Proposal of Method to Make Pole Figure using Imaging Plate

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Introduction
State of texture in polycrystalline materials can be understood through pole figures measured by an X-ray diffraction technique. In this study, the method to measure the pole figure with imaging plate (IP) \cite{1-3} was proposed. Because two dimensional X-ray detector IP can measure the whole Debye-Scherrer ring, it can measure more wide-ranging pole density distribution than zero dimensional X-ray detectors such as a scintillation-counter. Moreover, because diffraction X-ray data recorded on IP are digital data, image analysis of them can be easily carried out by a computer. It is also thought that multiple pole figures can be obtained by one measurement when multiple Debye-Scherrer rings can be obtained by one exposure. Use of IP enables a more efficient pole figure measurement than a conventional method which uses Schulz goniometer and a scintillation-counter. The pole figure measurement device was made, and the pole figure of a rolled aluminum sheet having texture was measured. Finally, the measurement accuracy of the present method was proven by comparing the pole figures obtained by this method and the conventional method.

Pole figure measurement method using IP

The specimen was cut out from a rolled sheet as shown in Fig.1(a), and was put on a sample stand of the pole figure measurement device as shown in Fig.1(b). Incident X rays are diffracted at the diffraction angle 2θ, and cause the Debye-Scherrer ring on IP. A locus of a perpendicular line to crystal planes which causes this Debye-Scherrer ring is shown as a cone of vertical angle 2θ in Fig.1(b). The Debye-Scherrer ring is recorded on IP by a cylindrical X-ray camera of the pole figure measurement device. Debye-Scherrer rings can be more clearly recorded on IP than Laue camera. A shape of the recorded Debye-Scherrer ring becomes line. However, only a semicircle of the Debye-Scherrer ring is recorded on IP by the cylindrical X-ray camera which have a window of a central angle 180°. The specimen was designed so that incident X rays could enter the specimen surface always vertically. Diffraeted X rays are not interrupted with the sample by this specimen shape and can reach IP.

The locus of the perpendicular line of crystal planes can be also shown as a circle Ω on the pole figure as shown in Fig.1(c). We shall call this circle a locus circle in the following.
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(a) Shape and sampling position of the specimen.

(b) Direction of diffracted X-rays by the specimen and Debye-Scherrer ring recorded on IP.

(c) Normal direction of the diffraction plane (locus circle) on pole figure.

Fig. 1 Measurement method of pole figure using IP.

The bigger locus circle can be obtained when the diffraction angle $2\theta$ is close to 90° (the biggest at $2\theta = 90°$). The locus circle also moves to the position of circle 2 by an anti-clockwise $\beta - \beta_2$ rotation of the specimen. Therefore if we measure some Debye-Scherrer rings turning the specimen at regular intervals, the pole density on the pole figure can be obtained. The measurable area on the pole figure is $\phi - \eta \leq \alpha_R \leq \phi + \eta$ in radius direction and $0° \leq \beta \leq 360°$ in circumference direction. $\phi$ indicates an incidence angle of X rays to the plane of the rolled sheet.

**X-ray observation**

The specimen shown in Fig.1 was cut out from a rolled aluminum sheet of 1.5mm in thickness. The diameter of its maximum part was 10mm. Two Debye-Scherrer rings of Al(311) and Al(222) diffraction line were recorded on IP by using Co target. The Debye-Scherrer ring of Al(222) diffraction line was selected to measure the {111} pole figure of the rolled aluminum sheet. The diffraction angle $2\theta$ of Al(222) diffraction line was 99.9. The incidence angle $\phi$ of X rays to the plane of the rolled sheet was set to 50°. Therefore the measurable area of radius direction on this pole figure is $0° \leq \alpha_R \leq 90°$. Tube voltage, tube current and exposure time were 30kV, 10mA and 300sec, respectively. The pinhole diameter of the collimator was 1.0mm. A random sample which did not have texture was made from an aluminum powder for the comparison.
Results and discussions

Debye-Scherrer ring recorded on IP

Figure 2 shows an image of the linear Debye-Scherrer ring measured on IP by the pole figure measurement device. Figure 3 also shows a diffraction intensity distribution along the broken line (pixel line) across the Debye-Scherrer ring and the diffraction intensity distribution along the Debye-Scherrer in Fig.2. The background of the peak profile of

(a) Rolled aluminum sheet.

(b) Random sample.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Peak profile</th>
<th>Intensity distribution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rolled Aluminum Sheet</td>
<td><img src="image1" alt="Graph" /></td>
<td><img src="image2" alt="Graph" /></td>
</tr>
<tr>
<td>Aluminum Powder</td>
<td><img src="image3" alt="Graph" /></td>
<td><img src="image4" alt="Graph" /></td>
</tr>
</tbody>
</table>
Fig. 4. (111) pole figure of a rolled aluminum sheet measured by the present method.

Fig. 5. Data positions on (111) pole figure.

Al(222) diffraction line in Fig. 3 was removed by a function approximation method, and the peak position (pixel position) was decided by a full width of half maximum intensity (FWHM) middle point method. Next, the peak position intensity was calculated, and a similar operation was repeated for remaining pixel lines on IP image. Peak intensity values on 1024 pixel lines are shown in Fig. 3 as the diffraction intensity distribution along the Debye-Scherrer ring. The diffraction intensity distribution along the Debye-Scherrer ring measured from the rolled aluminum sheet noticeably depended on the orientation. This non-uniformity of the diffraction intensity distribution proves that this sample has texture. On the other hand, the diffraction intensity distribution along the Debye-Scherrer ring measured from a random sample did not depend on the orientation and was almost constant. The uniformity of the diffraction intensity distribution proves that optical accuracy of the present pole figure measurement device is excellent.

