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Symmetrisation of synchrotron X-ray powder diffraction peak profiles

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Introduction

We have previously reported the mathematical expressions of the axial divergence aberration for the powder X-ray diffractometer on the beam-line BL-4B$_2$ at the Photon Factory [1]. We have also suggested that instrumental peak shift, line broadening and deformation in peak shape can be removed by our originally developed deconvolution procedure applying non-linear abscissa mapping method [2]. However, there were some difficulties in application of the method to the data measured with the multiple-detector diffractometer on BL-4B$_2$, because (i) profile of strong peaks are affected by losses in counting devices, (ii) instrumental functions may be varied between different detectors, and (iii) the axial divergence aberration function has a singularity at a certain diffraction angle. Recently, we have developed an advanced method for correction of counting losses [3] and also a Fourier connection method automatically adjusting difference between segmented intensity data supplied by the multiple-detector systems [4]. In this study, we have developed a practical algorithm to avoid numerical instability at the singularity point, and automatically restore the observed profiles to be symmetric. The method is applied to the powder diffraction intensity data from standard ZnO powder (NIST SRM674).

Method

Treatment of singularity in instrumental function

Axial divergence aberration function for high-resolution powder diffractometer with the analyser Bragg angle of Θ_A has singularity at the diffraction angle of $2\theta = 2\Theta_A+\pi/2$, where the function coincides with the Dirac delta function [1]. The centre 2θ region near the singularity where the width of the aberration function is smaller than one-tenth of the sampling interval (0.005º) was separated and kept unchanged. The rest of data on both sides of the centre region were treated by the deconvolution, and merged to the center data. The segmentation, deconvolution and combination process could be completed automatically with a simple algorithm.

Experimental and analysis

ZnO powder was filled into a 0.5 mmϕ glass capillary and the diffraction intensity data were collected by a multiple-arm measurement at the wavelength of 0.13 nm. The diffraction data were treated by a whole-pattern deconvolution method and it has been confirmed that all the diffraction peak profiles have become symmetric. The integrated intensities of the symmetrised peak profiles were extracted by a simple individual profile fitting method applying symmetric peak profile functions. The crystal structure of ZnO was refined by a conventional least-squares method for integrated peak intensities. The Rietveld method or whole-pattern fitting methods were not used for the structure refinement.

Results

Structure refinement

The refined structure parameters are listed in table 1. The structure parameters determined by the current method are in good agreement within experimental errors with the data refined from single-crystal diffraction data.

Table 1: Refined structure parameters

	This Work	Abrahams (1969) [5]	Albertson (1989) [6]
Specimen/ beam source	powder/ synchrotron X-ray	single crystal/ laboratory X-ray	single crystal/ neutron
No. of reflections	42	141	394
a (Å)	3.24987(18)	3.24990(4)	
c (Å)	5.20650(3)	5.220660(6)	
z(O)	0. 3827(8)	0.3825(14)	0.3819(1)
U(Zn)		0.0080(3)	
U(O)		0. 0086(9)	
U_{11}(Zn)	0. 0079(2)		0. 0073(4)
U_{33}(Zn)	0. 0080(4)		0.0094(4)
U_{11}(O)	0.0065(11)		0.0056(4)
U_{33}(O)	0.0095(21)		0.0064(4)
R(%)	0.85	3.78	2.31

References

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