BL02B2
Powder Diffraction

1. Introduction

BL02B2 is a bending-magnet beamline dedicated to high-resolution X-ray powder diffraction measurements of crystalline powder materials. Powder diffraction experiments clarify the correlation between the crystal structure and physical properties through phase identification, accurate structural analysis, and *in situ* powder diffraction experiments under various external conditions. This beamline provides monochromatic X-rays with an energy range of 12–37 keV ($\Delta E/E$ is approximately 2×10^{-4}). Powder diffraction patterns were recorded with six one-dimensional microstrip MYTHEN detectors [1]. Two types of experiment are conducted: (i) high-throughput powder diffraction experiments using a sample changer and (ii) *in situ*/time-resolved powder diffraction experiments under various conditions. The former type of experiment is automatically carried out for up to 50 capillary samples, and the measurement temperature can be changed from 30 to 1100 K.

For *in situ* powder diffraction experiments under other external conditions, an additional apparatus must be installed into the powder diffractometer. A furnace and a cryostat are available for high-temperature (up to 1473 K) and low-temperature (down to 5 K) conditions, respectively. The remote gas handling system is applicable for controlling the gas and vapor pressures inside a capillary [2]. In addition, users can perform *in situ* powder diffraction experiments using carry-in equipment such as an electric field generator for ceramics, a charging/discharging cell for batteries, and light irradiation systems. Recently, a two-dimensional (2D) flat-panel detector (FPD) XRD3025 has been installed to improve the performance of *in situ* powder diffraction measurements with high-energy X-rays. This FPD can also rapidly evaluate the crystalline grain size using an online readable 2D area detector.

2. Development of an efficient automatic equipment switching system

As described above, *in situ* powder diffraction experiments in various sample environments have been actively performed at BL02B2. The following equipment is frequently switched in accordance with the type of measurement as follows.

a) Sample changer and N_2 gas cooling (or hot) stream device for automatic measurement

b) Large temperature control unit stage for a high-temperature furnace or a low-temperature cryostat

c) Remote gas-handling system, other beamline instruments, and carry-in equipment without using an automatic sample changer and a large temperature control unit stage

Therefore, it is necessary to install/remove the equipment when the equipment used for measurements is changed. It takes much time and effort to install/remove these types of equipment and adjust their positions for X-ray powder diffraction measurements. In addition, the experimental setup may be changed multiple times, even during the user's beamtime of one day; efficient and highly reproducible device switching is required to effectively use the beamtime and allow flexible experimental plans of users.

In FY2020, for switching equipment around the sample environment, we developed an automatic equipment switching system to gain more effective beamtime and reduce hard work, as shown in Fig. 1 and Fig. 2. The details are as follows.

1) The automatic sample changer system and large temperature control unit stage of several hundred kilograms are mounted on a dedicated linear guide rail.

2) The equipment on the guide rail can be installed/removed from the powder diffractometer by using compressed air cylinders. In order to read the evacuation and/or mounted position status, we installed a magnetic sensor on the air cylinders. Furthermore, the positioning mechanism enables accurate alignment quickly using a pallet gripper with a compressed air lock.

3) The positions of apparatuses around the powder diffractometer, such as the N_2 gas stream devices, beam stopper, and CCD camera, are automatically adjusted using motorized stages when switching equipment.

4) The remote control of equipment switching is executed from outside the experimental hatch using the software developed with LabVIEW via an I/O unit.

The time required for equipment switching and position adjustment has been greatly reduced owing to this development. Beamline users can easily switch equipment themselves without the risk of human error, allowing for greater flexibility in experimental plans. In the previous system, the sample changer was mounted on the powder diffractometer, the cables were connected, and then the sample changer was re-adjusted. This process took about 1 h. With the new switching mechanism, re-alignment is no longer necessary owing to the improved positional reproducibility, reducing the switching time to about 1 min. Furthermore, it is

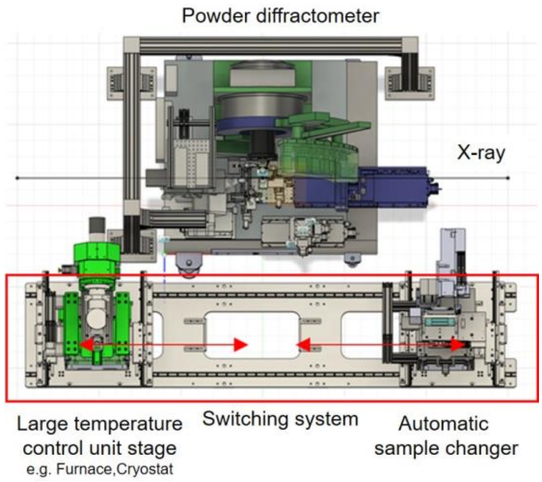

Fig. 1. Illustration of automatic equipment switching system to efficiently connect devices to X-ray position.

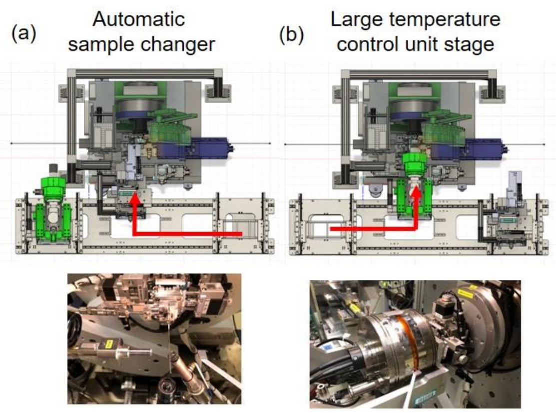

Fig. 2. Illustration of electric automatic installation of a) an automatic sample exchanger and b) a high-temperature furnace stage onto the diffractometer. Lower pictures show a) automatic sample mounting by the sample changer and b) automatic insertion of the high-temperature furnace.

possible to carry out multiple experiments with the switching system, which enables the efficient use of the beamtime. For example, X-ray diffraction measurements using automatic sample changer systems or manual measurements can be conducted while the another measurement sample is being cooled or heated in the cryostat refrigerator on the temperature control unit stage, which usually takes 3 h to cool samples from room temperature to 5 K. In the near future, it is expected that remote experiments will be conducted by switching multiple devices via an external PC even outside of SPring-8.

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