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Observation of Rocking Curves from Polar Crystal for Phase Determination of Crystal Structure Factor

R. Negishi[1,*], T. Fukamachi[1], S. Jongsukswat[1], Ken. Hirano[1], Kei. Hirano[2], and T. Kawamura[3]

[1]Saitama Institute of Technology, Fukaya, Saitama, 369-0293, Japan
[2] Photon Factory, Tsukuba, Ibaraki, 305-0801, Japan
[3]University of Yamanashi, Kofu, Yamanashi, 400-8510, Japan

We have previously reported phase determination of crystal structure factor by using the phase difference between the diffracted and transmitted rocking curves in Laue case [1]. In this paper, we propose another 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 the atomic site is known. Then the crystal structure factor is given by

$$F_h = |F_{hr}|\exp(i\alpha_{hr}) + i|F_{hi}|\exp(i\alpha_{hi}) \quad . \qquad (1)$$

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

$$|F_{\pm h}| = \frac{\omega^2 V \sin 2\theta_B}{8\pi}\Delta\Theta \quad . \qquad (2)$$

Here ω is the X-ray energy, V the volume of a unit cell and θ_B the Bragg angle. The crystal structure factor is also expressed as,

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

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

$$\alpha_{hr} = \alpha_{hi} - \delta \qquad (4)$$

holds and

$$\theta = \tan^{-1}[k\cos\delta/(1 \mp k|\sin\delta|)]. \qquad (5)$$

If the intensity ratio r between from the top and the bottom side of the surface is written as

$$r = |F_{-h|}|/|F_{+h}| \quad , \qquad (6)$$

then the phase δ is given by

$$\sin\delta = \frac{1}{2k}\cdot\frac{r-1}{r+1} \qquad (7)$$

The phase δ can be determined by measuring intensities from the top and bottom surfaces. If $\Delta\Theta$ and r are given by measuring rocking curves, α_{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, Tsukuba, Japan. We tuned the energy at 10.3770 keV by using two Ge 333 asymmetric monochromators. Fig. 1 shows the measuring optical system and Fig. 2 shows the measured ±333 rocking curves near the K-absorption of Ga in a GaAs crystal.

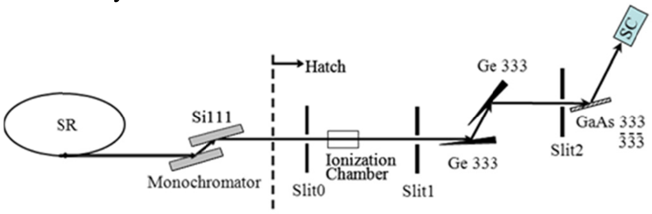

Fig. 1: The measuring optical system at KEK-PF. The asymmetry α of the two asymmetric monochromators at the K-absorption edge of Ga is approximately 12.

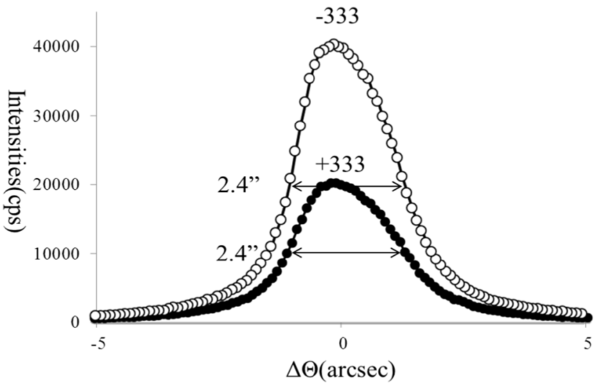

Fig. 2: The measured rocking curves of ±333 reflections from a GaAs crystal at 9.0 eV above the K-absorption edge of Ga.

The measured FWHM and intensity ratio r for GaAs ± 333 were $\Delta\Theta = 2.4''$ and $r = 2.0$, respectively. The phase $a_{\pm hr}$ was determined to be ∓ 40°. Because many protein crystals have polar faces, the current approach should be very useful for phase determination of a nearly perfect protein crystal [2], as it becomes available today.

References

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* negishi@sit.ac.jp