

BL40XU High Flux

1. Introduction

BL40XU mainly utilizes the fundamental peak of a 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. Utilizing these beam characteristics, various experiments such as diffraction, scattering, and imaging are conducted. Experimental hutch (EH) 1 is used for various experiments, including time-resolved SAXS/WAXS measurements, while EH2 is used for crystallography and pump-probe experiments.

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.

At BL40XU, we have updated a high-sensitivity, high-speed CMOS camera (Fastcam Nova S16, Photron) to realize time-resolved X-ray scattering and imaging measurements using high-flux X-ray beams^[1] (Fig. 1).

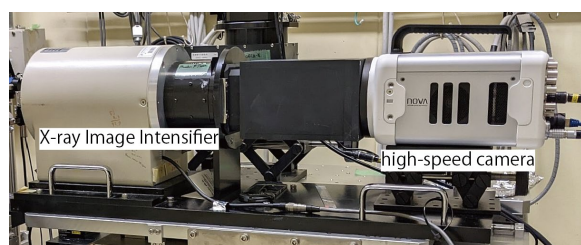


Fig. 1. X-ray image intensifier and CMOS camera system.

We use an X-ray imaging intensifier and a high-speed CMOS camera because the photon-counting

detectors commonly used in other beamlines are unsuitable for measurements with the high flux of incident X-ray beam of 10^{15} photons/s. The updated camera enables high-resolution, high-frame-rate measurements (512×512 pixels, 50 kfps) with high-speed data transfer (10 Gbps). A comparison with the previous camera is shown in Table 1. The previous CMOS camera (Fastcam mini AX200, Photron) required downloading experimental data stored in the camera memory to a local PC after each measurement. For example, it took several minutes to download the data, whereas the X-ray measurement time was only several seconds, which was inefficient. The newly installed camera not only has a larger memory capacity but also allows the camera memory to be divided into multiple parts, enabling X-ray measurements while transferring the data in the memory to a local PC. This function significantly increases the throughput of measurement, especially in cases of time-resolved mapping measurements.

Table 1. Comparison of CMOS camera specifications.

	Previous model	Current model
Resolution	1024 ²	1024 ²
ISO sensitivity	40,000	64,000
Max. frame rate (1024 ² pixels)	6,400 fps	16,000 fps
Max. frame rate (512 ² pixels)	22,500 fps	50,000 fps
Memory capacity	16 GB partitioning not possible	64 GB partitioning possible
Interface	1GBASE-T	10GBASE-T

In the BL40XU EH1, scattering measurements are performed in various sample environments, including tensile testers, temperature-controlled chambers, and animal samples with various sizes and properties. In general, the positioning of the X-ray irradiation is essential for conducting experiments, and we had prepared individual microscope cameras for each experimental environment and requirement. However, the setup was complicated and troublesome. In FY2021, we introduced a long-working-distance microscope (W.D. = 100 mm, optical magnification from $\times 1.2$ to $\times 15$, SEL-LWD 100, Selmic) that can be operated by remote zoom and focus adjustment, as shown in Fig. 2. By introducing a prism mechanism that can be remotely inserted and removed, the microscope can be flexibly adapted to many setup environments, improving X-ray positioning accuracy and unifying setups.

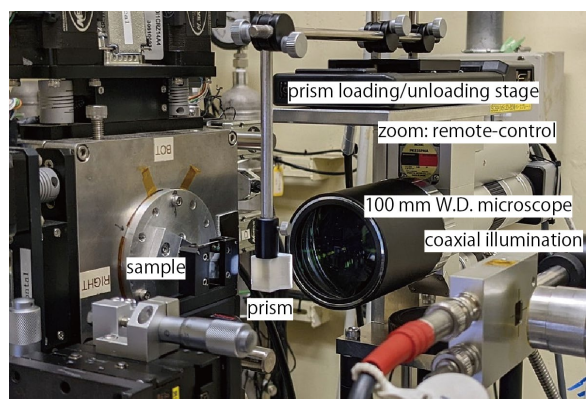


Fig. 2. Long-working-distance microscopy system.

In addition, a humidity control system (RH95, Linkam) was newly introduced. The humidity can be adjusted from 10% to 90% at 25°C with an accuracy of $\pm 2\%$. A cooling, heating, and tensile stage (10073A, Japan High Tech Co./Linkam), which is vital for polymer material evaluation, was

installed and used as a shared device in beamlines (BL40XU/40B2/43IR). A jig compatible with this tensile stage was also introduced (Fig. 3A). The jig has an aperture of about 40° (Fig. 3B), which enables small-angle and wide-angle scattering experiments. It is expected to be used to evaluate humidity-sensitive polymer materials such as hair, silk, and fuel cell separators. The humidity control unit can be operated using the tensile stage control software (Linkam Co.). It can also be controlled independently of the tensile stage from the touch panel on top of the unit. Then, we can utilize the humidity control unit in any experimental chamber by introducing a humidity/temperature sensor and a fixture for introducing humidity-controlled air.

