BL19B2
(Engineering Science Research I)

BL19B2, which is known as the Engineering Science Research I beamline, is one of the original beamlines constructed for industrial applications. It has played an important role in promoting industrial use of synchrotron radiation. To meet the diverse needs of industry, a variety of experimental apparatuses for X-ray scattering and diffraction are developed using its three experimental hutches (Fig. 1). A versatile high-throughput diffractometer (powder diffractometer) in the first hutch (EH 1) and a multi-axis diffractometer in the second hutch (EH 2) are installed. A two-dimensional detector PILATUS 2M for small-angle X-ray scattering (SAXS) with the camera length (L) of 0.7–40 m is installed in the third hutch (EH 3).

In FY2018, we reconstructed the beamline configuration by moving and abolishing some apparatuses in order to further improve the efficiency of powder diffraction and SAXS experiments. For the multi-axis diffractometer, a germanium (Ge) wafer mirror system, which provides the grazing incident X-ray beam on thin films grown on the air/liquid interface, was upgraded. In addition, a multi-axis oscillating/translating sample stage was developed to precisely evaluate the diffracted intensity from polycrystalline materials.

1. Powder diffraction and SAXS

A versatile high-throughput diffractometer named *Polaris* in EH 1 was installed in FY2017 as the successor to the large Debye-Scherrer camera (LDSC) at the downstream in EH 2 [1]. In FY2018, the sample-cooling and -heating devices operated on the LDSC were moved to *Polaris*, and the LDSC was abolished. Since the detector system was shifted from an off-line imaging-plate reader to an on-line silicon microstrip MYTHEN detector, powder diffraction experiments are completely automated, including *in situ* measurements with changing temperature.

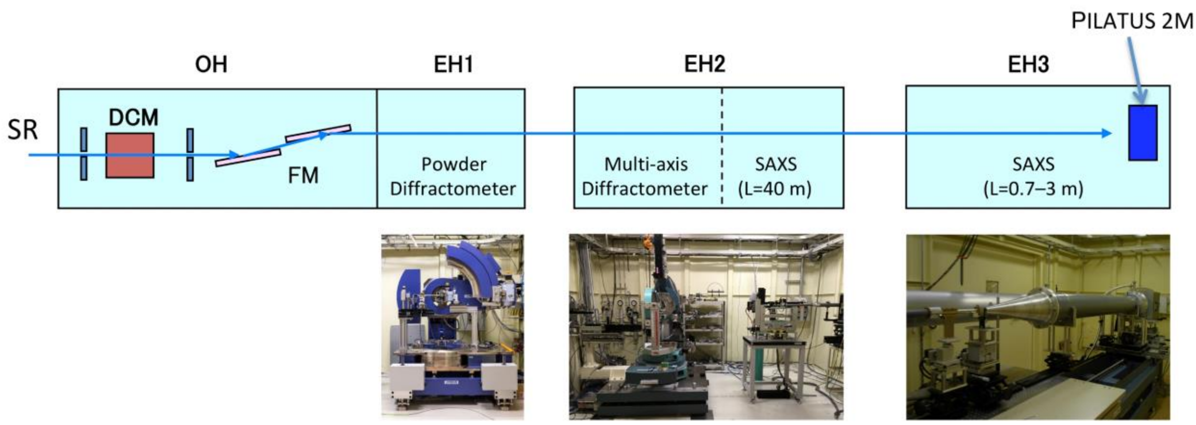

Fig. 1. Overview of BL19B2. SR: storage ring, OH: optical hutch, EH: experimental hutch, DCM: double-crystal monochromator, FM: focusing mirrors. Photographs of the equipment in each experimental hutch are shown.

For SAXS equipment, an experimental setup dedicated to SAXS with a 40-m camera length was permanently installed at the vacant lot created by abolishing the LDSC (Fig. 1). This rearrangement makes it easy to transfer the experimental setup when changing the camera length of SAXS. In addition, we can prepare another experimental setup using the multi-axis diffractometer or SAXS in EH 2 or EH 3 when a fully automated powder diffraction experiment is isolated in EH 1, which is the most upstream. These efforts have enhanced the operational efficiency of our beamline.

2. Multi-axis diffractometer

2-1. GIXD for the air/liquid interface

The multi-axis diffractometer installed in EH 2 is widely used by engineering science researchers. Due to the increased demand to study the crystalline structure of organic thin films grown on the air/liquid interface, in FY2017 we began to develop measurement systems to investigate the crystal structure grown on this interface using grazing incidence X-ray diffraction (GIXD). However, our conventional systems could not study the crystalline structure in detail because the background signal was higher than the weak diffraction signals. In FY2018, we increased the diffraction signal to background (S/B) ratio by adopting a four-dimensional slit system and a guard aperture.

