BL02B2
(Powder Diffraction)

BL02B2 is a bending-magnet beamline dedicated to high-resolution powder diffraction measurements of crystalline powder materials. Powder diffraction experiments are conducted to 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. The first optical component of this beamline is a total-reflection Si mirror (M1) coated with Pt and Ni. M1 is used to collimate X-rays and eliminate higher harmonics. A double Si(111) monochromator follows the mirror. In this configuration, monochromatic X-rays with an energy of 12–37 keV are available, and the energy resolution $\Delta E/E$ is approximately 2×10^{-4}.

SPring-8's website shows details of the current status and basic beamline performance of BL02B2 [1]. Two types of experiments are conducted: (i) high-throughput powder diffraction experiments using a sample changer and six microstrip MYTHEN detectors [2] and (ii) *in situ*/time-resolved powder diffraction experiments under various conditions. The former, which is temperature dependent, is automatically carried out for up to 50 capillary samples. The temperature ranges from 30 K to 1100 K. For *in situ* powder diffraction experiments under other external conditions, an additional apparatus must be installed to the powder diffractometer. The furnace and cryostat, which are shared with beamline BL02B1, are available for high-temperature (up to 1473 K) and low-temperature (down to 10 K) conditions. The recently developed remote gas handling system is available to control the gas and vapor pressure inside a capillary[3]. In addition, users can perform *in situ* powder diffraction experiments using carry-in equipment such as an electric field generator for ceramics, charging/discharging cell for batteries, and light irradiation systems.

FY2018 focused on two subjects: (1) increasing the photon flux using a Pt-coated cylindrical mirror and (2) improving the exchange efficiency between the high-throughput measurement system and the *in situ* powder diffraction system.

1. Improvement of data quality using a Pt-coated cylindrical mirror

X-ray beams with an energy of 25–35 keV are useful for research on the correlation between the crystal structure and physical properties. A long acquisition time is required for a powder diffraction pattern due to the low counting efficiency of the MYTHEN detectors for high-energy X-rays. In *in situ*/time-resolved experiments, the powder diffraction pattern must be collected in seconds. In some cases, the Bragg peak intensity is as low as 100 cps since the X-ray beams are not focused horizontally in this beamline. If the X-ray beams can be focused horizontally, the photon flux at the sample position will increase. The aim of this project is to improve the S/N ratio of *in situ* diffraction data and to shorten the measurement time as much as possible.

Eight years ago, the horizontal focusing Si mirror (M2), which is coated with Pt, was installed for experiments under laser heating conditions because the laser spot size was small (<50 μm). The

horizontal focusing mirror had a cylindrical shape with a curvature of 19.82 mm, and it was located downstream after the double-crystal monochromator in the optics hutch. Using the mirror, a focused X-ray beam size less than 100 μm was achieved. Further reducing the beam size to tens of microns, the particle statistics in powder diffraction were remarkably deteriorated, and it was difficult to obtain a uniform Debye-Sherrer ring. Consequently, M2 was not suitable for conventional powder diffraction experiments.

We optimized the horizontal focusing for high-resolution powder diffraction experiments. The horizontal aperture of the front-end slits was changed from 3 mm to 10 mm, and the mirror stage can be adjusted by the motor drivers on its slope (Ry), in-plane rotation (Rz), translation (Y), and height (Z). During the adjustment, the beam shape is monitored at the sample position using a CCD camera. Since the vertical beam divergence deteriorates upon horizontal focusing, the parameters for the M2 mirror can be optimized by changing the bending amount of the M1 mirrors to preserve the original symmetric and non-broadening powder diffraction profile of the standard sample. The optimum parameters of the optics are determined by adjusting the positions of the M1 and M2 mirrors. The obtained beam size along horizontal direction is 1.5–2.0 mm. Figure 1 shows the powder patterns of CeO_2 measured in FY2017 (before optimization) and FY2018 (after optimization). The full width at half maximum (FWHM) of Bragg reflections are approximately same (see inset), but the intensity in FY2018 is three times higher than that in 2017, indicating a three-fold increase in the photon flux. This improved optics not only decreases the total acquisition time

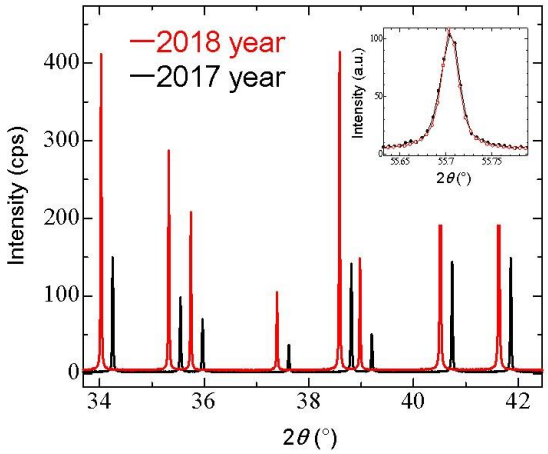

Fig. 1. CeO_2 powder patterns measured in FY2017 (before optimization) and FY2018 (after optimization). 2θ value of data in FY2017 is shifted by 0.2° toward a higher diffraction angle to display the intensity difference. Inset shows the diffraction profiles at the higher 2θ region. Intensity is normalized for comparison of FWHM of peaks.

but also improves the S/N ratio for *in situ* powder diffraction data. These upgrades should allow more users to use the BL02B2 beamline. It should be noted that focused X-ray beams have been available to public users since 2018A.

2. Improvement of the system exchange efficiency

Powder diffraction experiments in BL02B2 can not only automatically measure the powder diffraction using a sample changer but also perform *in situ* measurements under various external conditions. For the *in situ* powder diffraction experiments, the sample changer in front of the powder diffractometer must be moved to another place. In addition, the external goniometer must be combined with heavy equipment (*e.g.,* furnace and cryostat).

The procedure to exchange equipment requires 1–3 h. To minimize the loss time, an exchange system between the sample changer system and the external goniometer system was installed. Figure 2 depicts the exchange system. The sample changer and the goniometer can be easily moved on the rail. The installation improved the position repeatability, and in most cases, the systems do not need to be re-tuned. In addition, the exchange time is reduced to several minutes. Moreover, the development reduces human error and loss time during the system exchange. This development has realized more effective use of the limited beamtime.

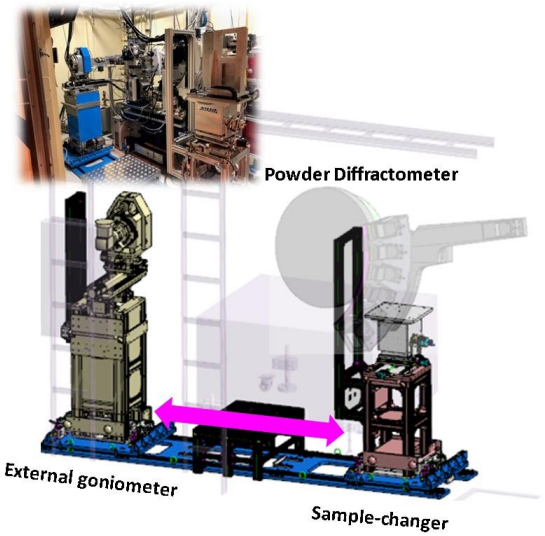

Fig. 2. Picture and schematic drawings of the exchange system.

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