Textures of Cold-Rolled Pure Aluminum Measured by Neutron and X-ray Diffraction

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(Received 21 July 1975; accepted 7 August 1975)

The textures of undirectionally and reversed rolled aluminum sheets were measured using thermal neutrons and Cu and Mo Kα radiation. The samples for the X-ray measurements were prepared in three different ways so that the degree of correspondence between textures as measured with neutrons and X-rays could be investigated. It is shown that agreement can be achieved if the X-rays used penetrate sufficiently into the sample and if a composite type sample is used.

1. Introduction

The X-ray diffraction technique offers an efficient tool for the investigation of crystallographic orientations in polycrystalline materials. Nevertheless it exhibits the following difficulties:

1. Problems due to high absorption:
   1.1. The small depth of penetration restricts investigations to thin surface layers, which are not always representative of the bulk material (surface textures).
   1.2. The irradiated volume may contain only a limited sample of crystallites (crystallite statistics).
   1.3. Uncertainties of the absorption correction in the transmission technique.

2. Problems due to the applied experimental procedure:
   2.1. In the commonly used back-reflexion arrangement defocusing of the diffracted intensity makes it impossible to measure the equatorial parts of the pole figure. To get complete pole figures data produced in reflexion are combined with data gained in transmission or a composite sampling technique is used. Both methods require great preparational efforts (sample preparation).
   2.2. The usual back-reflexion and transmission technique and also the spherical sample method proposed by Jetter & Borie (1953) do not permit irradiation of the same sample volume for every angular position. In the case of great inhomogeneities in the texture the pole figure does not represent an integral texture, but a mosaic of different textures corresponding to different parts of the sample (texture mosaic).

By using thermal neutrons for bulk texture measurements most of these problems are eliminated. The very small absorption cross section for nearly all elements makes it possible to irradiate sample volumes which are about three orders of magnitude bigger than sample volumes in X-ray work.

The high transparency of most metals to neutrons was first used by Brockhouse (1953) for his investigation of annealing textures in nickel wires and by Swalin & Geisler (1956), who studied the wire textures of several b.c.c. metals.

Texture inhomogeneities, crystallite statistics and sample preparation present no problems for the experimenter. If, e.g., spherical samples are used, which are smaller than the cross section of the incident beam, the same sample volume is irradiated at every angular position. Besides these great advantages neutron diffraction has also two drawbacks:

1. The intensity available from nuclear reactors makes experiments very time consuming.
2. Multiple scattering weakens the essential assumption of the proportionality between diffracted intensity and sample volume with planes (hkl) in diffraction position.

Multiple scattering and the self shielding of the sample are limits to the accuracy of the neutron diffraction technique. However, both effects can be controlled by choosing the right sample volume, so that corrections are either negligible or can be calculated numerically.

The great accuracy of recent texture analysis, especially the computation of the three-dimensional orientation distribution function requires pole figures which are as precise as possible. Therefore neutron diffraction has been applied at an increasing rate. The cyclic textures in cold-drawn Al wires (Schlafé & Bunge, 1972), the texture transition in α-brasses (Bunge & Tobisch, 1972), the development of the rolling textures...
of iron (Schläfer & Bunge, 1974) and the recrystallization textures in cold-rolled low-carbon steel (Bunge, Schleusener & Schläfer, 1974) were determined by neutron diffraction.

Kajamaa (1968) investigated the cold-rolling and recrystallization textures in copper sheets by neutron diffraction. In addition he made a comparison between X-ray and neutron pole figures of the cold-rolled sheet. The X-ray measurement was done by the transmission method, yielding only an incomplete pole figure. He concluded that the correspondence between the pole figures depends on the angular distance from the normal direction of the sheet. The equatorial parts were nearly identical.

In the present work the rolling texture of Al was measured with X-ray and neutron techniques with the aim of investigating the degree of correspondence possible between neutron and X-ray data, when both diffraction techniques are carried out on identical samples – see § 3. In the X-ray work we varied the incident wavelength, sample preparation and experimental procedure.

