Phase Determination of Crystal Structure Factor by Measuring Rocking Curves from Polar Crystal

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We reported a phase determination of the crystal structure factor by the phase difference between the diffracted and transmitted rocking curves in Laue case, previously [1]. We reported a phase determination of the crystal structure factor by the phase difference between the diffracted and transmitted rocking curves in Laue case, previously [1]. In this paper, we propose a novel phase determination approach by using FWHM (full width at half maximum) and intensities of measured rocking curves from the front and bottom surfaces of a polar crystal. We assume that only one atom in a unit cell suffers anomalous scattering and its position is known. Then the crystal structure factor is given by

\[ F_h = |F_{hr}| \exp(i\alpha_{hr}) + i|F_{hi}| \exp(i\alpha_{hi}) \tag{1} \]

The first term is the real part to be determined, and the second one the imaginary part assumed to be known. If FWHM’s of rocking curves from the front and bottom surfaces in Bragg case are written as \( \Delta\Theta \) and the refractive index is equal to one, the crystal structure factor is given by

\[ |F|=\sqrt{(|F_{hr}|^2 + |F_{hi}|^2)} = \frac{\omega^2 V \sin 2\theta_B}{8\pi} \Delta\Theta \tag{2} \]

Here \( \omega \) is the X-ray energy, \( V \) the volume of a unit cell and \( \theta_B \) the Bragg angle. The crystal structure factor is also expressed as,

\[ F_{\pm h} = |F_{hr}|(1 \mp 2k |\sin \delta|)^{1/2} \exp[i(\pm \alpha_{hr} + \Theta)], \tag{3} \]

with \( k = |F_{hi}|/|F_{hr}| \) being assumed to be much less than one. In eq. (3),

\[ \alpha_{hr} = \alpha_{hi} + \delta \tag{4} \quad \text{and} \quad \Theta = \tan^{-1}[k \cos \delta/(1 \mp k |\sin \delta|)]. \tag{5} \]

If the intensity ratio \( r \) from one side of the surface to the other side is written as

\[ r = |F_{-h}|/|F_{+h}| \tag{6} \]

then the phase \( \delta \) is given by

\[ \sin \delta = \frac{1}{2k} \cdot \frac{r-1}{r+1} \tag{7} \]

The phase \( \delta \) can be determined by measuring intensities from the top and bottom surfaces. If \( \Delta\Theta \) and \( r \) are given by measuring rocking curves, \( \alpha_{hr} \) is to be determined.

We measured \( \Delta\Theta \) and \( r \) from a GaAs crystal by using X-rays from synchrotron radiation at KEK-PF, Japan. We tuned the energy at 10.3770 keV by using two Ge 333 asymmetric monochromators. The measured FWHM and intensity ratio \( r \) for GaAs \( \pm 333 \) were \( \Delta\Theta = 2.4^\circ \) and \( r = 2.0 \) respectively. The phase \( \alpha_{chr} \) were determined to be \( \mp 40^\circ \). It is noteworthy that this approach should be very useful for phase determination of a nearly perfect protein crystal [2], as it becomes available today.

References