4B2/2011G015

Particle statistics in rotating-specimen powder diffractometry

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Introduction

Powder X-ray diffractometry has been utilized in identification, qualitative and quantitative analyses of minerals and synthesized materials, including natural resources, ceramics, metal, alloys, and pharmaceutical products. Discussions about the accuracy and reliability of powder diffraction analysis are practically getting more important, but theoretical difficulty about particle statistics, particularly for continuously rotating specimens, still remains. De Wolff has already suggested a theory about particle statistics for rotating specimens [1]. The effective numbers of diffracting crystallites for stationary and rotating specimens in reflection mode measurements have been expected to be proportional to cosecθ and cosec$^2\theta$, respectively, for the diffraction at the Bragg angle θ. However, it is difficult to justify the assumptions made by de Wolff, and the theory has not been justified by any experiments. The angular dependence of the particle statistics has been experimentally investigated in this study.

Experimental

A cylindrical sample holder was filled with standard LaB_6 powder (NIST SRM 660a), the volumetric median particle diameter of which has been estimated at 10 µm by a laser scattering analysis. The bulk penetration depth for the synchrotron X-ray at the wavelength of 0.1197 nm is estimated at μ^{-1} = 18 µm.

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 23 LaB_6 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 dependence of the observed effective number of diffracting crystallites (N_{eff}) evaluated for the symmetric condition ($\Omega = \theta$), divided by the effective multiplicity of reflection (m_{eff}) evaluated by the analysis of Ω–scan diffraction intensity profile of the LaB_6 reflections measured for the stationary and rotating specimens, respectively. The observed dependences on 2θ are both fitted by a model profile:

$$N_{eff} / m_{eff} = (N_{eff} / m_{eff})_0 \operatorname{cosec} \theta.$$

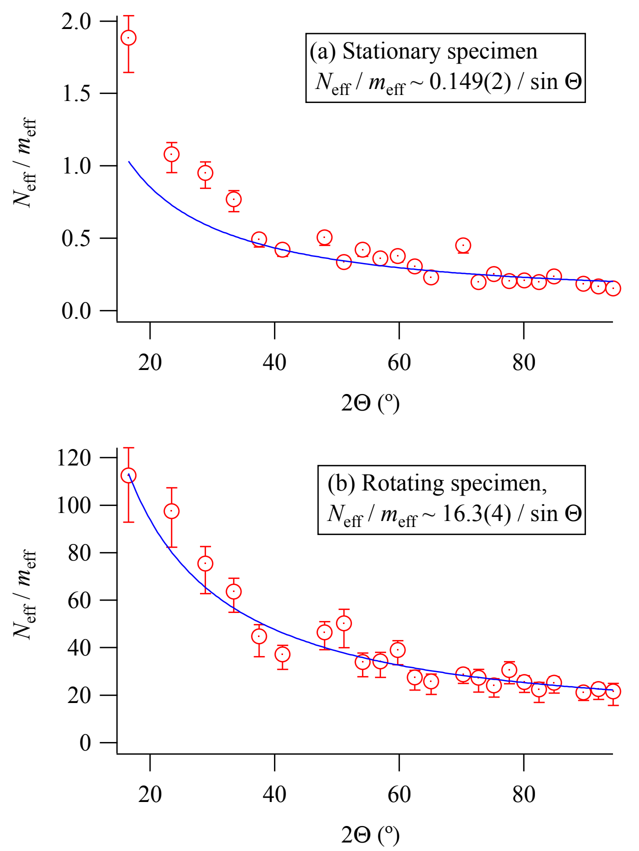

Fig. 1 2Θ-dependence of the effective number of diffracting crystallites divided by the effective number of multiplicity (N_{eff} / m_{eff}) evaluated by Ω–scan diffraction intensity measurements for (a) stationary and (b) rotating specimens of LaB_6 powder.

It has been confirmed that the effective number of diffracting crystallites appears to be increased by rotation by a factor of 100 over the observed angular range.

However, the experimental 2Θ-dependence of N_{eff} / m_{eff} for the rotating specimen (Fig. 1(b)) appears to be almost proportional to cosecΘ, rather than cosec^{2}Θ, contrary to the prediction from the theory of de Wolff.

Even though the experimental results do not necessarily mean that the assumption of de Wolff should thoroughly be rejected, it is concluded that we can practically apply the cosecΘ-dependence for maximum-likelihood analysis of powder diffraction data for rotating specimens.

References

[1] P.M. de Wolff, Appl.Sci.Res. B 7, 102 (1958).
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