BL19B2
Engineering Science Research I

1. Introduction

BL19B2, which is known as the Engineering Science Research I beamline, is one of the original beamlines dedicated to industrial applications. It plays an important role in promoting industrial use of synchrotron radiation. To meet the diverse needs of industry, its three experimental hutches contain a variety of experimental apparatuses for X-ray scattering and diffraction. The first hutch (EH1) contains *Polaris* [1], a versatile high-throughput powder diffractometer, while the second hutch (EH2) has a multi-axis diffractometer. The third hutch (EH3) contains PILATUS 2M, which is a two-dimensional detector for small-angle X-ray scattering (SAXS) with a camera length of 0.7–40 m. A unique feature of BL19B2 is ultrasmall-angle X-ray scattering (USAXS) with a camera length of 40 m.

In FY2019, a high-resolution data acquisition method for powder diffraction was developed using *Polaris*. For the multi-axis diffractometer, an apparatus for heating/cooling planar samples was prepared. For SAXS, a new sample changer robot was installed in EH3.

2. High-resolution data acquisition of powder diffraction

Polaris, which is a versatile high-throughput

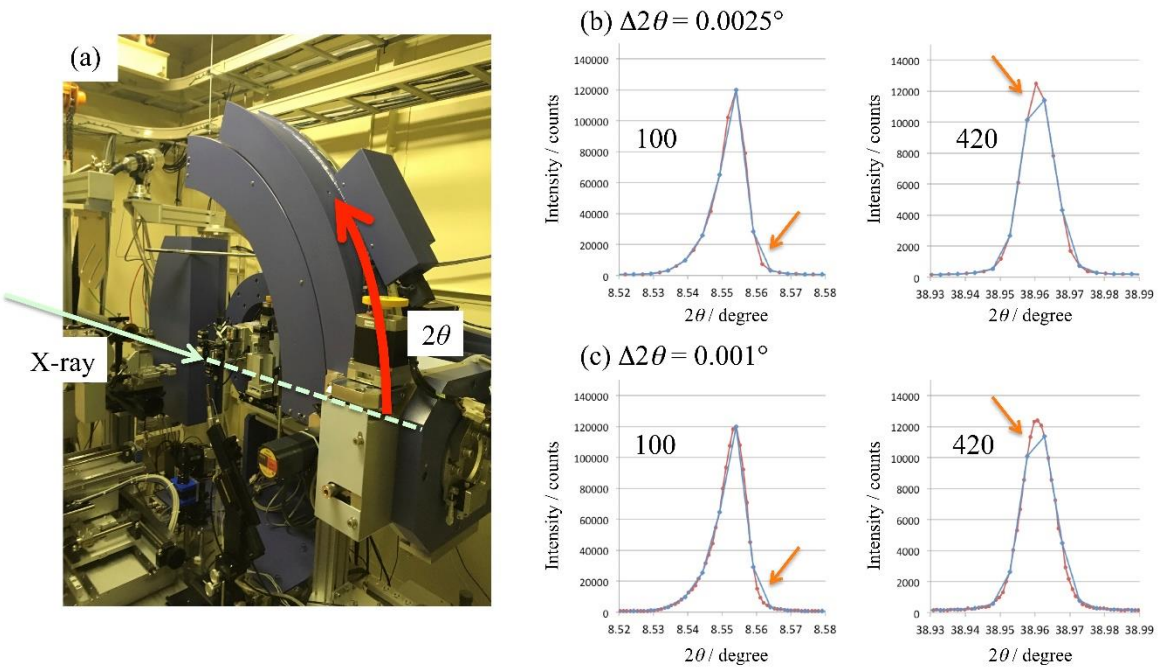

Fig. 1. High-resolution data acquisition using *Polaris*. (a) Overview of *Polaris*. Red arrow indicates the 2θ axis used for displacing the detector array by the displacement $\Delta 2\theta$. Diffraction peaks of the LaB_6 powder sample of (b) $\Delta 2\theta$=0.0025° and (c) 0.001°. Blue and red are plots of the normal mode and the microstep modes, respectively.

diffractometer in EH1, consists of a one-dimensional detector array of MYTHEN modules with a camera radius of 573 mm and an angular resolution of $2\theta = 0.005°$. Because there is a high demand for higher angular resolution measurements of samples with a high crystallinity, in FY2019, a new measurement mode, called the "microstep mode", was developed. This measurement mode realized a higher angular resolution by combining a diffraction profile with one measured by slightly displacing the position of the detector array. The detector array was displaced using the 2θ axis (shown in Fig. 1(a)) by a displacement of $\Delta 2\theta$, which was a finer angle than 0.005°. The red plots in Fig. 1(b) and (c) indicate a closeup of the diffraction peaks of LaB_6 powder samples (SRM 660c) using different microstep modes of $\Delta 2\theta$=0.0025° and 0.001°, respectively. The blue plots indicate the peaks measured by a "normal mode" ($\Delta 2\theta$=0°). Compared to the normal mode, the peaks are more accurately observed by increasing the number of measurement points with the microstep mode (orange arrows).

