BL40XU
High Flux

1. Introduction

BL40XU mainly utilizes the fundamental peak of helical undulator radiation as a quasi-monochromatic X-ray beam without a crystal monochromator. The fundamental undulator radiation has an energy peak width of 2% and a flux as high as 1 × 10^{15} photons/s at 12 keV. Various diffraction, scattering, and imaging experiments have been conducted using these beam characteristics. Experimental hutch (EH) 1 is mainly used for time-resolved small-angle X-ray scattering (SAXS) and wide-angle X-ray scattering (WAXS) measurements, whereas EH 2 is used for X-ray crystallography for microcrystals.

2. EH1

EH1 usually supports time-resolved X-ray diffraction, X-ray single-molecule measurements, and microbeam diffraction/scattering experiments on mainly bio–soft materials.

In FY2022, we introduced stopped-flow equipment as a shared device in BL40XU and BL40B2. A stopped-flow device capable of high-speed mixing of solutions is essential for tracking changes in response to sample mixing reactions and instantaneous changes in solution conditions (pH, salt concentration, etc.). In the past, users had brought in their own stopped-flow equipment, but it was difficult to efficiently perform the experiments owing to the problems caused by the transport of the equipment. We introduced a 4-syringe independently driven stopped-flow device (SFM-4000, BioLogic) [Fig. 1(a)]. This apparatus can be driven independently for four types of solutions, so mixing ratio, mixing pairs, and flow rate can be set as desired [Figs. 1(b) and 1(c)] (mixing ratio from 1:1 to 1:100, maximum flow rate of 8 mL/s/syringe). Efficient operation is expected by externally operating the mixing of solutions, including the reference sample and washing solution. The solution cell holder is compatible with capillaries with an outer diameter of 1.5 mm, and commercially available quartz capillaries can be replaced as needed. The beam-outlet aperture has an open angle of 60°, which enables coverage of both SAXS and WAXS scattering.

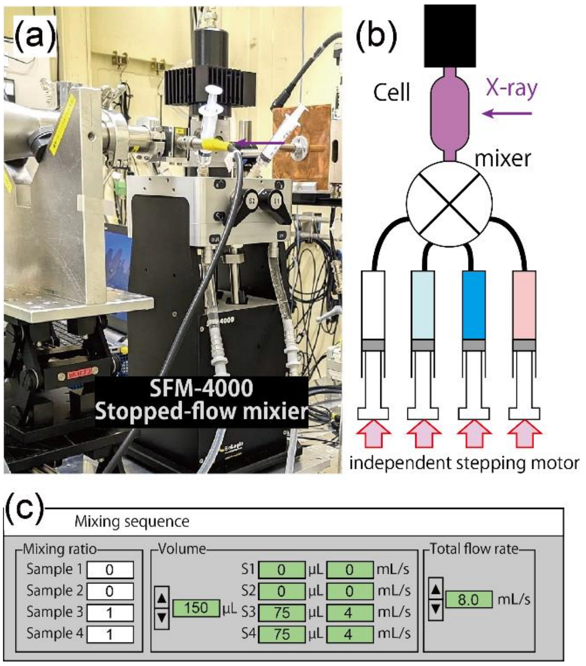

Fig. 1. Stopped-flow equipment.

The X-ray detector comprising a combination of an X-ray image intensifier (Hamamatsu Photonics) and a high-speed camera (Fastcam Nova S16, Photron, 10 kHz) [1] or the Pilatus3 X 300k (Dectris Inc., max. 500 Hz) is applicable as the time-resolved detector in BL40XU. The stopped-flow system that consists of a fast X-ray shutter, the stopped-flow equipment, and the detector has already been tested on protein complexes and liposomes. Users are advised to consult with BL staff in advance because the incident X-ray flux, X-ray exposure time, etc., need to be discussed in accordance with the target phenomena and samples to be observed.

We developed a unit that enables the simultaneous measurement of SAXS and transmission X-ray imaging. The unit is based on an X-ray beam monitor (Hamamatsu Photonics) and has been modified with a thin scintillator (Ce:YAG, 0.03 mmt) with an X-ray (12.4 keV) transmittance of about 90% and a prism with a hole (2 mmΦ) [Fig. 2(a)]. X-ray transmission images are observed through the scintillator and prism, and scattered X-ray signals from the sample (together with those from the scintillator) are acquired through the prism hole. This unit is placed 50–60 mm downstream of the sample and upstream of the vacuum path in a normal SAXS setup, and the X-ray beam is loosely focused (beam size at the sample position is about 0.2 mm square) compared with that in the normal SAXS measurement. Figure 2(b) shows a transmission X-ray image of a microchart specimen (RT-RC-04, Japan Testing Instrument Manufacturers Association) acquired using this unit. Portions with 4.0 µm, 3.0 µm, and 2.0 µm lines and spaces were imaged, and their cross sections are shown in Fig. 2(b). The spatial resolution of the transmission X-ray imaging is to be about 2 µm. Figure 2(c) shows the SAXS scattering images of natural rubber in a tensile test using the simultaneous measurement system. The scattering signals are obtained through the thin scintillator, and a clear difference can be seen between the scattering images of natural rubber with and without tension.

