

Crystal orientation measurements of samples containing coarse grains

Introduction

Pole figure measurement is an important technique for determining the crystal orientation of a sample or calculating the volume fraction of the orientation. Especially for the latter, it is crucial that the measured intensities are equal to the true intensities. Some reduction in intensity due to defocus can be compensated for using defocus corrections, but for samples containing coarse grains, the further development of measurement techniques is necessary.

Measurements and results

To achieve accurate diffraction intensities, it is essential that the grain size of crystals in a sample is less than 10 μm . For bigger grains, diffraction intensities vary due to shifts of the measurement point or the X-ray incident angle to the sample. Since the pulverization of samples is not possible for pole figure measurements, it is necessary to perform measurements while oscillating the sample. Performing pole figure measurements while oscillating the sample in a direction (γ axis) different from the tilt axis (α axis) or rotation axis (β axis) as shown in Fig. 1 makes it possible to perform crystal orientation measurements of samples containing coarse grains.

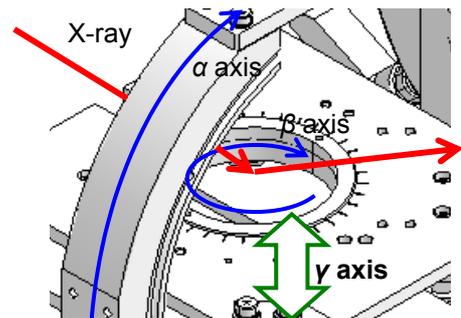


Fig. 1: Movement of each attachment axis

Fig. 2 shows the rocking curve measurement results of a sample with an average grain size of about 45 μm (red: with oscillation of the γ axis; blue: without oscillation of the γ axis). Without oscillation, sharp peaks attributed to coarse grains were observed. However, by oscillating the γ axis during measurement, it was possible to suppress the influence of these coarse crystals.

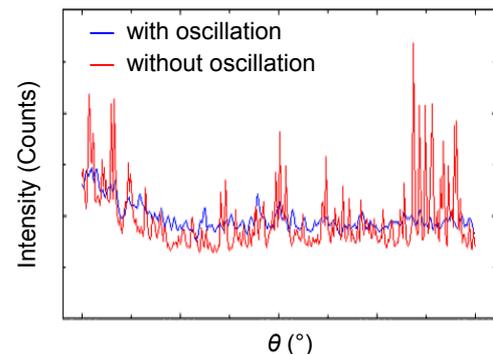


Fig. 2: Rocking curve measurement results

The pole figures shown in Fig. 3 – one achieved by oscillating the γ axis during measurement, the other calculated – are almost equal. This shows that even for samples containing coarse grains, pole figures suited for analysis can be achieved by oscillating the γ axis during measurement.

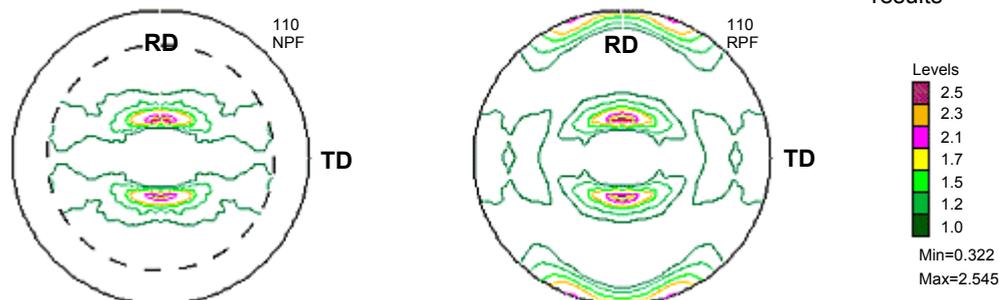


Fig. 3: Pole figure measured while oscillating the γ axis (left) and calculated pole figure (right)

Measurement data provided by Prof. Osamu Umezawa, Yokohama National University, Graduate School

Rocking curve measurements are performed by changing the incident angle θ while keeping a fixed diffraction angle 2θ towards the sample. For coarse grains, intensity varies depending on the incident angle, making it possible to determine immediately whether a sample contains coarse or fine grains.

Recommended equipment and software

- ▶ Multipurpose X-ray Diffractometer Ultima IV + Multipurpose Measurement Attachment ML4
- ▶ SmartLab Automated Multipurpose X-ray Diffractometer + $\alpha\beta$ Attachment