BL40XU
(High Flux)

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, XAFS, and imaging are conducted. Experimental hutch (EH) 1 is used for various experiments, while EH 2 is used for crystallography and pump-probe experiments.

1. EH 1

EH 1 is used for time-resolved X-ray diffraction, X-ray single-molecule measurements, and microbeam diffraction/scattering experiments on mainly bio-soft materials. In FY2018, we evaluated the features of the quasi-monochromatic X-ray beam, which is also known as a pink beam, for bio-soft materials by comparing the results obtained by a monochromatic X-ray beam. The channel-cut monochromator (Si 220) can be moved in and out of the X-ray path (Fig. 1 (a)). We compared protein SAXS profiles by in-line Size Exclusion Chromatography SAXS (SEC-SAXS) measurements, which can resolve components in polydisperse mixtures for SAXS analysis. The protein solution, bovine serum albumin (BSA), was injected to the SEC-column (Superdex 200 10/300GL) with a flow rate of 0.5 ml/min with a Tris buffer (50 mM Tris-HCl, 150 mM NaCl, pH=7.5), the eluted solution flowed into a quartz capillary (2-mm diameter) for SAXS measurements (0.5-s exposure, 1.2-s interval, X-ray shutter controlled by a galvanometric motor type shutter). The absorbance (280 nm) of the eluted solution was recorded between the SEC-column and the quartz capillary by UV monitoring (Bio-Rad).

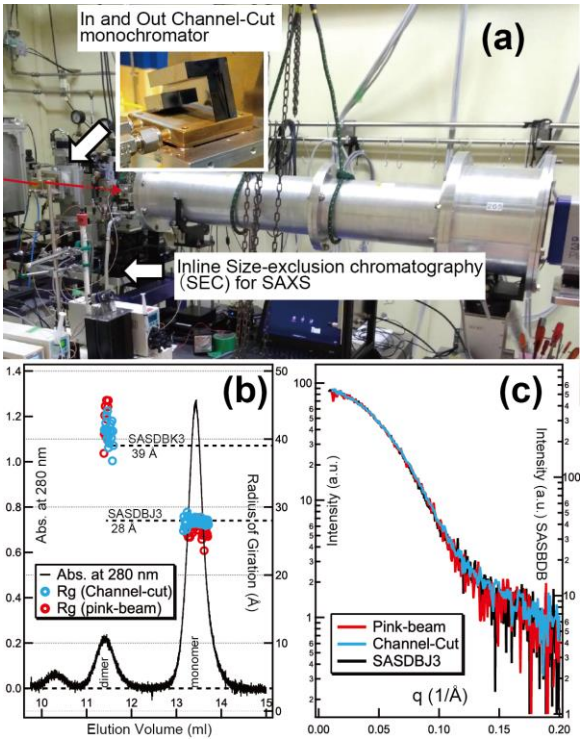

Fig. 1. (a) SEC-SAXS analysis with and without the channel-cut monochromator. (b) Elution profile of the BSA solution and Guinier analysis under SEC-SAXS. (c) Comparison of SAXS profiles of the BSA monomer with different X-ray sources.

Figure 1 (b) shows the elution profile evaluated by UV absorbance and the results of Guinier analysis. The absorbance profile contains three peaks. The second and third peaks correspond to the dimer

and monomer of BSA, respectively [1]. The result of Guinier analysis with the pink beam (with 1.75-mm-thick Al absorber) and the monochromatic beam showed the same tendency. The radii of gyration obtained by monochromatic and pink beam were 27.5 ± 0.3 Å and 27.0 ± 0.7 Å (monomer) and 40.6 ± 1.7 Å and 41.3 ± 2.6 Å (dimer), respectively. The SAXS profiles overlapped within q-range up to 0.15 $Å^{-1}$ (Fig. 1 (c)), and the results are consistent with previously published and deposited results in SASBDB [2] (SASDBK3 for dimer and SASDBJ3 for monomer). Hence, the difference in the SEC-SAXS results obtained by the pink beam and the monochromatic beam is small, at least for protein-SAXS measurements. Hence, the pink beam can be used when radiation damage of the sample is a concern.

The photon flux after the channel-cut monochromator (12.4 keV) is about 2×10^{12} photons/s, and the variance of flux is 0.5% during one series of the experiment (0.5-s exposure, 1.2-s interval, 2,000 times) (0.2% for pink beam). The channel-cut monochromator system is useful for users who need time-resolved WAXS/SAXS simultaneous measurements since there is a limited number of public undulator beamlines for such measurements at SPring-8.

