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Two-dimensional Distribution of Incident Beam Intensities at 14A

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Introduction

BL14A is equipped with the horizontal-type four-circle diffractometer and APD detector, utilizing X-rays emitted from the vertical wiggler. The beamline has helped us to perform very accurate diffraction measurements for many years. Our recent several experiments, however, suffered from a lack of accuracy. Although we can enumerate several causes for this, there seemed to be a tendency, when the size of crystal becomes small, the integrated intensities of the same and equivalent reflections fluctuate significantly. Since the number of mosaic pieces that meet Bragg's law simultaneously becomes small for smaller crystals, fluctuations of direction and/or intensity of the incident beam critically affect the integrated intensity of the diffracted beam. To eliminate problems underlying instability of diffracted intensities, properties of the incident beam have to be investigated. In this paper, we report the intensity distribution of the incident beam around the sample position of the 14A diffractometer.

Experimental

Beam alignment was carried out as usual. From the vertically polarized white X-rays, those of 0.75281Å were chosen using a Si 111 double crystal monochromator and focused by a curved fused quartz coated with platinum. Angle of the first Si 111 crystal and translation of the second Si 111 crystal were adjusted to maximize the incident intensity passing through the incident pinhole of 0.4 millimeter in diameter. Then, heights, translation and tilts of the base stage of the four circle diffractometer were adjusted so that the X-rays pass through a straight line connecting the exit of mirror chamber, the incident pinhole of the diffractometer, and the 0.1 millimeter exit pinhole at specimen position.

A single crystal of Si shaped in a sphere in diameter of 50 micrometer was mounted on the diffractometer. Intensity of -2 6 4 reflection was measured after optimizing four angles, 2-theta, omega, chi and phi. The specimen was then translated along the horizontal (x) and vertical (z) directions on a plane nearly perpendicular to the incident beam, by adjusting the goniometer head. Thus, the intensities were collected on a 2D mesh of about 50 micrometer intervals on the plane. The incident slit of 0.8 millimeter in diameter was then used to examine intensities along the two directions, horizontal x with $z = 0$ and vertical z with $x = 0$.

Results and Discussions

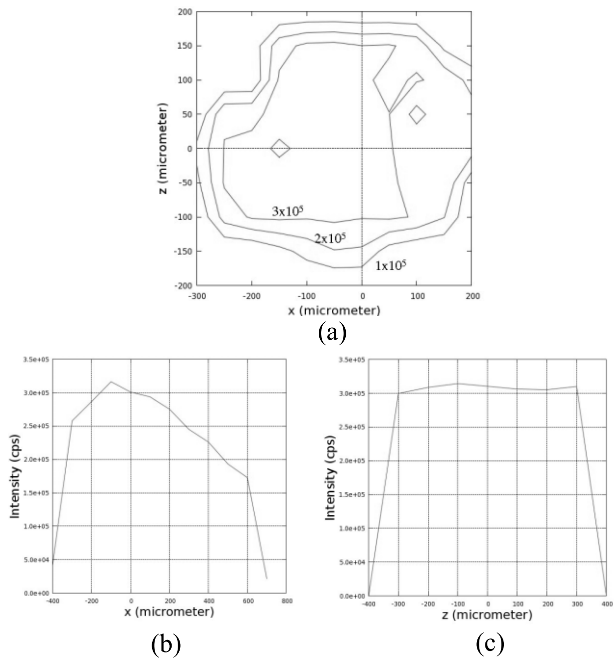

Fig. 1: The relationship between diffracted intensity of Si -2 6 4 reflection and the specimen position; a) x-z map using 0.4 millimeter incident pinhole, b) linear distribution at $z = 0$ using 0.8 millimeter pinhole, and c) linear distribution at $x = 0$.

Size and shape of the incident beam at the crystal position were at the acceptable levels, as shown in Fig. 1. The intensity distribution in Fig. 1b was slightly asymmetric, which could be ascribed to the mirror focusing. During this preliminary 2D measurement of intensity distribution around the crystal position, no significant fluctuation of the incident beam was detected. A sequential intensity data collection on the crystal after this preliminary measurement, however, suffered from instability which was typically seen in the fluctuation of the intensities of standard reflections measured repeatedly after every 200 reflections. Equipping a position-sensitive high-speed devise to monitor incident beam stability throughout the period of data collection seems to be of prime importance for the 14A diffractometer.

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