BL03XU
(Advanced Softmaterial)

1. Introduction

BL03XU is the first Japanese beamline designed specifically for the polymer field. This beamline is managed and operated by an industrial and academic joint organization (frontier soft-material beamline consortium: FSBL). FSBL consists of 18 research groups from leading Japanese chemical and textile companies and academic researchers from universities. (Fig. 1).

Fig. 1. Logo of FSBL.

The structural features of soft matter such as macromolecules form a hierarchical structure on very wide time and space scales. Each hierarchical structure does not exist independently but instead has a structural correlation, which dominates the physical properties. Therefore, dynamic and structural evaluations should consider the widest possible time and space scales to clarify the correlation between the structure and physical properties of soft matter. BL03XU supports experimental systems focused on small-angle X-ray scattering and wide-angle X-ray diffraction methods to clarify the structure of such soft matter. Here, the features of BL03XU are described, and the microbeam minimum angle for structural evaluation of several hundred nm ($q > 0.006$ nm^{-1}) in the region of several μm was introduced as developed research in FY2018.

2. Experimental hutches

BL03XU employs standard equipment at SPring-8, including a vacuum-sealed undulator, standard transport channel, double crystal spectrometer, and KB mirror configuration. X-rays are focused with a high brightness and uniform wavelength.

The first hutch is equipped with a thin-film horizontal diffractometer (Fig. 2a), which is used for grazing-incidence small-angle X-ray scattering measurement (GISAXS). GISAXS measurements can realize time-resolved tracking of thin-film structures during heating processes. Additionally, simultaneous measurements with bring-in analysis equipment are possible [1].

The second hutch has a large equipment installation space (3 m × 3 m × 4 m) and a large double door that large experimental apparatuses can be carried through (Fig. 2b). Because there are few restrictions imposed by the size and wiring of the device,

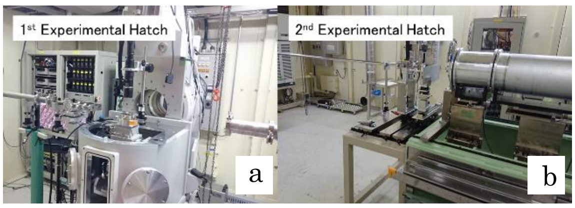

Fig. 2. Photo inside the experimental hutch. (a) Horizontal diffractometer on the first hutch, and (b) Large equipment installation space on the second hutch.

experimental apparatuses can be arranged quite freely. This makes it possible to perform *in situ* analysis of large-scale heating furnaces and melt spinning machines, which have traditionally been difficult [2].

3. Microbeam ultra-small-angle X-ray scattering system for structural evaluation of several hundred nm in the region of several μm

Soft matters such as rubber and polymer materials have various hierarchical structures. Such structural information is indispensable to design high-performance materials. For example, rubber exhibits characteristic scattering due to its structure even in a lower wavenumber region than the q range, which can be measured by ordinary SAXS (camera length of 1–4 m). To measure this, it is necessary to construct an ultra-small-angle X-ray scattering (USAXS) experimental system that can evaluate a smaller-angle region. In X-ray scattering measurements of soft materials, the resultant scattering pattern is derived from the average structure of the part irradiated with X-rays. In other words, average structural information that exists over a wide area can be obtained with a wide beam. On the other hand, in soft materials with complex structures, the local structure and functionality may be strongly correlated. Consequently, the X-ray beam size must be reduced to obtain local information. There are various condensing elements for focusing X-rays such as mirrors, refractive lenses, and diffractive lenses. These elements can reduce the X-ray size, but the focused X-rays diverge after the focal point. Therefore, it is difficult to use a focusing element to form minute X-rays in USAXS measurements when the distance between the condensing point and the detector position is long.

In this beamline, a microbeam is formed by removing X-rays except those through a minute pinhole. In this microbeam small-angle scattering optical system, a guard pinhole is installed immediately before the sample to remove parasitic scattering generated from the minute size pinhole. Specifically, parasitic scattering is removed using Bragg diffraction and by constructing a microbeam USAXS measurement system with a crystal collimator apparatus (Fig. 3). This crystal collimator consists of two sets of two Si-single-crystal units (total of four Si single crystals). When the microbeam X-ray from the small pinhole is incident on the first Si single crystal, it is diffracted at a specific incident angle. The diffracted X-ray enters the second Si single crystal arranged in parallel with the first Si single crystal and it is diffracted again. X-rays generated from the second Si crystal are emitted in parallel with the incident X-rays. Because the parasitic scattering generated at the pinhole has a slightly different angle with respect to the microbeam X-ray, it cannot be reflected by the crystal plane. Hence, this parasitic

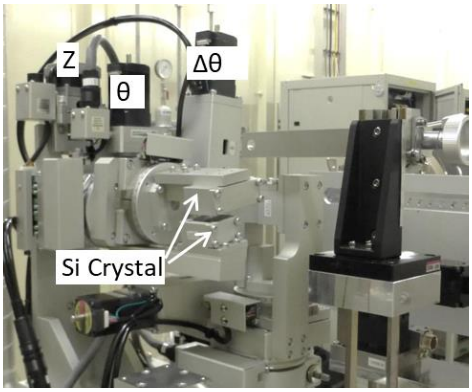

Fig. 3. Photograph of crystal collimator apparatus. *z*- and θ-axes change the height and angle of the first crystal pair. $\Delta\theta$ is the axis that makes the first and second crystals parallel.

scattering is removed. Subsequently, the microbeamam X-ray is reinjected to the next Si-crystal unit, which is arranged vertically. By this operation, it is possible to generate microbeam X-rays suitable for the USAXS experimental system. Figure 4 shows the profiles of air scattering near the beam and a normal pinhole/slit collimation system. The intense parasitic scattering in the stop measured using this crystal collimator system and pinhole/slit collimation system was significantly reduced compared to that measured by the normal system. The micro-beam profile by a wire scan confirms that the beam size is reduced to 5 μm despite the USAXS experimental system. Hence, a microbeam ultra-small-angle scattering optical system is constructed using this crystal collimator system.

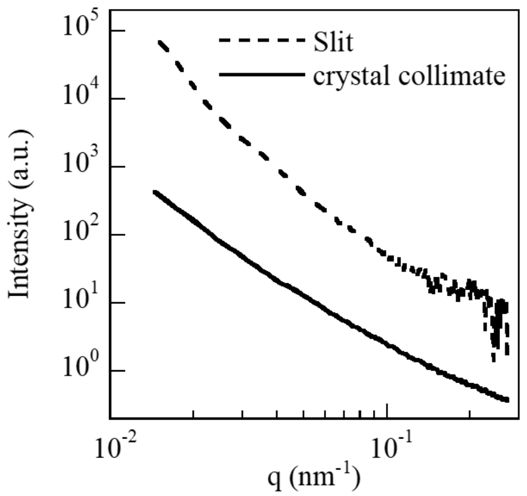

Fig. 4. Intensity profiles by air scattering (no sample) using a crystal collimator apparatus and a slit collimate (dashed line) system.

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References:

[1] H. Ogawa et al., *Polymer Journal* **45**(1): p. pj2012194 (2012).

[2] H. Masunaga et al., *Polymer Journal* **43**(5): p. pj201118 (2011).