Figure 2 shows a schematic illustration and photograph of the GIXD measurement setup. The monochromatic X-ray is guided downward by tilting a Ge wafer mirror by angle (θ_1). The incidence angle to the sample (θ_2) can be controlled by changing θ_1. We recently adopted a four-dimensional slit system and a guard aperture downstream of the Ge wafer mirror to remove the X-ray scattering from this mirror

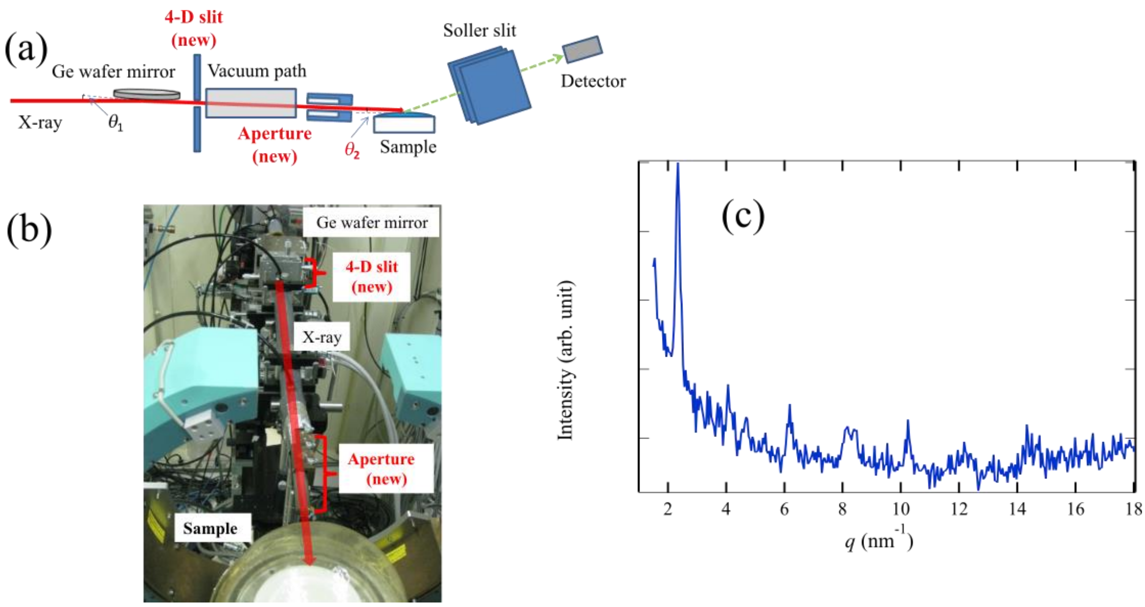

Fig. 2. GIXD measurement setup for air/liquid interface. (a) Schematic illustration, (b) photograph, and (c) GIXD profiles of organic thin films grown on air/liquid interface.

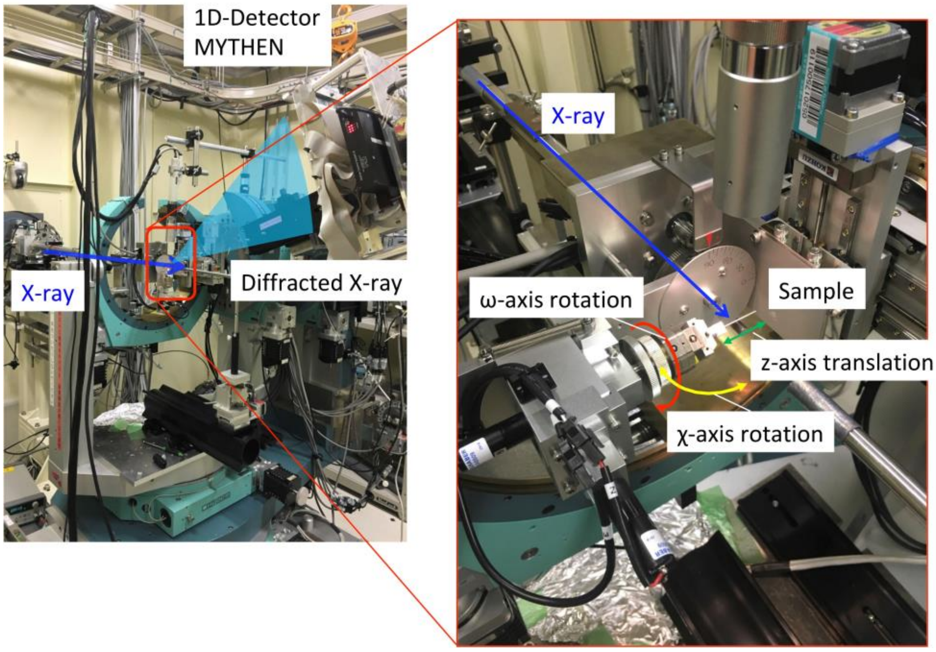

Fig. 3. View of the apparatus to rotate and oscillate samples for X-ray diffraction measurements of polycrystalline samples.

affecting the background signals. This apparatus can be remotely controlled by combing with auto x and z stages. Figure 2(c) shows GIXD profiles of organic thin films grown on the air/liquid interface obtained using our new measurement systems. Consequently, the S/B ratio was improved from 1.2 to 2.7, and we successfully observe many weak diffraction peaks.

2-2. Multi-axis Gandolfi sample stage

An apparatus to rotate and oscillate polycrystalline samples was developed using the multi-axis diffractometer installed in EH 2. The X-ray diffraction profiles from polycrystalline samples are influenced by their crystal texture. If the texture has large crystal grains or an anisotropic crystalline orientation, its influence makes it difficult to evaluate the volume fraction of the crystal phases in a sample from the X-ray diffraction profile. This is often seen in experiments on metallic structural materials. To solve this problem, we introduced a new apparatus to rotate and oscillate samples (Fig. 3). The design of this apparatus is based on a Gandolfi camera with two rotation axes (ω and χ) to average the anisotropic crystalline orientation. Additionally, one translational oscillation axis z parallel to ω axis is equipped to solve the problem due to large grains by averaging the X-ray diffraction signals from many grains.

Keiichi Osaka, Takeshi Watanabe, and Masugu Sato

Industrial Application Division, JASRI

Reference:

[1] K. Osaka et al., AIP Conference Proceedings 2054, 050008 (2019).