Development of technique for three-dimensional visualization of grain boundaries by white X-ray microbeam

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Abstract. A technique for three-dimensional visualization of grain boundaries was developed at BL28B2 at SPring-8. The technique uses white X-ray microbeam diffraction and a rotating slit. Three-dimensional images of small silicon single crystals filled in a plastic tube were successfully obtained using this technique for demonstration purposes. The images were consistent with those obtained by X-ray computed tomography.

1. Introduction

The three-dimensional (3D) distribution of grains in structural materials strongly influences their mechanical properties. A technique for nondestructive observation of the 3D distribution of grains is necessary for clarifying the correlation between the mechanical properties and the grain distribution. The visualization of grains in polycrystalline material is difficult for Computed tomography (CT), which is a typical 3D observation technique because absorption contrast is hardly detected at grain boundaries. X-ray diffraction technique is an effective technique for observation of grain distribution detecting the deference of crystalline orientation.

A technique of grain boundary imaging by the Laue pattern (GILP) was developed at BL28B2 at SPring-8 to visualize the distribution of grain boundaries in polycrystalline materials [1]. The GILP technique uses X-ray diffraction by the transmission Laue method using white X-ray microbeams. Images of the Laue pattern were acquired using a two-dimensional (2D) detector by scanning the beam positions on the specimen. The grain boundaries were identified by using the differentials between the Laue pattern images at each position. However, this technique was not applicable to 3D observations because GILP cannot discriminate between Laue patterns from grains on a beam path in a specimen. This problem can be solved by using a rotating slit (figure 1), which was developed to restrict the observation area inside a bulky specimen in X-ray diffraction measurement using a 2D detector [2].

The purpose of this study was to apply GILP technique to 3D observation by using the rotating slit.

2. Experimental

The experiment was conducted at BL28B2 at SPring-8. The experimental setup is shown in figure 2. The white X-rays generated by a bending magnet light source were shaped in a $20 \times 20 \mu m^2$ cross section by the quadrant slit. The white X-ray microbeam was irradiated on the specimen in the transmission layout. Laue patterns from the specimen were detected by a 2D detector. A rotating slit unit was placed between the specimen and the 2D detector. This device consists of a pair of tantalum disks, as shown in figure 1. The disks were 0.5 mm thick and had diameters of 100 mm and 200 mm, respectively. Slits
with the shape of an Archimedean spiral were cut into the disks. The slits were 0.2 mm wide. These slits specified the observation areas in the specimen. The disks with the slits were rotated at a rate of 120 rpm so that the aperture of the slits could cover the detection area of the 2D detector. This rotating slit made it possible for the 2D detector to discriminate the Laue patterns from the grains on the beam path. The exposure time of a Laue pattern image was 0.5 s. The position of the specimen was scanned by motorized translation stages at a step interval of 0.01 mm.

Images obtained by the new technique were compared with ones obtained by CT, for verification of the new technique. A mass of small silicon single crystals filled in a plastic tube was prepared as a sample for demonstration (figure 3) because actual distribution of grains in structural materials cannot be visualized by CT. The distribution of silicon crystals in a plastic tube was able to be visualized by both techniques. The silicon crystals were a few millimeters in diameter, and the inner diameter of the plastic tube was 7 mm. A beam path typically hit one to five silicon crystals.

Figure 1. Schematic of a rotating slit.

Figure 2. Schematic of the experimental setup.

Figure 3. Photograph of the sample and silicon single crystals.

3. Results and discussion
Figure 4 shows cross-sectional images of the grain boundaries measured by GILP using the rotating slit. Figure 4 (a) and (b) are images of the cross sections in the $xz$ plane and $xy$ plane, respectively. The black
contrast indicates the grain boundaries. Figure 5 shows the edge-enhanced CT images corresponding to figure 4. The images in figure 4 consistently reproduce the morphological features shown in figure 5.

**Figure 4.** Reconstructed grain boundary images obtained from Laue patterns in the range of $\Theta = 17–26^\circ$. Cross sections in $xz$ plane (a) and $xy$ plane (b).

![Reconstructed grain boundary images](image)

**Figure 5.** CT images. Cross section in $xz$ plane (a) and $xy$ plane (b). The images were processed by an edge enhancement filter. The X-ray energy was 30 keV. The interval of the rotation step was 0.5°. The exposure time of an image was 0.05 s.

The relationship between the diffraction angle and the spatial resolution of GILP with the rotating slit was examined. Figure 6 shows a schematic of the observation area restricted by the rotating slit. The spatial resolution of GILP with the rotating slit was expected to depend on the size of the observation area. The size of the area in the $y$ direction, $A_y$, should increase as the diffraction angle decreases. Grain boundary images obtained from the Laue patterns in the area of low diffraction angle are shown in figure 7. The diffraction angles in figures 4 and 7 were 17–26° and 8–17°, respectively. The spatial resolution in the $y$ direction in figure 7 (b) was lower than that in figure 4 (b), in agreement with the prediction. On the other hand, the difference between figures 4 (a) and 7 (a) was small. This is because the size of the observation area in the $x$ and $z$ directions is not affected by the diffraction angle.
Figure 6. Schematic of the observation area restricted by the rotating slit.

Figure 7. Reconstructed grain boundary images obtained from Laue patterns in the range of $\Theta = 8$–$17^\circ$. Cross sections in $xz$ plane (a) and $xy$ plane (b).

4. Conclusion
A technique for 3D observation of grain boundaries was developed by applying a rotating slit to the GILP technique. The spatial resolution of this technique depended on the diffraction angle of the Laue patterns; patterns in the area of higher diffraction angles provide a higher spatial resolution.

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References
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