2. Experimental technique

The neutron diffraction measurements were made at a swimming-pool 7 MW Reactor using a conventional triple-axis neutron spectrometer in a two-axis mode. The collimations, beginning with the in-PILE collimator were \( \alpha_1 = 30^\circ \), \( \alpha_2 = 25^\circ \) and \( \alpha_3 = 25^\circ \). A Zn (002) crystal with a mosaic spread of 15' was used as monochromator. The measurements were performed at \( \lambda = 1.225 \) Å. The samples were mounted on a goniometer, with a rotational range of \( \pm 20^\circ \) and were sufficiently small to be bathed fully in the neutron beam. The experimental setup, the sample rotations through the angles \( \alpha \) and \( \phi \) and the corresponding stereographic projection are shown in Fig. 1. A sector of 30° was measured automatically in steps of 2 \( \times \) 2°. Three sectors formed a quadrant of the pole figure. The accuracy of the sample adjustment was \( \pm 0.2^\circ \). One-quadrant \((111), (200)\) and \((220)\) pole figures were taken for three Al-specimens. The measurement time per sample was 68 h. About one third of the time was used for sample positioning. The diffraction data were corrected for background scattering and normalized in random units.

The X-ray work was carried out using a Siemens texture goniometer and filtered Cu and Mo K\( \alpha \) radiation. The back-reflexion technique was applied in all measurements up to a specimen-tilting angle of 75°. The same pole figures as in the neutron work were measured. Most of the pole figures were obtained by continuously recording the diffracted intensity; the composite sample (sample 2) and the quadrants of sample 1 were measured in steps of 2 \( \times \) 2°. The observed intensity was corrected for defocusing and background scattering. The counting rate with Mo K\( \alpha \) \((V=24 \text{ kV}, 18 \text{ mA})\) radiation irradiating a sample area of \( 4.7 \text{ cm}^2 \) is about a factor of five higher than the counting rate in the neutron experiment with a sample volume of \( 19.2 \text{ cm}^3 \).

3. Sample preparation

Aluminum slabs of 99-99.9% purity were rolled in 20 passes to a final thickness of 2 mm, which corresponds to a reduction of 90%. Circular discs with a diameter of 35 mm were cut from the sheets. For the measurements with neutrons three samples were prepared. Every sample consisted of 10 discs stacked one upon another so as to preserve the rolling direction. They were fixed and pressed together by the clamps of the sample holder. The rolling operation was carried out in three different ways: for sample 1 the sheets were rolled unidirectionally; for sample 2 as for sample 1 but the sheets were rotated about the rolling direction between the successive passes, and for sample 3 the sheets were reversed end to end after each pass.

For the X-ray measurements the discs of sample 1 were etched with dilute hydrochloric acid solution. Approximately 0.1 mm of the surface layer was removed. The discs of sample 2 were used to obtain a composite type sample as described by Lopata & Kula (1962). They were bonded together by a one-component fluid adhesive (Kores K 1). A cut was made at 54.7° to the normal, transverse and rolling
directions of the sheet with a spark-erosion technique. No variation in the diffracting power of the specimen surface due to the bonding medium was observed. The discs of sample 3 were measured in the as-rolled condition without etching or polishing.

A random sample was produced by pressing fine-grained Al powder in a cylindrical shape to a density of 80\%.

4. Results

The intensity contours on all the pole figures are plotted in multiples of the random-intensity level. Figs. 2 to 4, 5 to 7 and 8 to 10 show the pole figures of the Al samples 1, 2 and 3 respectively. The pole figures of Figs. 2, 5, 8 were measured with thermal neutrons, those of Figs. 3, 6, 9 with Cu Kα radiation and those of
Figs. 4, 7, 10 with Mo Kα radiation. As shown in Fig. 11 the (111), (200) and (220) poles of the ideal orientation (124) [211] are identified by 1 to 4, 1 to 3 and 1 to 6 respectively. One prime over the corresponding pole number indicates poles which are produced by reflexion through the plane formed by the normal direction (ND) and the rolling direction (RD) and two primes indicate the pole's reflexion through the plane formed by ND and the transverse direction (TD). The four components of the ideal orientation (124) [211] are labeled I to IV.

The textures measured by neutron diffraction could be described satisfactorily by the ideal orientations (124) [211], (135) [21T] or (2510) [1065]. The highest pole density in the (111) pole figures of samples 1 and 2 occurred at θ = 5° and α = 31°, which is near the pole 1

Fig. 4. (a) (111), (b) (200), (c) (220) pole figures of one disc of sample 1 measured with Mo Kα radiation: △. (124) [211]; • poles of rotated component I.