3. Heating/cooling apparatus for a multi-axis diffractometer

On the upstream side of EH2, a HUBER multi-axis X-ray diffractometer is installed. This diffractometer can perform not only general X-ray diffraction/scattering measurements but also various types of diffraction/scattering experiments such as high angular resolution diffraction, residual strain measurements, anomalous X-ray scattering, grazing-incident X-ray diffraction (GIXD), X-ray reflectivity, and various *in situ* measurements. To perform various X-ray diffraction/scattering measurements, different detectors (a point detector, a line detector, and a two-dimensional detector) can be attached to the detector arms of this diffractometer.

In FY2019, a heating/cooling apparatus (DCS 500, Anton Paar GmbH) was introduced for X-ray diffraction measurements conducted below room temperature (Fig. 2). The DCS 500 is a unique apparatus for *in situ* X-ray diffraction studies at temperatures between −180 °C and 500 °C. It can be fitted to most common four-circle goniometers. The X-ray transparent dome (graphite or PEEK) mitigates condensation at low temperatures or chemical reactions at high temperatures, enabling measurements of samples in a vacuum or inert gas. DCS 500 is now available to users.

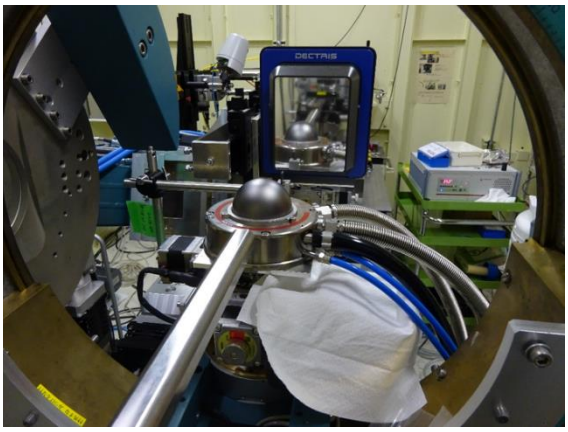

Fig. 2. Heating/cooling apparatus (DCS 500) attached to the HUBER multi-axis X-ray diffractometer.

4. Sample changer dedicated to SAXS

An automated measurement system using a sample changer robot *HummingBird* [2] is a unique feature of SAXS experiments in BL19B2 and USAXS. For a camera length (L) shorter than 3 m (SAXS mode), the sample is positioned in EH3, but L = 40 m (USAXS mode) in EH2. Since FY2014, the

robot has been operating between the two hutches as the experiment mode (SAXS mode, USAXS mode) changes. In FY2019, a new sample changer robot was installed in EH3 for the SAXS mode (Fig. 3). Now, *HummingBird* is exclusively for the USAXS mode. As a result, the labor and time required to move the robot have been reduced, significantly improving the beam time efficiency.

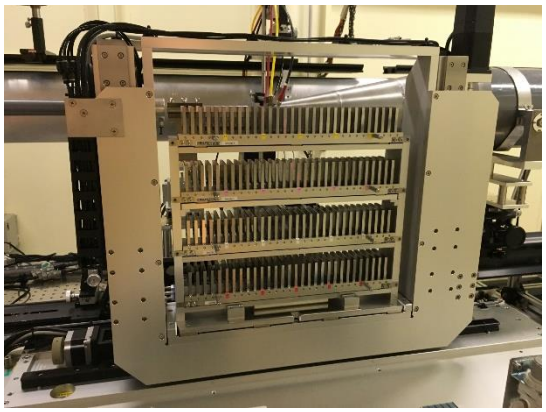

Fig. 3. Sample changer robot dedicated to SAXS measurement in EH3.

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References:

[1] Osaka, K. et al. (2019). *AIP Conference Proceedings*, 2054, 050008.
[2] Osaka, K. et al. (2016). *AIP Conference Proceedings*, 1741, 030003.