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Particle statistics in synchrotron powder diffractometry

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Introduction

Recently, we have reported the results of experimental investigation about particle statistics in diffraction intensities measured by a powder method, applying a newly developed spinner-scan measurements with a laboratory powder X-ray diffractometer [1]. It is expected that the effect of particle statistics will be more pronounced in synchrotron powder diffractometry, because the deviation of the orientation of a crystallite from the normal direction should be more strongly restricted by the small effective size of the synchrotron X-ray source to satisfy the diffraction condition. In this study, statistical properties of the observed diffraction intensities obtained by an ω-scan method at a synchrotron powder diffraction beam-line are investigated.

Experimental

A cylindrical sample holder was filled with standard Si powder (NIST SRM 640c), the effective crystallite diameter of which has been estimated at 5.6 μm by SEM image analysis [1]. The bulk penetration depth for the synchrotron X-ray at the wavelength of 0.1197 nm is estimated at $\mu^{-1} = 0.147$ mm.

A high-resolution powder diffractometer at the beam-line BL-4B2 at KEK-PF was used for the diffraction measurements in flat-specimen reflection mode. The ω - scan intensity profiles of 10 Si reflections were recorded on stepwise rotation of the powder specimen about the ω–axis of the diffractometer. A couple of ω-scan profiles measured for a stationary and (in-plane) rotating specimens were compared for each reflection.

Results

Figures 1 (a) and (b) show the ω–scan diffraction intensity profile of the Si 111-reflection measured for the stationary and rotating specimens, respectively. The observed dependence on ω is fitted by a model profile:

$$I(\omega) = 2\, I_0 \,\mathrm{cosec}\, \omega / [\mathrm{cosec}\, \omega + \mathrm{cosec}(2\,\theta - \omega)] .$$

No significant systematic deviations have been detected in the difference plots.

The variance observed for the stationary specimen is by far beyond the values predicted from counting statistics, which clearly indicates that the in-plane rotation of flat specimens is important to evaluate accurate diffraction intensities in synchrotron powder diffractometry.

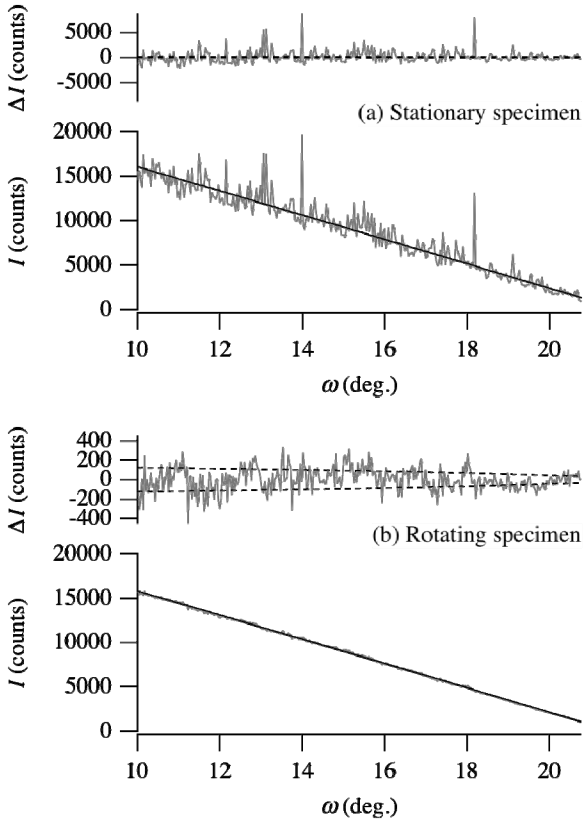

Fig. 1 ω-scan intensity profile of Si 111-reflection measured for (a) stationary and (b) rotating specimens. Observed and fitted profiles are shown as gray and black lines in the lower panels. The broken lines in the upper panels show the standard deviation predicted from counting statistics.

Even though the variance observed for the 111-reflection from the rotating specimen is almost comparable with the counting error, difference from counting variance can be detected by a detailed statistical analysis. It has been found that the effective number of diffracting crystallites estimated from the ω–scan profiles measured for the 10 reflections are strongly correlated to the multiplicity of reflections. It is concluded that the effect of particle statistics on the diffraction intensities from a rotating powder specimen can quantitatively be evaluated by this method.

References

[1] T. Ida et al., J. Appl. Cryst. 42, 597 (2009).

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