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Peak profile in synchrotron X-ray powder diffractometry

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Introduction

Application of synchrotron X-ray radiation with negligible beam divergence to powder diffractometry supplies not only highly resolved experimental peaks for well crystallized samples, but also a definite way in theoretical simulation of the peak profiles.

In this report, we propose a practical model peak profile function based on the convolution with the exact formulae of the instrumental function for a high-resolution synchrotron X-ray diffractometer, equipped with a flat crystal analyser and a set of Soller slits for limiting the axial divergence of the diffracted beam. The effect of a slight tilt angle of the analyser crystal, which may be caused by insufficient precision in crystallographic orientation of the analyser, is also taken into account in the current model.

Model profile function

Instrumental function

The general formulae of the instrumental function for the diffracted-beam axial divergence are expressed by the following equations:

$$w_{\mathrm{H}}(x) = \int_{-1}^{1} \delta(x - Au^2 - Bu - C)(1 - |u|)du \tag{1}$$

$$A \equiv -\frac{\Phi_{\mathrm{H}}^2}{2}\left(\cot 2\theta + \tan\Theta_{\mathrm{A}}\right) \tag{2}$$

$$B \equiv \Phi_{\mathrm{H}}\Phi_{\mathrm{A}} \sec\Theta_{\mathrm{A}} \tag{3}$$

$$C \equiv -\frac{\Phi_{\mathrm{A}}^2}{2}\tan\Theta_{\mathrm{A}} \tag{4}$$

where $\delta(x)$ is the delta function, 2θ the diffraction angle, Θ_{A} the analyser Bragg angle, Φ_{H} the axial divergence angle, and Φ_{A} the tilt angle of the analyser defined as the deviation of the crystal normal direction out of the goniometer plane. More concrete formulae applicable to numerical calculation are given in ref. [1].

Peak profile function

The peak profile function $p(x)$ is given by the convolution of a Lorentzian function with the full width at half maximum of $2\gamma_{\mathrm{L}}$:

$$f_{\mathrm{L}}(x) = \frac{1}{\pi\gamma_{\mathrm{L}}}\left[1 + \left(\frac{x}{\gamma_{\mathrm{L}}}\right)^2\right]^{-1} \tag{5}$$

with the above instrumental function $w_{\mathrm{H}}(x)$, that is,

$$p(x) = \int_{a}^{b} f_{\mathrm{L}}(x - y) w_{\mathrm{H}}(y)dy \tag{6}$$

where a and b are respectively the lower and upper limits of the variable having non-zero values of $w_{\mathrm{H}}(x)$.

Analysis of experimental profiles

Figure 1 shows experimental diffraction peak profiles of Si 111, 220 and 311-reflections for 0.0707 nm X-ray measured with a powder diffractometer MDS [2] on the beamline BL4B2 at the Photon Factory in Tsukuba, together with the calculated profiles based on eq.(6) fitted only by varying the position, intensity, Lorentzian width γ_{L}, and the analyzer tilt angle Φ_{A}. The mesurement was conducted after the apparent tilt angle of the Ge(111) crystal analyser had carefully been adjusted to be 0.0(1) deg. The difference between the experimental and calculated profiles plotted in the lower panel of Fig. 1 lies almost within statistical uncertanties.

Furthermore, it has been found that heavily distorted profiles arising from misalignment of the analyser crystal are also well reproduced only by varying the parameter for the tilt angle Φ_{A} [1].

As the instrumental parameters in the model peak profile function can be naturally treated as specific constants for a given diffractometer, the current model will provide a straightforward way to extract intrinsic structural information from experimental diffraction data by applying a profile-fitting method or a deconvolution technique.

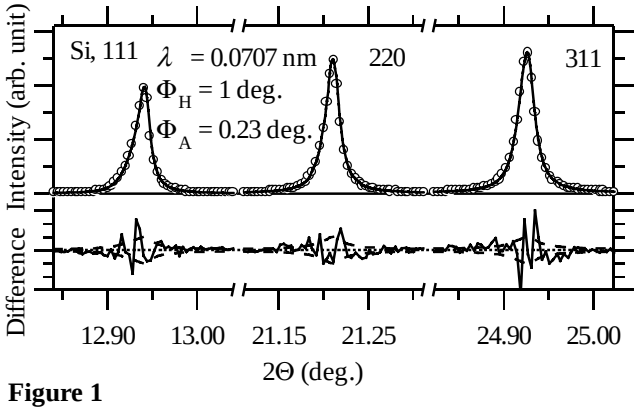

Figure 1
Profile fitting (solid line) to synchrotron diffraction data (open circles) from Si (NIST SRM640b) collected with 0.0707 nm X-ray and a Ge(111) analyser. The lower part shows the difference plot (solid line) with the standard uncertainties (dashed line).

References

[1] T. Ida et al., J. Appl. Cryst. 34, 144 (2001).
[2] H. Toraya et al., J. Synchroton Rad. 3, 75 (1996).