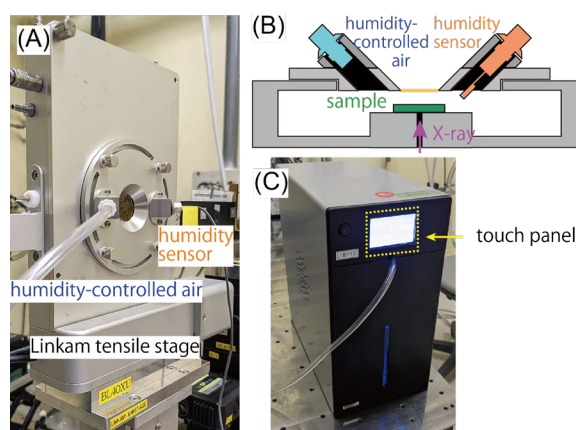


Fig. 3. Humidity control chamber.

3. EH2

EH2 supports single-crystal X-ray diffraction, diffraction mapping using a focused beam, and time-resolved X-ray imaging experiment.

For single-crystal X-ray diffraction experiments, the intersection of a micro-focus X-ray beam and the rotation axis is very important to measure the diffraction intensity accurately. In particular, the eccentricity of the ω -axis causes significant intensity fluctuation. To reduce the

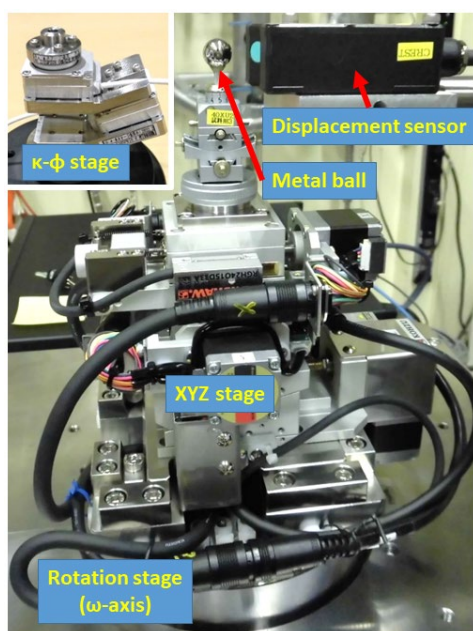


Fig. 4. Goniometer system for new diffractometer.

eccentricity during rotation, two types of bearing and motor were installed in the high-precision diffractometer at BL40XU EH2. Recently, a new diffractometer has been designed, which will be more usable for experiments combined with a micro-focus X-ray beam. An air-bearing-type rotation stage, therefore, is selected as the ω -axis because of its high stability and low eccentricity.

A picture of a goniometer with an air-bearing-type rotation stage is shown in Fig. 4. A small κ - ϕ stage is mounted on the rotation stage to measure a single crystal. To align the crystal on the rotation axis, an XYZ stage with 10 nm position sensors is also mounted on the rotation stage.

To evaluate the eccentricity of the rotation stage, the displacement of a metal ball was measured with a laser displacement sensor. After aligning the metal ball, the displacement data were measured at rotation speeds of 1, 5, and 10 \circ /s. Figure 5 is an eccentricity plot at 10 \circ /s, and the

eccentricity at each rotation speed is shown in Table 2. As the rotation speed increased, the eccentricity became smaller. The eccentricity is almost small enough for the micro-focus X-ray measurement. To reduce the eccentricity at a lower rotation speed, the position feedback given by the XYZ stage needs to be developed.

Other components such as the optical microscope, two-dimensional detector, and control software are also being upgraded for more accurate single-crystal X-ray diffraction experiments.

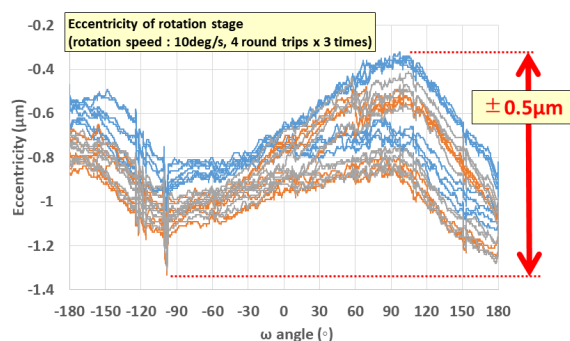


Fig. 5. Eccentricity of rotation stage at 10 \circ /s.

Table 2. Eccentricities at rotation speeds of 1, 5, and 10 \circ /s.

Rotation speed (\circ /s)	Eccentricity (μm)
1	± 1.0
5	± 0.6
10	± 0.5

Sekiguchi Hiroshi¹, Yasuda Nobuhiro², Fukuyama Yoshimitsu², Kobayashi Shintaro², Nakamura Yuiga², and Aoyama Koki¹

¹Scattering and Imaging Division, JASRI

²Diffraction and Scattering Division, JASRI

Reference:

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