BL02B1
Single Crystal Structure Analysis

1. Introduction

BL02B1 is designed for single-crystal structure analysis and is equipped with a dedicated diffractometer with a two-dimensional hybrid pixel detector, PILATUS3 X CdTe 1M (Dectris). A Si (311) double-crystal monochromator selects monochromatic X-rays from synchrotron radiation, and most current experiments utilize these X-rays in the range from 18 to 60 keV. BL02B1 currently promotes charge density studies and *in situ* experiments for functional materials. The detector with CdTe sensors is very useful for detecting high-energy X-rays because of their high efficiency. By using high-energy X-rays, we can perform crystal structure analysis on inorganic materials containing heavy atoms. Because the PILATUS3 X CdTe 1M provides statistically accurate data owing to the wide dynamic range, it is used for precise structure analyses, especially in charge density studies.

2. Update of Motor Drivers and Controller for Single-crystal X-ray Diffraction Measurement

In FY2022, we replaced the aged controller (Rigaku) of the diffractometer with the SPring-8 standard motor driver and controller (Fig. 1). This is aimed at improving operational stability and speed for efficiently measuring multiple samples consecutively. In FY2021, we developed a program for single-crystal X-ray diffraction measurement using Python3. This software can control the diffractometer and the detector and generate the files necessary for the single-crystal structure analysis. Additionally, the program implements software limit functions to the motor control that were not previously included, eliminating possible instrument errors caused by incorrect inputs of experimental conditions, and improving safety. While replacing the controller in FY2022, we needed to maintain compatibility with our existing diffractometer. We designed dedicated circuits to convert control signals to the motor. Furthermore, to communicate with the pulse controller, there was a need to update the measurement software, leading us to implement the necessary modifications. As a

Fig. 1. New motor drivers and controller for the single-crystal X-ray diffraction measurement.

result of the equipment update, the measurement time was reduced by approximately 4 minutes per measurement. The measurement for sample screening, which used to take about 7 minutes, can now be completed within around 3 minutes, cutting the measurement time by more than half. Additionally, the operational stability has significantly improved, resolving the issue that operations were halted about once every two weeks.

3. Update of Data Processing Software for Precise Structure Analysis

Recently, there has been an increasing demand for precise structural analysis in charge density studies. To achieve the required precision in measurements, an extensive number of frames need to be exposed for each scan, necessitating the handling of vast amounts of data. In particular, as the frame count increases, the time required to output files containing angle information for each frame becomes significant and cannot be ignored. Under the previous specifications of BL02B1, when exposing approximately 100,000 frames, it took tens of minutes to output the angle information files. In FY2022, we made changes to the specifications of the angle information file. As a result, the time taken to output the angle information files has been reduced to less than one second.

4. Stabilization of Beam Position by Updating the Water-cooling System in the Double-Crystal Monochromator

There was an issue that the X-ray beam position drifted for about 6 hours after changing the X-ray wavelength. This drift was attributed to a change in the thermal conditions of the double-crystal monochromator. With the assistance of the optics team, we changed the water-cooling system used for the Si (311) first crystal from direct cooling to indirect side cooling, as shown in Fig. 2. The new cooling geometry/method notably reduced the drift of the beam position.

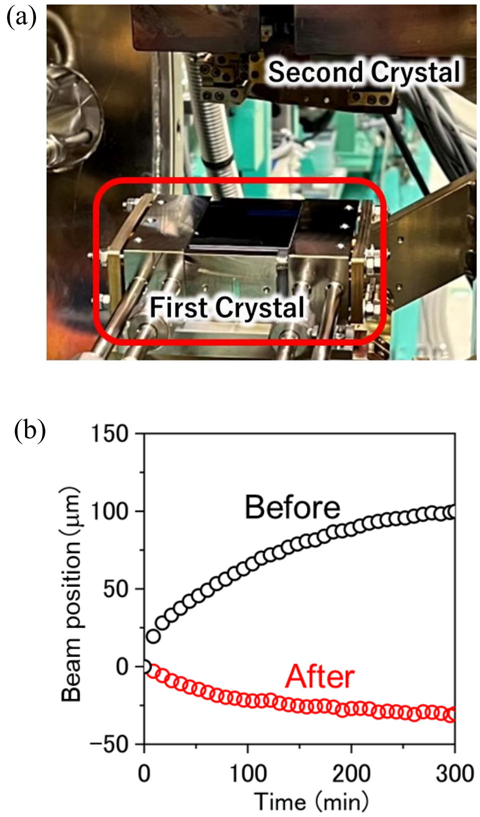

Fig. 2. (a) Si (311) double-crystal monochromator used at BL02B1 and (b) time dependence of beam position before and after replacing the water cooling system.

In summary, we contributed to the enhancement of the device's operational stability and a significant increase in measurement throughput. We plan to continue our efforts towards the high throughput of single-crystal X-ray diffraction measurements, including further automation.

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