Although BL40XU is a beamline with high-flux X-rays of 10^{15} photons/s (12 keV), the flux should be reduced to about 10^{11} photons/s because of the insufficient durability of the thin-film scintillator, for the simultaneous measurement of transmission imaging and scattering.

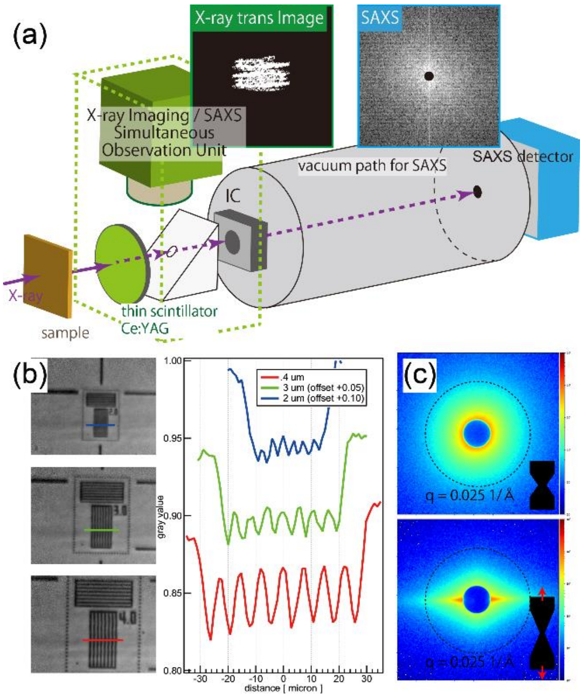

Fig. 2. X-ray imaging/SAXS simultaneous observation unit.

3. EH2

EH2 supports single-crystal X-ray crystallography and diffraction mapping using focused beam and time-resolved X-ray imaging experiments.

It is known that some molecular crystals have different photo-functional properties, etc., depending on their crystal polymorphism. The hard X-ray microbeam at BL40XU is an effective probe for structural analysis of their complex molecular microcrystals. It is very important to determine the molecular structure of a material under light irradiation because the photo-functionality of a material is correlated with its molecular and crystal structure. Until now, X-ray structural analysis before and after light irradiation has been performed by reacting the materials under a microscope. The reacted materials have been measured using X-ray crystallography equipment at EH2. Crystals with a typical size of 50 μm are too large to match the penetration depth of X-ray and UV-VIS light, so structural information could not be extracted with that structure after light irradiation. The micro-focusing technique at EH2 allows users to reduce the sample size and relax the mismatch in the penetration depth between the X-ray and UV-VIS.

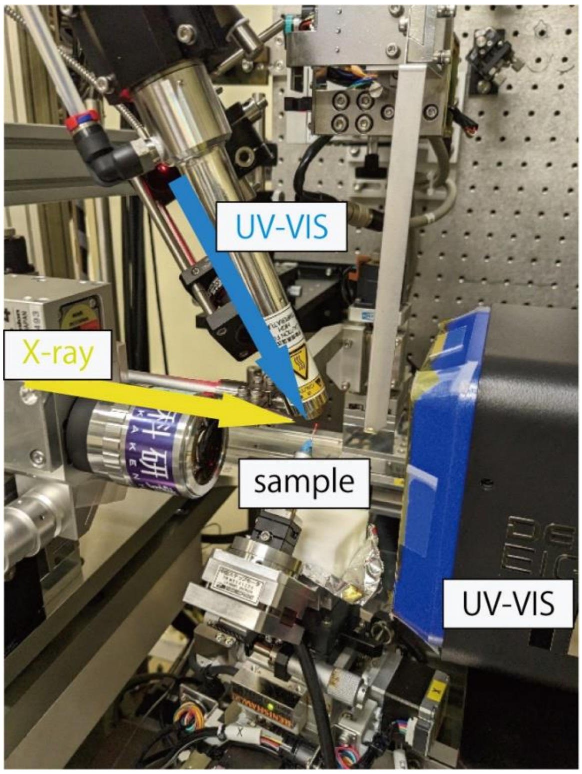

Fig. 3. Photograph of the single-crystal X-ray crystallography system combined with UV-VIS light irradiation system.

In FY2022, we developed a measurement system that enables X-ray structure analysis with UV-VIS light irradiation. UV-VIS LEDs of various wavelengths are available as excitation light sources, and the system can switch the light on and off from outside EH2. Each fiber-coupled high-power LED has various wavelengths and outputs more than 4 mW. The maximum power depends on the wavelength. The LED light is transmitted by optical fiber to the vicinity of the sample, which is mounted on the goniometer of the X-ray crystallography equipment. The excitation light emitted from the end of the optical fiber can be focused by the convex lens from 200 to 600 μm in a diameter at the sample position, as shown in Fig. 3. In order for the device to be user-controllable, the UV-VIS irradiation system will be added to the automatic measurement program. The single-crystal X-ray crystallography combined with the light irradiation system and micro–X-ray beam will be available for user experiments in 2023A.

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Reference:

[1] Yagi, N. & Aoyama K. (2015). *J. Instrum.* **10**,T01002.