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Multiple Diffraction Study Using Four-Circle Diffractometer at BL14A

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1 Introduction

Utilization of simultaneous diffraction onto the conventional diffraction measurement has possibility to enable direct phase observation together with quite accurate structure amplitude determination. This should be a breakthrough and truly direct electron density measurement free from conventional statistical estimations to restore the lost phase will become possible. However, multiple diffraction condition can easily fluctuate when reciprocal space is dense or incident wavelength is short. Therefore, for a strict prediction of multiple diffraction condition and estimation of its diffracted intensity, consideration of errors in setting parameters and miss orientation of specimen might be required. In addition to these, some theories estimating the diffracted intensities are not firmly established in practical use. Therefore, we are using a slightly rough theory [1] for diffracted intensity estimation and utilize it to avoidance of simultaneous diffractions for high quality X-ray diffraction measurement. However, the more accurate diffraction measurement is becoming to be needed to detect only a faint breaking of extinction rule or for more accurate electron density measurements. From these backgrounds, aiming to figure out the problem, we have collected basic data of 2θ-ω scan profiles for some reflections including extinction.

2 Experiment

Si standard sample for beamline 14A which is spherically shaped in diameter of 75μm is used as a specimen. Incident wavelength was set to 0.753668Å using double Si-111 single-crystal monochnometer and focused by platinum coated curved fused quartz mirror. 2θ-ω scan profiles are collected using psi-rotation for each reflection ranging from -0.3˚ to +0.3˚ incrementing δψ=0.01˚. To calculate these angle settings a software MDC++ [2] was used. As a standard reflection, 333 is measured after every 61 scans.

3 Results and Discussion

Figure 1 shows the 2θ-ω scan profiles of forbidden 222 reflection as the function of psi-rotation angle and omega. Where intensity is in arbitrary unit. Although 222 reflection is forbidden under *Fd-3m* spacegroup, it is well known that this reflection is not forbidden actually. However, intensity and peak top position of omega was fluctuated by simultaneous diffraction. However, fluctuation of peak top position was also observed for standard reflection of 333 as shown in Figure 2. Although this fluctuation is less than 0.02˚ in omega, this might be the severe difference for the precise estimation of multiple diffraction condition and its intensity. Since standard reflection is always collected at the same four-circle angle position, the main cause of this fluctuation might be a deviation of incident beam direction. Although correction of these fluctuations is not so easy, Collected data were under analysis.

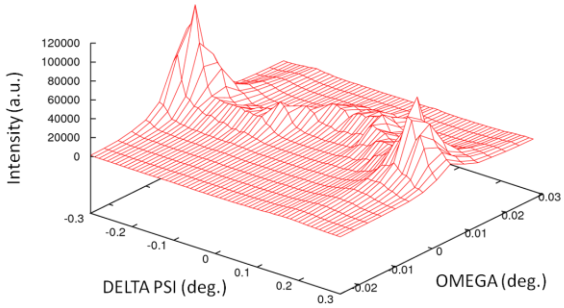

Fig. 1: Si222 2θ-ω scan profiles, as the function of ω and ψ.

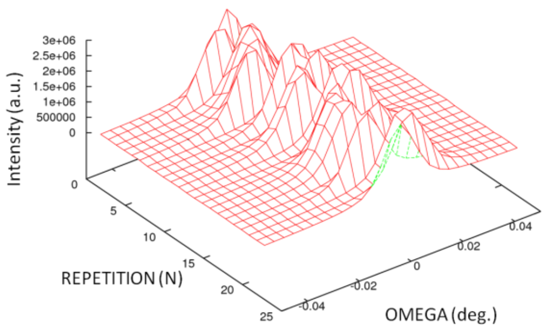

Fig. 2: 2θ-ω scan profiles of standard Si333 reflections, as the function of ω and repetition *n*.

References

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