BL02B1
(Single Crystal Structure Analysis)

BL02B1 is designed for single-crystal structure analyses and is equipped with a large cylindrical image-plate (IP) camera, a hybrid photon counting detector, and a four-circle diffractometer. The double silicon crystal monochromator with an included geometry can select monochromatic X-rays between 8 keV and 115 keV from synchrotron X-ray radiations of the bending magnet. Most of the current experiments use monochromatic X-rays between 18 keV and 60 keV. BL02B1 currently promotes charge density studies and *in situ* experiments for functional materials using a large cylindrical IP camera and a hybrid photon counting detector.

The large cylindrical IP camera and hybrid photon counting detector are useful for crystal structure determinations of inorganic and organic materials. Using high-energy X-rays, crystal structure analysis can be performed for inorganic materials with heavy atoms. Because the IP camera provides statistically accurate data due to the wide dynamic range of IP, it is used for precise structure analyses, especially in charge density studies.

In FY2018, we installed a two-dimensional hybrid pixel detector, PILATUS3 X CdTe 1M (Dectris) to improve the performance of single-crystal structure analyses with high-energy X-rays. This detector adopts CdTe for the X-ray detection module, which has a higher detection efficiency for high-energy X-rays compared to the Si module. The detector area is 168.7 mm × 179.4 mm by arranging 10 detection modules of 83.8 mm × 33.5 mm in a 2 × 5 layout.

BL02B1 promotes ultrahigh precise structural analysis with the large curved IP camera to directly visualize the charge density. These kinds of experiments with high-energy X-rays are not realized in other synchrotron radiation facilities. The IP camera provides highly accurate data since the IP can fit into the large curved area. One drawback is a loss time of nearly 10 min for reading and erasing, making it difficult to perform time-resolved structural analysis.

The installation of PILATUS3 X CdTe 1M into the diffractometer realizes a time-resolved charge density analysis with a readout time of 0.95 ms and a frame rate of up to 500 Hz. The detector operates as single-photon counting without noise, and it has the 20-bit counter whose dynamic range is as wide as the IP detector. This configuration enables charge density visualization with high-energy X-rays. Figure 1 shows the diffractometer, which is equipped with a 1/4χ goniometer, IP detector, and PILATUS3 X CdTe 1M. In this setup, a large curved IP camera is still available and can be easily switched with the PILATUS3 X CdTe 1M detector.

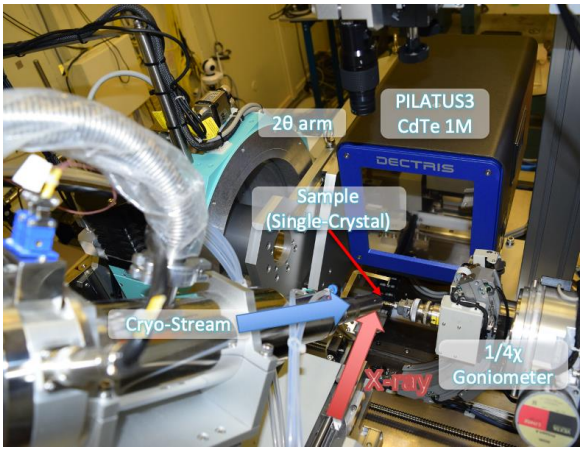

Fig. 1. Diffractometer for single-crystal analysis combined with a 1/4χ goniometer, IP detector, and PILATUS3 X CdTe 1M.

Two-dimensional integration-type detectors such as CCDs and IPs need time to open and close the shutter, to position the diffractometer, and to read the images. PILATUS3 X CdTe 1M enables shutter-less data collection, which means that it is not necessary to close the X-ray shutter and stop the goniometer. Compared to that of Mercury2 CCD (Rigaku), the total data collection time is reduced to 1/10 or less. That is, the time-resolved measurement is 10 times more efficient. This enhancement allows for experiments and structural dynamics analyses at the charge density level.

Through the commissioning in FY2018, we confirmed that the diffractometer with PILATUS3 X CdTe 1M can perform shutter-less measurements, which significantly reduce the measurement time. For instance, we compared PILATUS3 X CdTe 1M with Mercury2 CCD under the same conditions, and measured the entire region of $d > 0.77$ Å using a standard sample of cytidine micro single crystal. For Mercury2 CCD, the fastest data collection time to acquire high-quality data suitable to solve the crystal structure is about 1 h 30 min. For PILATUS3 X CdTe 1M, the time is dramatically shortened to about 5 min. In this commissioning, we demonstrated that the total data measurement time is reduced to 1/10 or less using PILATUS3 X CdTe 1M. In addition, PILATUS3 X CdTe 1M has a dynamic range of 20 bits, which can cover the missing range of Mercury2 CCD with a 16-bit range. This improvement allows diffraction images to be acquired without additional exposure using an X-ray absorber.

Since data reduction is dependent on the software developed by the diffractometer company, the data collection software at BL02B1 supports several data formats. Currently, RAPID-AUTO (Rigaku), CrysAlisPro (Rigaku), and APEX3 (Bruker) are available on the PCs at the beamline.

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