Development of the High-Throughput and High-Accuracy Measurement System for Powder Diffraction

An automatic measurement system for powder diffraction experiments with synchrotron radiation has been developed to achieve both high throughput and high accuracy at the powder diffraction beamline **BL02B2** [1]. The system mainly consists of three parts: an automatic sample changer, an image analyzer for automatic sample position alignment, and an online X-ray CCD detector. These three parts have been successfully synchronized with an original control program written using *LabVIEW*. With this system, the time taken except for X-ray exposure was decreased from several tens of minutes to one minute and the sample position accuracy was improved from several tens of microns to one micron.

The standard procedures of powder diffraction experiments at BL02B2 are as follows.

1. Sample set
2. Sample position alignment
3. Imaging plate set
4. X-ray exposure
5. Imaging plate removal and readout

Here, we describe the concepts of the automatic sample changer and image analyzer, which automate procedure Nos.1 and 2, respectively.

We first had two different ideas for the arrangement of an automatic sample changer. One is to install a sample changer in the existing diffractometer. The other is to develop a sample changer apart from the diffractometer. In the end, we adopted the latter idea, because a thin-film form with reflection geometry as well as capillary form with transmission geometry can be available by utilizing sufficient space. In the latter case, however, the easy and fast setup of the sample changer to the diffractometer was required. Therefore, fine alignment using the stepping motors follows rough alignment using the manual handles. By taking advantage of the two-step alignment, the setup can be completed within 5 min. In the automatic sample changer, a dedicated sample stage was used. Although four axes are usually required, the sample stage is equipped with only two axes to reduce the time for sample position alignment. In order to enable the sample position alignment using only two axes, the design of the sample holder is very important. We used a stainless holder with a sufficiently long groove, which can keep a capillary straight. In addition, a circular sample holder is available for reflection geometry.

Sample position alignment was performed by manually moving the four axes of the goniometer head through the microscope. To achieve automatic sample position alignment, the image analyzer system was introduced. The system can recognize the sample image through the dedicated CCD with one micron resolution. We developed a program to identify the sample edge by differentiating the grayscale and move the sample to the beam center using two axes of the sample stage. Figure 1 shows photographs of the automatic sample changer and a schematic of the automatic sample position alignment.

The system allows us to collect 36 data continuously and automatically. If the X-ray exposure time is sufficiently short, the system acts as a very powerful tool for powder diffraction experiments. The basic design can also be applied to other experiments at synchrotron radiation facilities. Improvements in sample position accuracy and reproducibility by automation contribute to materials science research as well as analytical research.

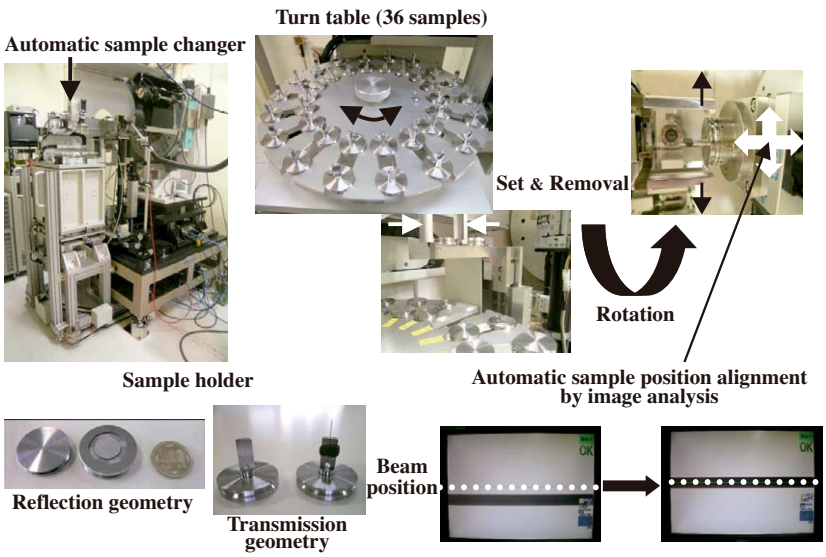

Fig. 1. Automatic sample changer and automatic sample alignment system installed at BL02B2.

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References

[1] *K. Kato, M. Takata and T. Ishikawa: Advances in X-ray Analysis* ***51*** *(2008) 36.*