BL02B1
Single Crystal Structure Analysis

1. Introduction

BL02B1 is designed for single-crystal structure analyses and is equipped with a two-dimensional hybrid pixel detector, PILATUS3 X CdTe 1M (Dectris). 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 the PILATUS3 X CdTe 1M detector.

The 2-D detector with CdTe modules is very useful because of their high efficiency in detecting high energy X-rays. To use high-energy X-rays, crystal structure analysis can be performed for inorganic materials with 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. Development of centering system for single-crystal structure analysis of small molecule

In FY2019, we have started to develop a fully automated measurement project for single-crystal structure analysis of small molecule. The aim of this project is to simply load the mounted sample into the sample magazine and start the measurement and obtain the data needed for single crystal structure analysis. One of the most challenging elemental techniques in this project is the centering of the samples. Currently, sample centering is done by a person who understands the equipment, and the accuracy is dependent on the person. Thus, it is necessary to manually center the crystals of each sample for accurate measurements. Therefore, the data quality depends on the experience of the single-crystal diffraction experiments. Here, we are preparing to introduce a fully automated measurement system for high-throughput single-crystal structure analysis.

As a first phase of this development, a motorized goniometer with three-dimensional adjustment of XYZ directions was installed, which allowed us to adjust the sample positions remotely from outside the experimental hutch. We developed a graphical user interface (GUI) for intuitive and interactive sample alignment by Python. In this software, non-experienced users can intuitively control a single crystal sample to move to a center position of

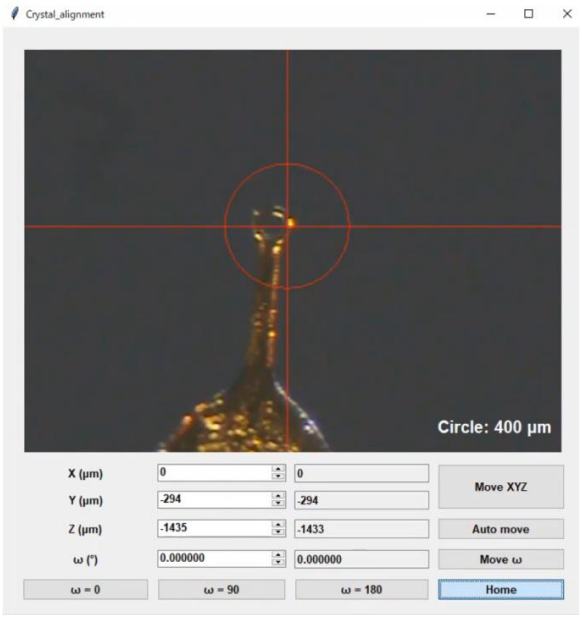

Fig. 1. The picture of graphical user interface of a centering program developed by Python.

goniometer by clicking on the crystal positions displayed on the screens, as shown in Figure 1.

In the second phase, toward the goal of full automation, we have developed an automatic centering program for the mounted single crystal. At the first procedure, it is necessary to automatically recognize the mounted single crystal by the software. The current status is not perfect; however, the vicinity of the sample can be automatically recognized and moved to the center position of the goniometer by the developing software. In order to improve accuracy, we plan to develop a program that recognizes the diffraction pattern and automatically adjusts the sample position precisely to obtain high-intensity diffraction.

In summary, in this project, we are planning to fully automate the whole of single-crystal X-ray diffraction measurements by combining centering system with an automatic exchange robot for single-crystal samples.

Kunihisa Sugimoto, Nobuhiro Yasuda, and Yuiga Nakamura

Diffraction and Scattering Group I, Diffraction and Scattering Division, JASRI