6A/2000G317

Development of a 3 x 6 arrayed CCD-based X-ray detector for automated macro-molecular crystallography.

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Introduction

A 3×6 module (72µm × 72µm pixel size, 3072×3072 pixel, 221.2×221.2 mm^2 active area size) arrayed CCD-based X-ray detector has been developed for automated macromolecular crystallography with use of the synchrotron radiation at Photon Factory. We have also developed software for correcting the image distortion and non-uniformity of response of the detector. The advantage of our detector over the conventional CCD detectors is concerned with the unique CCD readout scheme, which enables us to record successive x-ray exposures without interruption between them. In other words, neither the interruption of the rotation of the sample crystal nor the X-ray shutter for intermitted exposures is required during the data acquisition. With this detector, it is expected to reduce a length of the data collection time considerably.

Detector configuration

A module of the arrayed CCD-based X-ray detector consists of a phosphor (Gd_2O_2S:Tb, 15 mg/cm^2) which is deposited on an aluminized mylar sheet, fiber optic-taper (FOT) and a CCD as an image sensor. The FOT has a demagnification ratio of 3.2:1 and a high X-ray resistant. The CCD has an area of 25 mm × 25 mm with 1024×1024 pixels of 24 µm × 24 µm. Only the upper half area (1024×512 pixel) of each CCD is used as an active image area, whereas the bottom half is used as a data storage area. Image data recorded in the active area are transferred to the data storage area with a time of 1.6 ms. The next image data can be recorded in the image area while the previous image data stored in the storage area are transferred to a frame grabber in a computer with a time of 2 s. This scheme enables us to detect successive X-ray diffraction patterns without intermission, that is, with a 100% duty-cycle rate. The CCDs are cooled to -30 degree with Peltier devices. The photograph of the 3×6 arrayed CCD-based X-ray detector is shown in Figure 1.

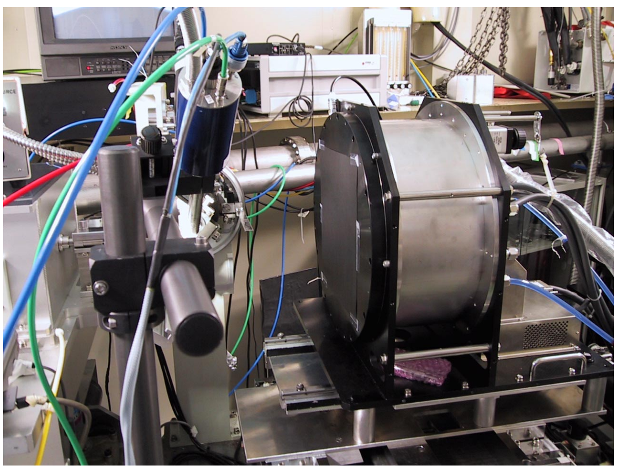

Fig. 1: Photograph of the 3 x 6 arrayed CCD-based X-ray detector.

Experiment and result

We have tested the detector with several protein single-crystals (Lysozyme, AMF, etc.) rotated continuously during data acquisition at BL-6A. Figure 2 shows one of the diffraction patterns taken with a rate of 1-deg rotation per x-ray exposure. An exposure time per frame is 200s with an X-ray wavelength of 0.1nm. Data are processed by “auto” which is written by Higashi. The processed result shows R_{merge} = 9.1% with a resolution of 2.6A. However, R-factor in the low resolution (< 5A) is not reasonably small. The reason is attributed to the system fluctuation which cannnot be corrected by software.

Conclusion

A 3×6 arrayed CCD-based X-ray detector and its correction software was tested. With this, we could obtain a dataset with no interruption between frames. Though the dataset was processed appropriately, there still remained a problem that low resolution statistic was not reasonably small. This problem is expected to be resolved when the system fluctuation in the hardware is improved.

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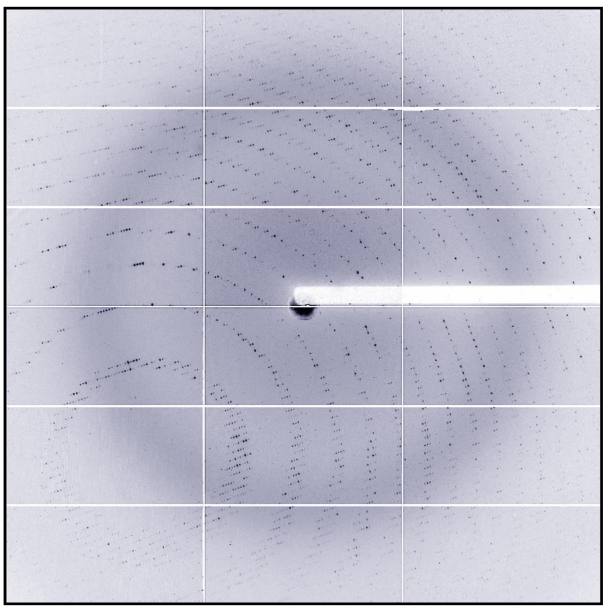

Fig. 2: X-ray diffraction pattern of a AMF (1 deg./frame, 0.1nm wavelength and 200s exposure).