We installed a photon-counting area detector, Eiger 2S 500k (Dectris), mainly for the WAXS detector in WAXS/SAXS simultaneous measurements. (See Table 1 in BL40B2's report for technical specifications as the same type of detector is also installed in BL40B2). The installed detector can be replaced by the flat panel detector (FPD, C9728DK, Hamamatsu Photonics) that is currently used as a WAXS detector. The advantages of the installed detector are a sampling frequency of 40 Hz (FPD: 3 Hz) and no dark current.

Table 1. SEM specifications

Light optical	Magnification range: 20-135x
	Sample loading time: < 5 sec
Electron optical	Electron source: CeB_6
	Magnification range: 80-150,000x
	Sample loading time: < 30 sec
Resolution	≤ 8 nm SED and ≤ 10 nm BSD
Acceleration voltages	5 kV, 10 kV and 15 kV
Detector	Backscattered electron detector
	Secondary electron detector
	Silicon Drift Detector (for EDS)
Sample size	Up to 32 mm diameter
	Up to 100 mm height
EDS Specifications	Element detection: B to Am
	Energy resolution: MnKα ≤ 132 eV

2. EH 2

EH 2 supports not only single-crystal structure analysis of sub-micrometer small crystals and the diffraction mapping using focused beam but also X-ray imaging to develop next-generation fuel injection nozzles using a picosecond X-ray pulse with a single bunch at SPring-8.

2-1. SEM system for observations of small samples

For single-crystal structure analysis with micrometer and sub-micrometer scale crystals, a single crystal is attached to the top of a thin glass needle. To measure accurate data, identification of the crystal morphology and sample condition are very important for diffraction experiments using a small crystal and diffraction mapping with focused X-ray beam. However, in principle, these samples are too small for optical microscopy to measure its shape and size. Therefore, to identify very small samples, a desktop size scanning electron microscope (SEM) system has been installed.

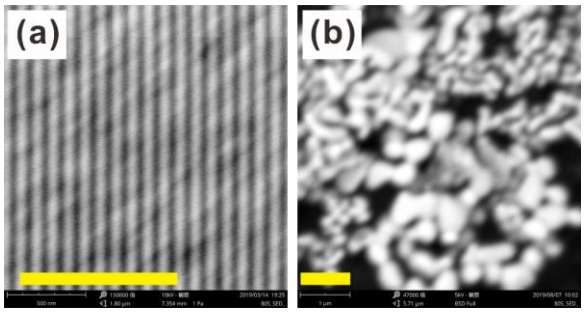

Fig. 2. SEM images of (a) the 100-nm line pattern and (b) CeO_2 powder (NIST 674b, particle size: 0.53–2.18 μm). Each scale bar indicates 1 μm.

The specifications of SEM and typical images are shown in Table 1 and Fig. 2, respectively. A short loading time is convenient to observe an electron microscope image. Two detectors, a backscattered electron detector (BSD) and a secondary electron detector (SED), are available to observe the chemical contrast and surface information of the samples, respectively. Energy-dispersive spectrometer (EDS) is also available to analyze element information. Chemical composition analysis of the sample with an EDS technique is useful to confirm if it is a target sample or target position. SEM and EDS techniques are useful not only for diffraction measurements of small samples but also for spectroscopy of small surface regions. This SEM system can be moved to other beamlines to check a sample immediately.

2-2. ω Axis rotation stage

An air-bearing stage was used for the sample rotation axis (ω axis) for a high-precision diffractometer installed in BL40XU because a low sphere-of-confusion is necessary to align the small sample on the microbeam during the diffraction measurement. To achieve a higher stability and accuracy of the ω rotation, a direct-drive rotation stage and *xyz* stage have been installed. The sphere-of-confusion of the new rotating stage is almost the same as the air bearing–type stage. The cables for the *xyz* stage and the position sensors are connected with the slip ring. Consequently, the ω axis can rotate continuously. The driver module can communicate with a PC by simple TCP/IP commands as well as synchronize the detectors such as EIGER, which was installed last year.

In 2017 and 2018, the main components of the high-precision diffractometer, the detector, and ω rotation axis were replaced successfully. Using the

upgraded diffractometer and SEM system, experiments with very small samples are promoted with high accuracy of data and rapid assessment of sample conditions.

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References:

[1] C.M. Jeffries et al., *Nature Protocols* **11**, 2122–2153 (2016).
[2] Small Angle Scattering Biological Data Bank (SASBDB) https://www.sasbdb.org/