BL02B1
Single-Crystal Structure Analysis

1. Introduction

BL02B1 is designed for high-energy X-ray diffraction experiments on single crystals and features a 4-axis (ψ, υ, φ, 2θ) diffractometer along with a two-dimensional hybrid pixel detector, PILATUS3 X CdTe 1M (DECTRIS AG). This beamline utilizes a Si (311) double-crystal monochromator to select monochromatic X-rays from synchrotron X-ray radiation produced by the bending magnet, with most experiments conducted in the 18 to 60 keV range. BL02B1 is particularly suited for charge density studies and *in situ* experiments on functional materials. The CdTe sensor in the PILATUS3 detector excels at high-energy X-ray detection because of its high efficiency, making it ideal for the crystal structure analysis of inorganic materials with heavy atoms. The detector's wide dynamic range also ensures statistically accurate data, which is crucial for precise structure analyses, especially in charge density studies.

2. Removal of the Imaging Plate Detector for *in situ* Measurements

In FY2023, we removed the imaging plate detector (Rigaku) (Fig. 1), which allowed us to expand the space around the sample, making it possible to install a variety of devices. This expansion enabled the implementation of various *in situ* observation measurements. Following the removal of the IP detector, we successfully conducted *in situ* measurements with light irradiation and electric field application. Additionally, the newly installed PILATUS3 detector, which replaced the IP detector, performed flawlessly during these *in situ* measurements, confirming a successful transition to the new detector system.

3. Preparation of the Source Meter for Electric Field Application *in situ* Measurements

There is substantial demand for *in situ* studies at BL02B1, driven by the high-brightness X-rays and the advanced PILATUS3 detector, which significantly outperforms traditional laboratory systems. To address this demand, we have integrated a source meter for electric field application measurements. While the current challenge is to expand this demand further, we have already successfully conducted an *in situ* observation using the newly installed source meter. Additionally, the

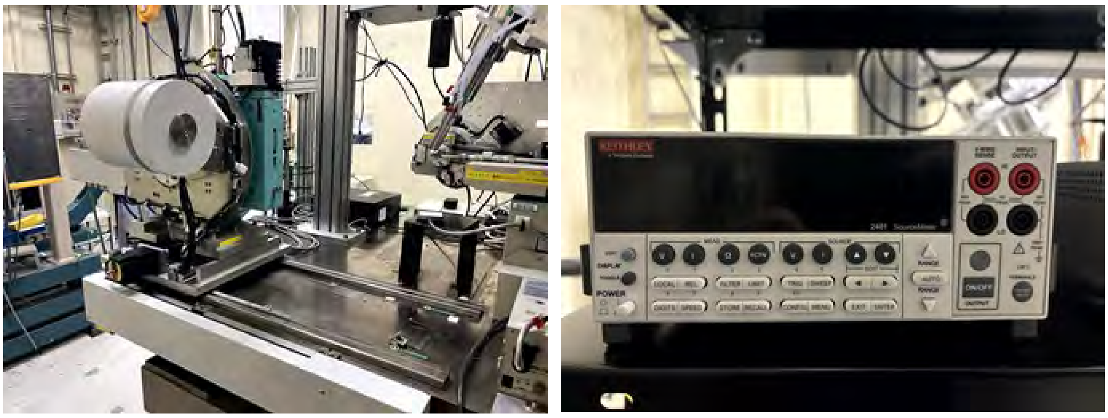

Fig. 1. Diffractometer without the imaging plate detector for the single-crystal X-ray diffraction measurement (left) and a source meter (right).

control software is nearly complete, and efforts are underway to enable remote control from outside the experimental hutch.

4. Development of a data compression and transfer system

In charge density studies, fine slicing is crucial for achieving the highest precision in mapping electron distributions. The additional data points obtained through this method enable more accurate modeling of electron density, which leads to a deeper understanding of bonding and electronic properties. However, this approach generates vast amounts of data. As the frame count increases, transferring this data to a portable SSD can take over a day. To overcome such challenges at BL02B1, we have developed a system that automatically compresses the data after it is transferred from the server, utilizing the Snappy compression format to speed up the process [1].

In our measurement system, after the measurement, data is transferred from the PILATUS Processing Unit (PPU) to the storage for transfer, and subsequently, it is automatically transferred to a storage drive defined by the user. The newly developed data compression and transfer system groups the data stored in the transfer storage into sets of 100 frames, compressing them using the Snappy format before transferring. Snappy is a compression format developed by Google, specifically optimized for compression speed. Typically, one raw data frame output from PILATUS is 4 MB, but compression reduces the size to approximately 1/20 (20 MB for 100 frames). Although the measurement and analysis PCs use Windows for user convenience, a large number of files can significantly increase the load on Explorer. Therefore, by grouping multiple files together during transfer, we achieved a balance between transfer speed and ease of subsequent handling. In addition, one out of every 100 images is copied as uncompressed data so that the quality of the diffraction data can be judged simultaneously. This transfer system enables the completion of data transfer during beam time.

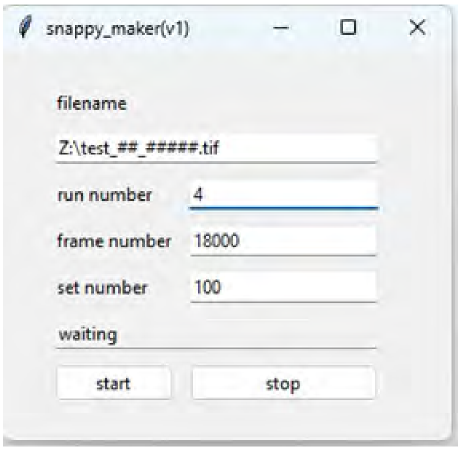

Fig. 2. GUI for data compressing program.

In summary, these improvements have significantly enhanced the experimental environment for *in situ* measurements and greatly improved data transfer efficiency. Moving forward, we will continue developing the system to further advance its capabilities and maximize outcomes.

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Reference:

[1] Google. "Snappy." Google Snappy, https://google.github.io/snappy/. Accessed September 2, 2024.