Fig. 5. (a) (111), (b) (200), (c) (220) pole figures of sample 2 determined by neutron diffraction: △. (124) [211]; □. (135) [21T]; •. (2510) [1065].

Fig. 6. (a) (111), (b) (200), (c) (220) pole figures of sample 2 measured with Cu Kα radiation: △. (124) [211]; □. (135) [21T]; •. (2510) [1065].
of the orientation (2 5 10) [10 6 5]. In the case of sample 3 the maximum of this pole concentration was recorded at $\phi=0^\circ$ and $\gamma=28^\circ$, which is exactly the position of pole 1 of the orientation (124) [211]. The $(hkl)$ pole densities of the ideal orientation (124) [211] were calculated by integrating the diffracted intensity in an element of solid angle which is centred around the poles of this orientation. Table 1 lists the pole densities normalized in units of the random sample. A comparison between the pole densities belonging to different samples is made, giving the percentage change relative to the pole densities of sample 1.

The comparison between the three data groups yields the following results:

Fig. 7. (a) (111), (b) (200), (c) (220) pole figures of sample 2 measured with Mo Kα radiation: ▲. (124) [211]: □. (135) [211]: ●. (2 5 10) [10 6 5].

Fig. 8. (a) (111), (b) (200), (c) (220) pole figures of sample 3 determined by neutron diffraction: ▲. (124) [211]: □. (135) [211]: ●. (2 5 10) [10 6 5].

Fig. 9. (a) (111), (b) (200), (c) (220) pole figures of one disc of sample 3 measured with Cu Kα radiation: ▲. (124) [211].
4.1 Comparison of sample 1 data:

In Figs. 3 and 4 the X-ray pole figures of one of the ten discs of sample 1 are shown. They exhibit a strong asymmetry about RD and TD.

The pole concentrations of the Mo Kα figures are related to the unprimed poles of the orientation (124) [211], the component I, by a rotation, which can be characterized by the orientation distance γ. As shown in Fig. 4 this quantity is the largest of the three angles between the ideal (200) poles and the corresponding pole concentrations and has a value of 18°. To ensure that this disc is representative of the sample, the (111) pole figures of all ten discs were measured with Mo Kα radiation. Only pole concentrations corresponding to the rotated components I and II were observed. The poles of the components III and IV have low pole densities or were not observed at all.

The pole figures measured with Cu Kα radiation show the same asymmetric distortion of the pole concentrations. The quadrants of Fig. 3(d) and 3(e) are an average taken from measurements of three discs. Even here the poles of the rotated components I and II describe the pole concentrations satisfactorily.

The neutron pole figures show a much higher degree

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Fig. 10. (a) (111), (b) (200), (c) (220) pole figures of one disc of sample 3 measured with Mo Kα radiation; ▲, (124) [211].

Fig. 11. (a) (111), (b) (200), (c) (220) pole figures showing the poles of the ideal orientation (124) [211].

Table 1. The (111) and (200) pole densities P in random units measured with neutrons

<table>
<thead>
<tr>
<th>Sample</th>
<th>(111)poles</th>
<th>(200)poles</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>P₁</td>
<td>P₂</td>
</tr>
<tr>
<td>1</td>
<td>5.2±0.13</td>
<td>5.4±0.14</td>
</tr>
<tr>
<td>2</td>
<td>3.1±0.08</td>
<td>3.9±0.10</td>
</tr>
<tr>
<td>3'</td>
<td>2.6±0.07</td>
<td>3.7±0.10</td>
</tr>
<tr>
<td>4'</td>
<td>1.9±0.05</td>
<td>2.3±0.06</td>
</tr>
<tr>
<td>1</td>
<td>5.9±0.14</td>
<td>5.7±0.14</td>
</tr>
<tr>
<td>3'</td>
<td>3.5±0.08</td>
<td>3.0±0.07</td>
</tr>
<tr>
<td>2'</td>
<td>2.4±0.06</td>
<td>3.0±0.07</td>
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of symmetry; however the (111) poles 3" and 4" and the (200) poles 3' and 2' have lower pole densities than the poles with unprimed numbers. These poles also have lower pole densities than the poles in the neutron pole figures of samples 2 and 3.

4.2 Comparison of sample 2 data

The X-ray and neutron pole figures show a very high degree of correspondence.

