I_R . The agreement will depend, in part, on the number of datum points used in the summation for $I_{R'}$, and upon how near ideal is the prepared random sample* used in determining I_R .

After scanning the intensity data for maxima and minima, the volume of material within t degrees of the peak intensity can be determined by integrating over a solid angle of t degrees.

* Besides being ideally randomly oriented the standard should be in a strain free condition, otherwise the random intensity will be reduced.

Mathematically this may be expressed as

$$I_t = \int_{\phi_i - \Delta\phi}^{\phi_i + \Delta\phi} \int_{\alpha_i - \Delta\alpha}^{\alpha_i + \Delta\alpha} I_E(\phi, \alpha) \sin\phi \, d\phi \, d\alpha \quad (13)$$

where (ϕ_i, α_i) are the position of an intensity maximum (or minimum) and $\Delta\phi$ and $\Delta\alpha$ are the limits of interest (defining the solid angle). The relative volume can be determined by taking the ratio of I_t to I_R , as follows:

$$V_{rel} = \frac{I_t}{I_R} \quad (14)$$

The total relative volume within t degrees of a set of diffraction maxima associated with a given texture component, x , is the sum of the relative volumes for each maximum associated with the x component divided by the multiplicity factor, n . This can be expressed as;

$$V_x = \frac{(I_{t_1} + I_{t_2} + I_{t_3} + \dots + I_{t_n})}{I_R} \frac{1}{n}, \quad (15)$$

where n is equal to the number of maxima associated with the x component and lying within the first quadrant.

The deviation of the material from an ideally randomly oriented condition can be determined by computing the standard deviation. This is a numerical index of the degree of preferred orientation. In this case the standard deviation, σ , may be calculated from the generalized expression as follows:

$$\sigma^2 = \frac{\sum_{i=1}^N X_i^2 - \frac{1}{N} \left(\sum_{i=1}^N X_i \right)^2}{N - 1} \quad (16)$$

If the number of datum points, N , is large so that $N - 1 \approx N$, letting I_E be the experimental observations X_i , and substituting equation (12c), then the expression for σ becomes;

$$\sigma = \sqrt{\frac{\sum_{i=1}^N I_E(\phi_i, \alpha_i)^2}{N} - (I_{R'})^2} \quad (17)$$

This computation, as well as those for V_{rel} (see equation (14)) can be done by the computer.

The integration can also be done by graphical means (8) but this would require considerable manual manipulation. In the first place the corrected intensity data, I_E , would have to be plotted on an equal area projection such as a Lambert azimuthal equal area projection. This would require manual plotting or yet another computer transformation of coordinates and plotting on a

rectilinear plotter. The integration could be done by standard means, such as using a planimeter. There is an inherent error in all equal area projections and in case of the Lambert projection the error increases as the arc distance from the center of the projection increases (the error is 15.5 percent at 60° of arc). It appears that the graphical integration is more laborious and less accurate than numerical integration by a computer routine.

CONCLUSION

The proposed method using present x-ray techniques and computer treatment of the raw preferred orientation data will correct the data for inherent errors, generate data to plot the pole figures using a rectilinear plotter, determine the relative volumes of materials associated with strong texture components, determine the degree of preferred orientation by calculating the standard deviation from the ideally randomly oriented state, and provides an independent check on the value of random intensity used to standardize the data.

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CAPTIONS

Fig. 1 Schematic description of Schulz goniometer (1). The rotations ϕ' and α' correspond to rotations in degrees of latitude and longitude respectively. If the initial starting position, i.e., the normal to specimen surface, coincides with the polar position then ϕ' and α' correspond directly to the ϕ and α stereographic coordinates.

Fig. 2 A polar stereographic projection. The coordinates ϕ and α are indicated.

Fig. 3 Polar stereographic projection rotated 54.74° from the origin, as in the offset reflection method. The starting position for the offset method is at the polar position. The origin is at the intersection of the x, y axes. In the Cartesian coordinates shown the positive z direction is into the plane of the drawing.

Fig. 1

Fig. 2

Fig. 3