Crystallography

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Deconvolution of synchrotron powder diffraction data

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Introduction

It has been strongly desired to establish the accurate formula of the instrumental function for the synchrotron powder diffractometry. Even though the instrumental function for the high-resolution powder diffractometer used for synchrotron measurements has already been analyzed in detail [1], the effects of aberrations caused by the synchrotron beamline optics is not negligible in precise diffraction peak profile analysis for the purpose of extraction of microstructural informations.

In this study, we try to evaluate the effects of the beamline aberrations in an empirical way, where the diffractometer aberrations are removed from the experimental data by a deconvolution method based on an analytical model originally developed by the authors.

Method

Experimental procedure

The diffraction data of standard LaB_6 powder [NIST SRM660] were collected with a high-resolution synchrotron powder diffractometer, MDS [3] on beamline BL-4B$_2$. The LaB_6 powder was loaded into a flat sample holder, which was rotated at 1 revolution s^{-1} about the normal to the sample face during the measurements. The sample face was inclined by 4.885° to the incident beam. A Ge(111) crystal analyzer adjusted at the Bragg angle 6.2° for the wavelength 0.707Å was attached on the diffracted beam side. The incident X-ray beam was restricted to 2.5 mm in width and 1 mm in height with a couple of incident slits. The diffraction pattern was scanned over the angular range 9.0-37.8° (2θ), with a step length of 0.004° (2θ) and a counting time of 4s step^{-1}.

Deconvolution

A whole-powder-pattern deconvolution method, which is based on fast Fourier transformation (FFT) combined with an appropriate scale transformation of the data [2], was applied to the experimental data, in order to remove the effects of the axial divergence aberration. The number of sampling points used for the FFT calculation was 32768.

The propagation of the statistical errors in the experimental data was also calculated by the FFT method. The details of the deconvolution method have been described elsewhere [4].

Profile fitting

Each reflection peak profile in the deconvoluted data was analyzed by an individual profile fitting method, using a model profile function synthesized by the convolution of the extended pseudo-Voigt function with a truncated exponential function to reproduce asymmetry in the deconvoluted profile.

Results

The results of the deconvolution and profile fitting analysis for the 100-reflection peak is shown in Fig. 1.

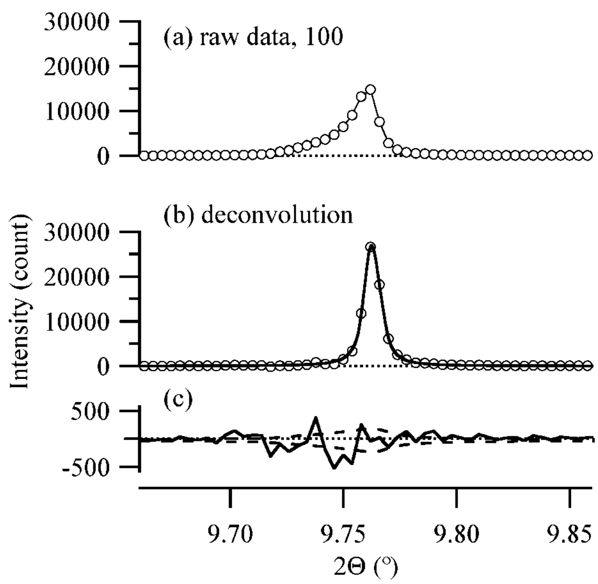

Fig. 1: (a) Raw data of LaB_6 100-reflection peak; (b) deconvoluted data (circles) and fitted curve (line); (c) difference plot (solid line) and estimated errors (broken line) for the fitting in (b).

As shown in Fig. 1, the significant asymmetry in the raw experimental profile has successfully been removed by the deconvolution method.

Slight asymmetry having longer tail on the higher angle side of the peak is found in the deconvoluted data, which is naturally ascribable to the aberrations of the beamline optics. Since the asymmetry is well reproduced by the convolution with an asymmetric model profile function, it is expected that the effect can also be removed by a deconvolution method with the empirical parameter for the asymmetry evaluated by the profile fitting method.

References

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