BL-4B2/2011G015

Evaluation of absorption factor for capillary specimens in powder diffractometry

Takashi Ida*
ACRC, Nagoya Institute of Technology, Tajimi 507-0071, Japan

1 Introduction

Transmission-mode measurement with a capillary specimen is widely used in synchrotron powder diffractometry. It is often more advantageous than reflection-mode measurement with a flat-plate specimen, because of the smaller amount of sample needed, geometrical equivalence at different diffraction angles, reduced effect of preferred orientation and more improved statistics by spinning the sample on measurement [1]. However, absorption correction should be applied on analysis of data measured in transmission-mode, while no correction is needed for reflection-mode. The effective transmittance for the beam diffracted by a cylindrical specimen depends on the diffraction angle and the parameter μR, where μ is the linear absorption coefficient and R is the radius of the cylinder. A practical method to evaluate μR is proposed in this report.

2 Experiment

The cross section of the incident beam at KEK-PF BL-4B2 beamline, monochromated at 0.1196 nm and attenuated with a 50 μm Cu foil, was restricted by a couple of entrance slits of 5 mm in width and 0.05 mm in height. The X-ray beam transmitted through a vacant or sample (mixuture of LiF and quartz) containing borosilicate glass capillary was directly detected with a scintillation counter, while the crystal analyzer, inserted on the diffracted beam path on ordinary diffraction measurements, was removed to avoid reduction of intensity caused by small-angle scattering in the capillary glass and crystalline powder. The intensity of the transmitted beam on scanning the vertical position of the goniometer were recorded at each 0.02 mm step.

3 Analysis

The smearing of the intensity profile of the transmitted X-ray caused by the finite height of the incident beam is modelled by a convolution of the transmission profile with a rectangular window function. The convolution is calculated by numerical integration, where 100-point mid-point method is applied. At the first stage, the thickness of the wall of a capillary glass is evaluated from the data measured for a vacant capillary, applying the values of the linear absorption coefficient of the glass 31.8 cm^{-1} calculated from the mass linear coefficient and the chemical composition and density of the glass as known parameters. The inner diameter $2R$ of the glass capillary and linear coefficient of the sample μ are evaluated by the curve fitting method to the intensity profile measured for the capillary containing sample powder.

3 Results and Discussion

Figure 1 shows the observed (red) and simulated (blue) intensity profiles of (a) vacant and (b) sample-containing capillaries, respectively. The thickness of the glass wall and inner diameter of the vacant capillary are estimated at 0.018(1) mm and 0.365(1) mm, while the nominal values are 0.01 mm and 0.48 mm, respectively. The estimated thickness of the glass wall is close to the value 0.017 mm measured with a scale attached to an eyepiece of a binocular microscope. The linear absorption coefficient μ and diameter $2R$ of the specimen are estimated at 10.8(1) cm^{-1} and 0.456(2) mm, while the value of μ calculated from the weight measurement is 11.4 cm^{-1}.

It is recommended that the value of the parameter for absorption correction μR on analysis of capillary-specimen powder diffraction intensity data should be experimentally estimated by the method proposed in this report.

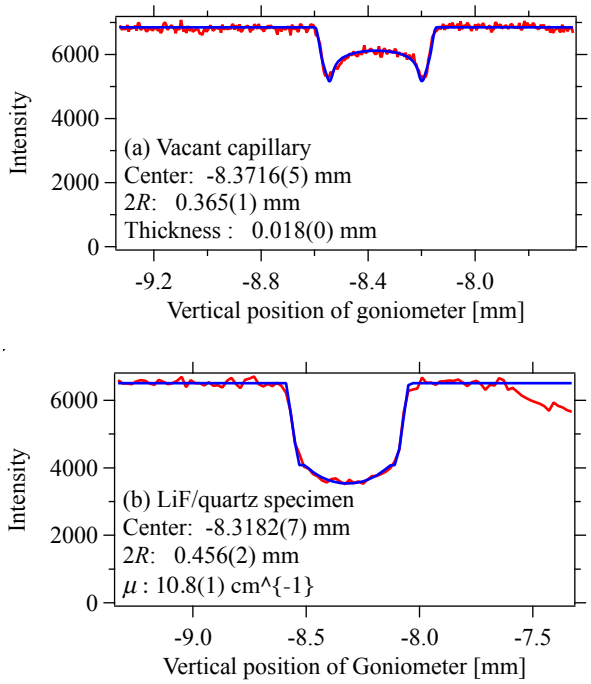

Fig. 1: Vertical-scan intensity profiles.

References

[1] T. Ida, *J. Appl. Cryst.* **44**, 911 (2011).

* ida.takashi@nitech.ac.jp