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*A Study on the Condition of Single Crystal Neutron Experiment

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Abstract

The reciprocal space method of increasing the signal to background ratio in X-ray diffraction work with single crystal is extended to the case of equatorial neutron diffraction works. The formulae of optimum width of the detectors with the various experimental methods are derived.

요 약

X-선회절 실험에서 불필요한 관측치에 대한 실제실험치의 비율을 높이기 위한 역격자공간방법을 중성자의 경우에 응용하고 여러가지 실험방법을 택하는 경우에 적합한 검출기의 크기에 대한 관계식을 도출하였다.

1. Introduction

Among the factors which have to be considered in setting the conditions of experiments with counter diffractometers, there are two main things as follows: (i) how to increase the signal to background ratio in the measurement of integral intensities, (ii) how to save the time obtaing the same degree of accruacy.

The effort for finding the optimum condition of the experimental methods with the above considerations has been continued for x-ray and neutron cases seperately. In x-ray case, the so called reciprocal space method has been used usually: [1-3]

Kheiker[3] recently studied on the errors in the measurements of integrated intensities with various methods using non-monochromatic radiation. He has shown that the errors are determined by: (i) the magnitude of the scan volum in reciprocal space, (ii) incorrect determination of thc background, *etc.* On the other hand, the method of tracing the paths of neutrons in a certain experimental geometry have been used in neutron case[4-6]. This technique may be called "tracing method." In these works, have been derived the influence of the collimator parameters(typically the angular divergence) on the luminosity and the resolution in neutron single crystal diffractometry on the basis of Sailor's hypothesis[4]. Caglioti *et al*[5,6] have developed general expressions for the resolution and luminosity of the diffraction peaks for single crystal samples.

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2. Diffraction Profile

To determine the structure factors in diffraction works with single crystals, reflected integral intensities are measured usually. The formula for this intensity may be written as follows:

$$I_{int}=\int_{\Delta\omega} J_o(\omega)\ d\omega \tag{1}$$

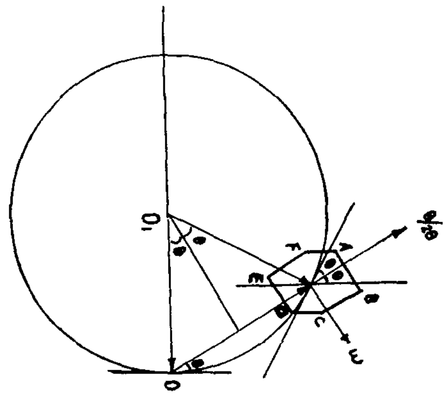
where $J_o(\omega)=J_\alpha(\omega)\ J_p(\omega)\ J_x(\omega)\ J_\lambda(\omega)\ J_m(\omega)$,

- ω: the angle in the real space
- $J_\alpha(\omega)$: the diffraction profile,
- $J_x(\omega)$: the dispersion profile of the incident rays,
- $J_\lambda(\omega)$: the spectrum of the incident ray,
- $J_m(\omega)$: the mosaicity of the sample,
- $J_p(\omega)$: the profile caused by the shape and dimension of the sample,

and $J_o(\omega)$ is the final profile caused by the rotation of goniometer. When the independency among the factors in the integrand is considered, equation (1) is reduced to

$$I_{int}=\text{const}\int J_\alpha(\omega)d\omega. \tag{2}$$

It is possible to compare the final diffraction profiles with the experimental results in order to determine the properly moving intervals of the detector and the smaple in the measurement. It is also possible to compare the results of various methods and to carry out the error calculations.



$AB+H\lambda M+2H\lambda P_{\parallel}\sin\theta$, $BC=H\Delta\lambda+2P_{\perp}\cos^2\theta$, $CD=X$

Fig. 1. Graphical representation of integrated intensity measurement in the equatorial case

3. Construction of Comparing Methods in Reciprocal Space

There is several methods for comparing the results of integral intensity measurements using various methods. The comparing method which has been derived by working in reciprocal space is very neat. The equatorial section of an Ewald sphere involved in the measurement of integral intensity is shown in Fig. 1.

The meanings of various notations in Fig. 1 are as follows:

H_λ: a lattice vector in reciprocal space($H\lambda=2\sin\theta$),

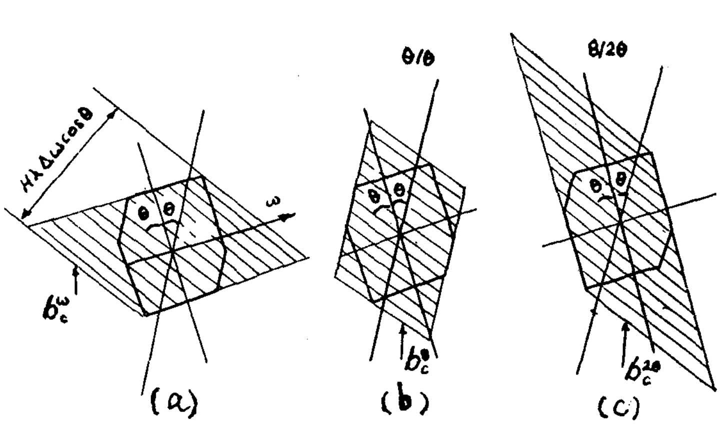


Fig. 2. Scanning volumes for different scanning methods

λ: the wavelength being used,

$\Delta\lambda$: the spectral dispersion in the incident beam,

M: the mosaic spread of the sample,

x: the angular divergence in the incident beam,

P_{11}: the dimension of the sample along the intersection of the reflecting plane and equatorial plane

and $P_{\perp}$ the dimension of the sample along the direction vertical to the reflecting plane.

