BL40XU
High Flux

1. Introduction

BL40XU is a beamline that uses a helical undulator as a light source and focuses the X-ray beam using two reflection mirrors without using a monochromator, making it possible to use a quasi-monochromatic, high-flux X-ray beam (e.g., 10^{15} photons/s, 12 keV, ΔE/E = 0.02).

Taking advantage of these beam characteristics, we are supporting time-resolved small- and wide-angle X-ray scattering (SAXS/WAXS) experiments with sub-millisecond time resolution and microbeam diffraction/scattering experiments mainly in experimental hutch (EH) 1 and X-ray crystallography for microcrystals in EH2.

The beamline was scheduled to be closed in December 2024 for refurbishments, including the replacement of the undulator that is compatible with SPring-8-II, and will be operated as a SAXS/WAXS-dedicated beamline from the 2025B period.

2. EH1

EH1 is typically used to support experiments on bio- and soft materials, such as time-resolved X-ray diffraction, X-ray single-molecule measurements, and microbeam diffraction/scattering experiments.

In FY2023, we worked on a compact unit that can simultaneously measure scattering and transmission imaging as a development method for measuring the dynamics of hierarchical structures. This unit is based on a visible light conversion X-ray imaging system and has been modified by introducing a thin scintillator (Ce: YAG, 12.4 keV X-ray transmission rate: 90%) and a hole-pierced prism (4 mm Φ) [1]. The X-ray transmission image is observed via the scintillator and prism, and the scattering signal from the sample and the scattering from the scintillator are acquired through the prism holes. Thanks to the compact design (45% in volume compared with the previous version of the unit [1]), it is possible to acquire transmission X-ray images while measuring small-angle and wide-angle scattering, as shown in Fig. 1. In addition to being able to evaluate the phenomenon of the growth of nanovoids and precursor structures during the tensile testing of polymer materials and their subsequent destruction in real space and reciprocal space, it is also possible to perform scattering X-ray experiments after positioning the X-ray beam using transmission X-ray images by inserting and removing this unit.

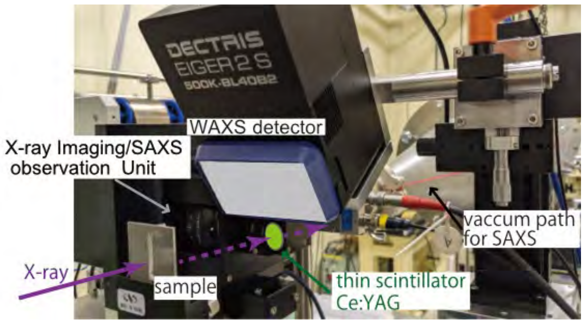

Fig. 1. X-ray imaging/SAXS simultaneous observation unit.

In preparation for the refurbishments to the SAXS/WAXS-dedicated BL from 2025B, we are currently working on introducing shared equipment for sample peripheral devices, mainly for time-resolved scattering experiments.

One such equipment is a stopped-flow device capable of mixing solutions rapidly (SFM-4000, Biologic, France) (Fig. 2). The main component of the stopped-flow device was introduced at the end of 2022. We introduced a diode array spectrophotometer (MMS UV-VIS, BioLogic, France) with high time resolution in FY2023, and the device is now available as a shared instrument. Fig. 2-right shows an example of the transition from a tetramer to a dimer of a protein (hemoglobin) measured at 5 ms intervals.

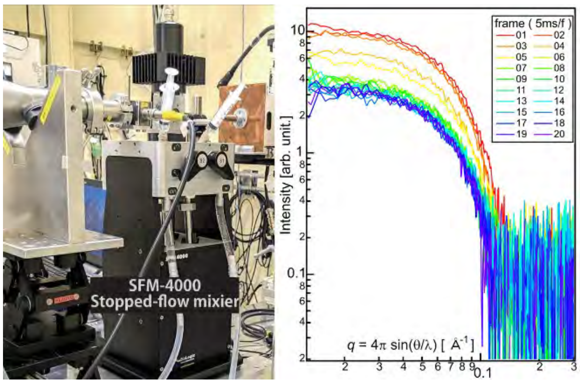

Fig. 2. Stopped flow equipment and time-resolved scattering profiles obtained with the equipment.

