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 of 12–37 keV ($\Delta E/E$ is approximately 2×10^{-4}), and two types of experiments are conducted: (i) high-throughput powder diffraction experiments using a sample changer and one-dimensional (1D) six microstrip MYTHEN detectors[1] 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 30–1100 K.

For *in situ* powder diffraction experiments under other external conditions, an additional apparatus must be installed to the powder diffractometer. A furnace and cryostat are available for high-temperature (up to 1473 K) and low-temperature (down to 5 K) conditions. The recently developed remote gas handling system is available to control the gas and vapor pressure 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, charging/discharging cell for batteries, and light irradiation systems. However, the lack of an online two-dimensional (2D) detector has limited preliminary measurements for user experiments and *in situ* experiments.

2. Development of a measurement system using 1D microstrip and 2D flat-panel detectors

In FY2019, a 2D flat-panel detector XRD3025 (FPD) was 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.

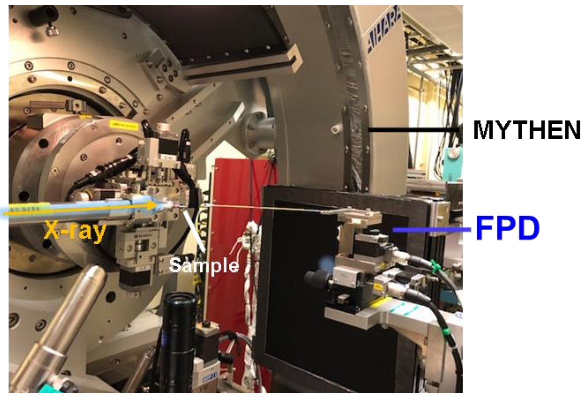

Fig. 1. Powder diffractometer with FPD and MYTHEN detectors.

This detector employs amorphous Si and CsI scintillators as the detection sensors, and high-energy X-rays above 20 keV can be high-efficiently counted. The size of detector area is approximately 250 mm × 300 mm, and the FPD has a dynamic range of 16 bits. For accurate estimations of the peak intensity and width, we adopted a 2D detector with a smaller pixel size, 100 μm × 100 μm. The FPD was placed on the large stage of the diffractometer, enabling various simultaneous measurements with the MYTHEN detector (Fig. 1). In addition, the FPD was placed on a motorized XYZ stage to avoid interference between the 2θ axis and the FPD detector. Lead attached to the front panel shields the circuitry portion of the FPD, while the installed water-cooling plate provides stable operations. This detector arrangement not only enables simultaneous measurements with MYTHEN detectors, but also can satisfy users' various needs. For example, the system can support non-ambient experiments of temperature dependence for chemical reactions. The operation software of the detector was developed with LabVIEW.

Due to its high sensitivity and readout speed, the detector achieved a readout time as fast as 190 ms. Figure 2 shows data for the CeO_2 standard obtained with an exposure time of 1 s. The maximum sample-to-detector distance for the FPD is 350 mm. In this configuration, the 2θ angle can be measured over 40º in a single shot of X-ray with an angular resolution comparable to that of an imaging plate detector. Although the single-shot data collection using MYTHEN measures a limited 2θ region due to the detector gaps, the FPD allows a wide range of 2θ angles to be measured without 2θ gaps. Therefore, future applications such as structural change observations during chemical reactions and phase transformations at high temperatures are expected to use the FPD. On the other hand, preliminary experiments are no longer necessary for grain evaluation using an imaging plate detector because the FPD system can quickly and easily obtain 2D data from the FPD and high angular resolution data from MYTHEN detector simultaneously. Moreover, this development reduces human error such as exchanging imaging plate and loss time during the system exchange. Consequently, the limited beamtime can be used more effectively. In the near future, we will continue to develop programs for automatic processing of 2D data synchronized with the measurements and to upgrade the measurement system to obtain high-quality data more efficiently.

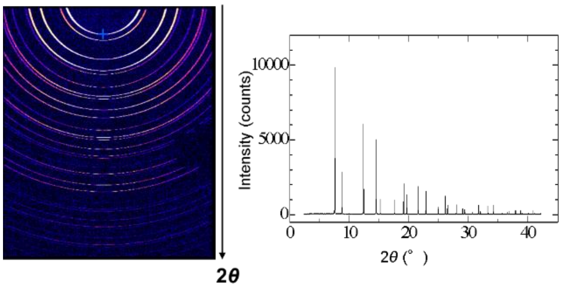

Fig. 2. (left): 2D powder pattern measured by the FPD. (right): 1D powder diffraction pattern calculated from a 2D diffraction image.

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References:

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[2] Kawaguchi, S. et al. (2020). *J. Synchrotron Rad.* 27, 616–624.