As easily can be seen in Fig. 2, when the integral intensities are measured, the scanned volumes in the reciprocal space depend on the measuring intervals, measuring methods, and the size of the detector(the height h_c and the width b_c).

The scanned volume has to be made to enclose the lattice point on the Ewald sphere and has to be minimized in order to obtain the largest signal to background ratio.

The scanned volume for every method becomes pararellopipe with the base of the projection of the detector width on the equatorial Ewald sphere. Every height for pararellopipes is the same as HM+X+P. When the ω-scan method(Fig. 2a) is used, the directon of the third side of the prararellopipe is perpendicular to the lattice vector. The third side for θ/θ method (Fig. 2b) becomes to be on the direction of C D and that for $\theta/2\theta$ method be on the direction of the lattice vector respectively. All above the height of the pararellopipes in three cases are the same with the value of $H\lambda\Delta\omega\cos\theta$.

Finally, the problem of making the scanned volume as small as possible turns out to be that how we take the smallest width of the detector b_c. By following the Kheiker's procedure[3], the formulae for b_c's can be obtained as follows:

$$b_c^{\omega}=2\frac{\Delta\lambda}{\lambda}\tan\theta+x+2P_{\perp}\cos\theta \tag{3}$$

$$b_c^{\theta/2\theta}=\frac{\Delta\lambda}{\lambda}\tan\theta+M+P_{\perp}\cos\theta+P_{11}\sin\theta \tag{4}$$

$$b_c^{\theta/2\theta}=2M+x+2P_{11}\sin\theta \tag{5}$$

$$h_c=2M\sin\theta+x_z+P_z \tag{6}$$

$$\Delta\omega=\frac{\Delta\lambda}{\lambda}\tan\theta+M+x+P_{\perp}\cos\theta+P_{11}\sin\theta \tag{7}$$

where the subscript z stands for the component in the meridian plane.

4. Extension to the Neutron Case

In neutron diffraction works, the incident monochromatic beams are selected by the reflections from monochromating crystals. When the angular divergence of the incident beam on the monochromator is large enough to make the mosaicity of this crystal high, the incident beam on the smaple has the divergence of the wavelength, $\Delta\lambda=\lambda_o\cot\theta_o$, where θ_o is the Bragg angle of the monochromator and λ_o is the mean wavelength of the monochromatic beam.

The situations becomes different whether x is bigger than the mosaic spread of monochromator x_o or the reverse.

If $x<x_o$, the beam with angular divergence of x falls on the sample. The wavelength of the falling beam is within the range of

$$\lambda_{min}=\lambda_o\left(1-\frac{x}{2}\cot\theta_o\right) \quad \text{and}$$

$$\lambda_{max}=\lambda_o\left(1+\frac{x}{2}\cot\theta_o\right).$$

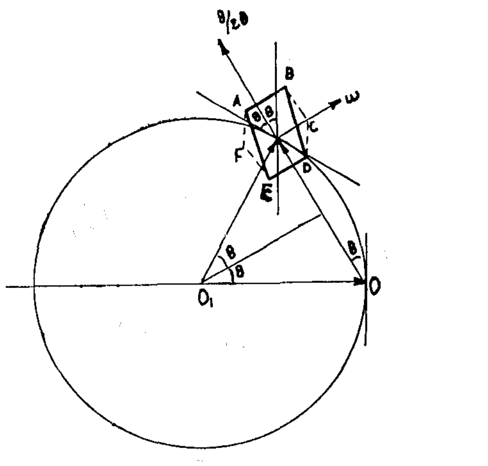


Fig. 3. Integrated intensity measurment in the case of $x>x_o$.

as mentioned above. The results such as equations (3) to (7) are adequate for this case with the substitution $x+x_o$ instead of x_o.

If $x>x_o$, the scanned volume is slightly different from Fig. 1 and is shown as in Fig. 3.

Let's ignore the mosaic spread of the monochromator and the size of the sample for convenience. All the results for b_c^{ω}, $b_c^{\theta/2\theta}$, $b_c^{\theta/2\theta}$, and for h_c turn out to be as the same as equations (3) to (7) respectively. Specially, when the scanning method along the direction of B D is chosen, the b_c can be made small by applying the relation between the rotational speed of the sample ω and that of the detector γ as follows:

$$\frac{\omega}{\gamma}=\frac{1}{1+2\cot\theta_o\tan\theta_o}. \tag{8}$$

The width of the detector and the measuring interval should be as the following:

$$b'_c=2M+x_o+2p \tag{9}$$

$$\Delta\omega=x(1+\cot\theta_c\tan\theta)+M+x_o+p. \tag{10}$$

5. Results and Discussion

The method of taking diffraction data can be chosen by taking the smallest detector width calculated by equations (3) to (5). In author's opinion, the problem of choosing optimum condition for diffraction experiment may be solved more elaborately if the above discribed reciprocal method and the tracing method may be combined. The expression for the full width at half maximum of the diffraction peaks for single crystal sample, $A_{\frac{1}{2}}$, has been given by Caglioti *et al*[5,6]. They have also derived the formula for the luminosity of the diffraction peaks, L[5,6]. The formulae for detector width, equations (3) to (5), and the above $A_{\frac{1}{2}}$ and L may be combined.

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