In addition, in FY2023, we introduced a rheometer (MCR302e, Anton Paar) that can apply shear to viscoelastic samples. To accommodate a wide range of viscoelastic samples, we have prepared concentric cylinder, cone-plate, and parallel-plate measurement systems. While preparing an environment for performing rheological measurements offline without using X-rays, we will conduct tests at other BLs during the BL renovation and plan to operate the system after the renovation is fully complete.

3. EH2

The high-flux X-ray from synchrotron radiation through a helical undulator at EH2 enables crystal structure determination using a single crystal with a size of less than a few micrometers [2]. This is a considerable advantage over a laboratory X-ray diffractometer, which needs a single crystal with a size over tens of micrometers. The time required for preparing single crystals suitable for X-ray diffraction can be reduced. On the other hand, the smaller the single crystals are, the more difficult they are to handle. Needless to say, handling tiny crystals is challenging and time-consuming not only for researchers without training but also for experienced crystallographers. This suggests that the determination of crystal structures may be hindered not by instrumental limitations but by human skill.

In FY2023, we developed a microcrystal handling system (Fig. 3). The system is composed of a microscope equipped with a motorized lens, an x-y stage, and manipulators that can be controlled by the GUI software “Axis Pro” (Micro Support Co., Ltd.). There are four manipulators and each of them can hold tools, e.g., knives, needles, vacuum tweezers, and motorized tweezers, for handling crystals. Because these tools fixed on a manipulator are controlled by precision motors via the software rather than human hands, crystals can be handled precisely, i.e., the manipulators, and thus the tools, can move along the x, y, and z axes in 0.1 μm/steps without trembling. Moreover, manipulator movements can be controlled by dragging a mouse over the GUI software. This intuitive operability enables users to use the system without special training and saves time for picking up crystals before measurements. In the future, the system will be upgraded to an automated crystal pick-up system by integrating AI-based crystal recognition.

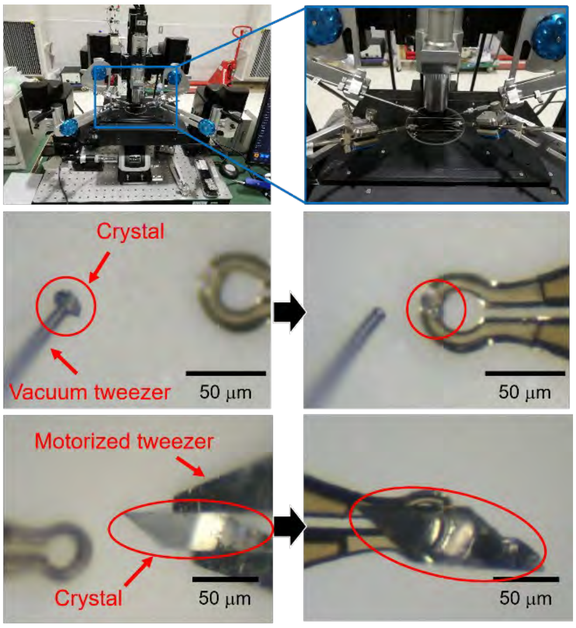

Fig. 3. Photographic images of the microcrystal handling system (top) and demonstration of crystal pick-up using a vacuum tweezer (middle) and a motorized tweezer (bottom).

SEKIGUCHI, Hiroshi[1], ICHIYANAGI, Kouhei[2], SASAKI, Toshiyuki[2], and AOYAMA, Koki[1]
[1]Scattering and Imaging Division, JASRI,
[2]Diffraction and Scattering Division, JASRI

References:

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