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Toward high-speed and high-accuracy single crystal X-ray diffraction measurements using the integration-type detector CITIUS

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Photon-counting two-dimensional (2D) detectors have been widely employed in X-ray diffraction experiments at many synchrotron facilities, contributing to improved signal-to-noise ratio and experimental efficiency. Recently, there has been a growing interest in in-situ and operando measurements, taking advantage of high-intensity synchrotron X-rays to observe transient phenomena. For instance, time-resolved measurements using high frame-rate photon-counting 2D detectors, operating at several kHz, have been explored. However, these detectors suffer from counting loss of strong X-rays, affecting data accuracy. While count-loss corrections are employed, their effectiveness is limited to intensities up to a few Mcps/pixel for commercially available photon-counting 2D detectors, posing challenges in accurately measuring X-rays with peak intensities exceeding this limit. Conversely, the integration-type detector CITIUS, which operates without counting loss and exhibits high saturation count rates, demonstrates linear measurement capabilities up to approximately 900 Mcps/pixel (at 10 keV). Deploying CITIUS detectors for X-ray diffraction measurements is anticipated to yield precise data even for highly intense X-ray diffractions.
At the synchrotron facility SPring-8, an upgrade to the low-emittance ring SPring-8-II is planned, expected to increase X-ray flux by approximately two orders of magnitude for experiments using focused X-rays. This increase in X-ray flux is anticipated to enhance resolution in structural analysis of micro-single crystals, enabling measurement of weaker reflections at higher angles, and thereby improving resolution in structural analysis. In addition, the ability to acquire data in a shorter time is expected to enable high-throughput experimentation, facilitating measurements on a larger number of samples with limited access to beam times. To achieve enhanced resolution and throughput, accurate measurement of diffraction intensities with high peak intensities is imperative. Therefore, we aim to integrate CITIUS into a newly designed micro-single crystal structural analysis system for an undulator beamline. We conducted measurements using a prototype CITIUS detector at SPring-8 BL29XUL, employing a standard ruby sample to reveal various challenges. Figure 1 shows a rocking curve of X-ray diffraction by the ruby ball measured with the CITIUS detector. The presence of two peaks, separated by about 0.05 degrees, is likely due to a partial fracture in the sample. We intend to discuss the results of this proof-of-concept experiment and the identified challenges in the presentation.

Figure 1 X-ray diffraction intensity profile of a ruby ball measured by the CITIUS detector with 30 keV X-rays.

I plan to submit also conference proceedings

Yes

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