Pole figure obtained by the present method

Figure 4 shows the {111} pole figure of the rolled aluminum sheet obtained by the present method. The data positions in this pole figure, 1800 in number, are shown in Fig. 5. In this measurement, Debye-Scherrer rings were measured rotating the specimen at intervals of 3°. Each Debye-Scherrer ring was also divided at intervals of 3°, and the diffraction intensity of those points have been extracted from diffraction intensity data of these Debye-Scherrer rings to draw the pole figure. The diffraction intensity data
sample and converted into pole density data. Figure 6 shows the result of the \{111\} pole figure obtained by a Schulz reflection method. The measurable area on this \{111\} pole figure is $0^\circ \leq \alpha_R \leq 75^\circ$ in radius direction. In the present method, almost entire pole density of the pole figure can be measured by only the back-reflection method when the diffraction angle $2\theta$ of the measured Debye-Scherrer ring is close to $90^\circ$. In both these pole figures, \{111\} poles accumulated in almost same positions. However, because a lot of small peaks existed on the pole figure which had been obtained by the present method, accumulation positions were not clear. Because the diffraction intensity distribution along the Debye-Scherrer ring shown in Fig.3 was not a smooth curve, a lot of small peaks appeared on the pole figure.

Fig. 7. Effect of the smoothing processing on the diffraction intensity distribution (left) and the pole figure (right).

(a) Smoothing number $S_{num} = 21$.

(b) Smoothing number $S_{num} = 61$.  

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Effect of the smoothing processing of diffraction intensity distribution data along the Debye-Scherrer ring on pole figure

Figure 7 shows the smoothed curve of the diffraction intensity distribution and the redrawn pole figure by using smoothed curve data. The smoothing processing was executed by a moving average method [4], and its influence on the pole figure was examined by changing the smoothing number $S_{num}$. For example, in the smoothing processing of smoothing number $S_{num} = 21$, intensity of a certain point (pixel) becomes intensity averaged by 10 points before and behind that. Smoother curves and clearer pole figures were able to be obtained by the larger smoothing number. However, excessive smoothing processing caused an extreme decrease of maximum intensity and a disappearance of the characteristic of the pole figure. It was judged that the smoothing condition of smoothing number $S_{num} = 61$ was the best from a balance between clearness and characteristic disappearance of the pole figure.

<table>
<thead>
<tr>
<th>Rotation interval</th>
<th>Number of data</th>
<th>Data positions on pole figure</th>
<th>Pole figure of $S_{num} = 61$</th>
</tr>
</thead>
<tbody>
<tr>
<td>6°</td>
<td>900</td>
<td>RD</td>
<td>RD</td>
</tr>
<tr>
<td></td>
<td></td>
<td>TD</td>
<td>90°</td>
</tr>
<tr>
<td>9°</td>
<td>600</td>
<td>RD</td>
<td>RD</td>
</tr>
<tr>
<td></td>
<td></td>
<td>TD</td>
<td>90°</td>
</tr>
<tr>
<td>12°</td>
<td>450</td>
<td>RD</td>
<td>RD</td>
</tr>
<tr>
<td></td>
<td></td>
<td>TD</td>
<td>90°</td>
</tr>
</tbody>
</table>

Fig. 8. Change in pole figure for each rotation intervals.
Effect of rotation intervals of the specimen on the pole figure

In the preceding section, pole figures which have been measured by rotating the specimen at intervals of $3^\circ$ were illustrated. In this section, the influence of rotation intervals on the pole figure was examined. Figure 8 shows $\{111\}$ pole figures obtained at rotation intervals of $6^\circ$, $9^\circ$ and $12^\circ$. The smoothing processing of $S_{\text{num}}=61$ was given to diffraction intensity distribution data of these pole figures. Positions and the number of data on each pole figure are also shown in Fig.8. As rotation intervals broaden, the number of data on the pole figure decreases. However, the movement of the peak position was not seen on these pole figures. In the case of $12^\circ$ intervals, if 30 Debye-Scherrer rings are measured, the pole density distribution of all quadrants on the pole figure can be understood. However, we judged that rotation intervals of $9^\circ$ was the most suitable for the pole figure measurement because the result of $9^\circ$ intervals is the most similar to the result of Schulz reflection method of Fig.6.

Conclusions

In this study, the pole figure measurement method with the imaging plate was proposed. The measurement device was made, and the $\{111\}$ pole figure of a rolled aluminum sheet which has texture was measured using the present method. The following results were obtained by comparing the pole figures which had been obtained by this method and a conventional method.

1. The $\{111\}$ pole figure which had been obtained by this method was similar to the $\{111\}$ pole figure which had been obtained by the Schulz reflection method. From this result, the measurement accuracy of the present method was proven.

2. We can easily understand the accumulation state of the pole on the pole figure by smoothing the diffraction intensity distribution data of the Debye-Scherrer ring.

3. If 30 Debye-Scherrer rings are measured, the pole density distribution of all quadrants on the pole figure can be obtained.

4. In the present method, wide-ranging pole density distribution can be measured sufficiently by the back-reflection method (Measurement by the forward-reflection method is unnecessary).

References