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Particle Statistics in Synchrotron Powder Diffractometry

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Introduction

We have recently developed a new analytical method to evaluate the statistical variance of measured diffraction intensity caused by the finite number of crystallites in crystalline powder specimens [1]. It has been found that the effective crystallite diameters over 5 μm can be evaluated by statistical analysis of diffraction intensity data collected by stepwise rotation of a flat powder specimen with a laboratory Bragg-Brentano powder diffractometer, while the measurable size achieved by conventional line broadening analysis is limited within the range from 5 nm to 200 nm. However, it is also suggested that measurement of the intermediate crystallite size ranging from 200 nm to 3 μm is practically impossible by using a laboratory diffractometer.

Use of synchrotron x-ray may expand the sensitivity of the crystallite size evaluation by the spinner scan measurement to smaller size, because smaller focal size is expected for synchrotron x-ray source. In this study, we have examined the effect of particle statistics on diffraction intensity data measured with the high-resolution powder diffractometer on the beamline BL-4B2 at KEK-PF.

Experimental

Step-scan diffraction intensity data about the rotation angle of a Si powder specimen (NIST SRM640c) were collected. The effective diameter of the Si powder evaluated by SEM image analysis was 5.6 μm.

Symmetric reflection mode was applied for the diffraction measurement. The cross section of the incident beam was limited with entrance slits of 1 mm in height and 10 mm in width. The calibrated peak wavelength of the x-ray was $\lambda = 0.1208568(4)$ nm.

Results and Discussions

The effective number of diffracting crystallites, n_{eff}, is calculated by $n_{\mathrm{eff}} = I^2 / (\Delta I_{\mathrm{particle}})^2$, where I is the mean intensity and $(\Delta I_{\mathrm{particle}})^2$ is the variance caused by particle statistics, which is evaluated by subtraction of variance caused by counting statistics from observed variance [2].

The theory proposed by Alexander et al. [2] suggests that n_{eff} is proportional to the effective multiplicity of reflection m_{eff} and also the cosecant of the Bragg angle θ, when the crystallites are randomly oriented. Figure 1 shows the values of $n_{\mathrm{eff}} \sin\theta$ evaluated by the analysis of the spinner scan data measured on BL-4B2 and the known multiplicity m_{eff} of Si reflections. It has been found that the behavior of the observed values of $n_{\mathrm{eff}} \sin\theta$ and m_{eff} are well corresponded, but systematic deviation depending on the diffraction angle 2θ is also detected.

A similar plot for the data collected with a usual laboratory diffractometer (Rigaku RAD-2C) is shown in Fig. 2. The effective number of crystallites in powder diffraction measurement on BL-4B2 is roughly 1/20 of that measured with the laboratory diffractometer. It means that the effect of particle statistics in powder diffraction measurement on BL-4B2 will be more pronounced, unless continuous rotation of specimen is applied. Since n_{eff} is inversely proportional to the particle volume, it is also suggested that the measurable crystallite size in diameter will become 1/3 by applying the BL-4B2 diffractometer, as compared with a laboratory powder diffractometer.

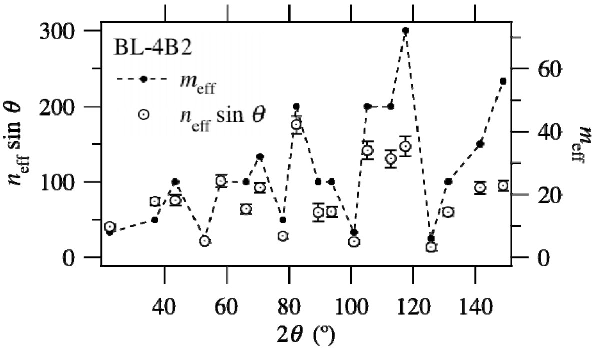

Fig. 1 Product of the effective number n_{eff} by $\sin\theta$ (open circles) and the known multiplicity of reflection m_{eff} for Si (dots connected with broken lines).

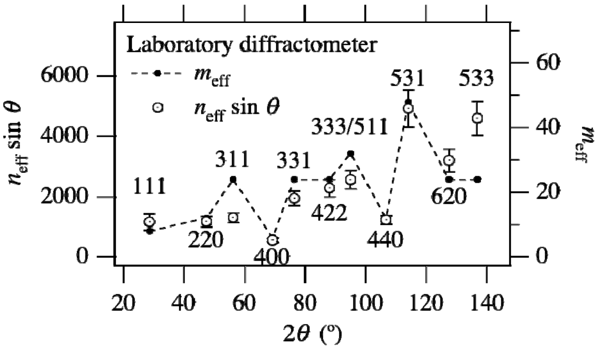

Fig. 2 Plot similar to Fig. 1, drawn with the data collected with a laboratory diffractometer.

References

[1] T. Ida et al., J. Appl. Cryst. (in press).
[2] L. Alexander et al., J. Appl. Phys. 19, 742 (1948).

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