The Mo Kα and the neutron pole figures are nearly congruent. Their pole concentrations are at the same angular positions. The (111) poles 1 and 3" of the Cu figure are shifted about 2° relative to the peaks in the neutron figure. The pole 1 is at φ = 0°, α = 29° and the pole 3" at φ = 16°, α = 90° instead of φ = 0°, α = 31° and φ = 18°, α = 90°, respectively. In Table 2 the ratio $I_{TD}/I_{2O}$ is shown, where $I_{TD}$ is the intensity at φ = ±90°, α = 90°, and $I_{2O}$ is the intensity at φ = ±20°, α = 90°. The error due to counting statistics is ±2.4°.

The ratio is proportional to the stacking-fault energy, as was demonstrated by Hu & Goodman (1963). The best correspondence between the neutron ratio and the X-ray ratio yields the Mo Kα data.

Table 2. The ratio $I_{TD}/I_{2O}$ measured with neutrons and X-rays

<table>
<thead>
<tr>
<th>Neutrons</th>
<th>$I_{TD}/I_{2O}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>X-rays</td>
<td>Mo Kα</td>
</tr>
<tr>
<td>X-rays</td>
<td>Cu Kα</td>
</tr>
<tr>
<td>Neutrons</td>
<td>0.72 ± 0.017</td>
</tr>
<tr>
<td>X-rays</td>
<td>0.75 ± 0.018</td>
</tr>
<tr>
<td>X-rays</td>
<td>0.77 ± 0.018</td>
</tr>
</tbody>
</table>

In spite of that overall good agreement there are still differences between the pole densities of the neutron and X-ray pole figures. Table 3 lists the intensities at the maximum point of the pole concentrations. The error due to counting statistics is between 0.1 and 2°.

The (200) poles show the strongest deviations. The peak intensity of both X-ray poles 1 is reduced by 26° relative to the high intensity of this pole in the neutron figure. On the other hand the peak intensities of the poles 3' and 2" increase by 24% and 39% for Mo Kα and 45% and 42% for Cu Kα respectively. The differences of the (111) peak intensities are in no case greater than 11°.

4.3 Comparison of sample 3 data

Figs. 9 and 10 show the pole figures of one disc of sample 3. The four components of the (124) [211] orientation account fairly well for all the high-intensity areas except those (111) poles which are near the RD at φ = 16° and α = 64°. No distortion of the pole concentration was observed. Also here the (111) pole figures of the 10 discs were measured with Mo Kα radiation to ensure that this disc is representative for the sample. In all ten discs the same agreement with the (124) [211] orientation was observed. The pole densities of the four components fluctuate very strongly within the ten samples investigated. Sometimes the maxima near the RD were missing. The azimuthal distance φ of the two (111) poles 2 and 2' was determined by averaging over the ten measurements. The distance φ is 179.5°, where φ is 180° in case of complete symmetry. In four discs the (111) pole 1" has higher pole density than the pole 1.

The pole figures measured with Cu Kα radiation have broad and smeared-out pole concentrations. Though some poles are missing or have very low densities, the whole pattern of the pole concentration corresponds quite well to the (124) [211] ideal orientation. The (111) pole concentrations near the RD are shifted to the ND pole. Their polar angle α is about 4° smaller than the one of the Mo Kα figure. The (200) pole concentrations 3, 3' and 3" are missing.

5. Conclusions

From the three sets of data the results of the composite-type sample 2 are the most interesting. There is a high degree of correspondence regarding the overall shape and the angular positions of the pole concentrations between the data taken with neutrons and with Mo Kα radiation. This agreement is already less pronounced when the neutron data is compared with the data taken with Cu Kα radiation where the poles are already slightly shifted.

The different pole densities obtained with X-rays and neutrons are mainly due to the limited volume seen by the X-rays. For the X-ray measurement sample 2 was prepared in such a way that the surface and the bulk volume of all ten discs contributed to the measured texture as in the case of the neutron measurements. Nevertheless the irradiated volume is still only a fraction of the volume over which the neutron measurement is averaging. In the cases of samples 1 and 3 the X-ray pole figures contain no contribution from the orientations.
bulk of the sample. The measured texture is only representative of the surface layers and does not give an average over the ten discs. Therefore correspondence with the neutron pole figures is not to be expected.

The authors would like to thank Professor P. Weinzierl for his continual support. We are indebted to Dr. Kunsch and D. I. Schoitsch for their help in using the triple-axis spectrometer. We also thank Dr. O. Blaschko for many valuable discussions.

References