

## BL40XU High Flux

### 1. Introduction

Since the start of public operation in April 2000, BL40XU has long been utilized as a high-flux beamline for time-resolution scattering, diffraction measurements, and imaging<sup>[1]</sup>. In FY2024, the beamline was shut down in late December 2024, and refurbishments were initiated in preparation for its re-opening in October 2025 as a dedicated SAXS beamline. The refurbishment project of BL40XU is part of the SAXS beamline reorganization at the SPring-8 site, planned with input from SPRUC research groups and user communities. The reorganization aims to consolidate SAXS-related activities at the SPring-8 site, thereby accommodating the diverse requirements for sample types and measurement ranges (temporal and spatial), while also incorporating provisions for the upgrade toward SPring-8-II.

Previously, BL40XU delivered a quasi-monochromatic X-ray beam generated by a helical undulator to the experimental hutches via horizontal and vertical focusing mirrors. In this configuration, EH1 primarily supported SAXS and WAXS measurements, while EH2 provided facilities for single-crystal structural analysis. As part of the refurbishment, the diffractometer in EH2 was

removed, and its activities will be transferred to the future BL05XU beamline.

This annual report presents the activities of EH1 and EH2, followed by a description of the beamline refurbishment.

### 2. EH1: Refurbishment into Dedicated SAXS BL

The refurbishment involved a complete redesign of the optical system and the installation of a new detector booth downstream of EH2, enabling USAXS measurements with a camera length of 10 m. The insertion device was also replaced with an undulator (IVU-II, 28 mm period) compatible with SPring-8-II. An overview of the refurbishment is shown in Fig. 1.

In the optics hutch, a mirror system comprising a horizontal focusing mirror (m1h), a vertical mirror (m2v), and a vertical focusing mirror (m3v) was installed for quasi-monochromatic operation, along with a double channel-cut monochromator [DCCM, Si(111)] for monochromatic operation. For each mirror, the reflecting surface can be selected between Si and Ru depending on the usable energy range, operated at a grazing angle of 3 mrad. The DCCM can be inserted or retracted along the beam axis, enabling the use of quasi-

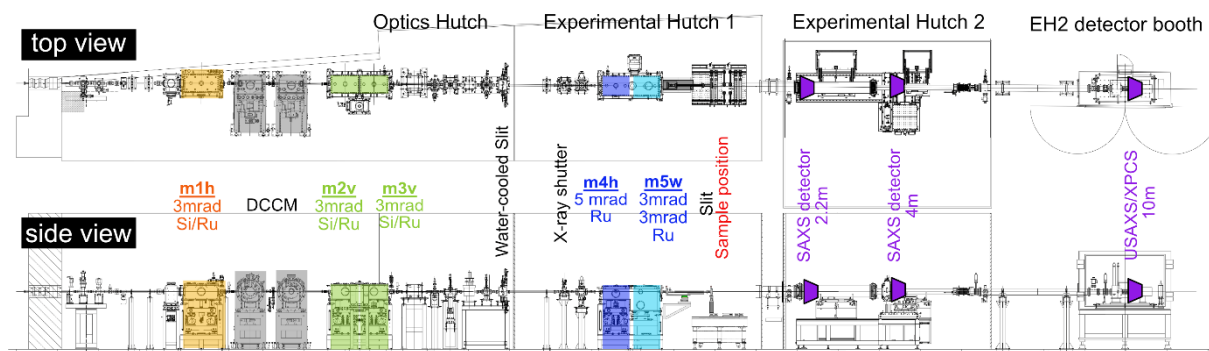


Fig. 1: Schematic diagram of the BL40XU layout after 2025B term.

monochromatic (8–20 keV) or monochromatic (8–18 keV) beams on the same sample position. The focusing mirrors can provide a beam spot at arbitrary sample positions, with a typical focus size of  $200\ \mu\text{m}$  (hor.)  $\times$   $10\ \mu\text{m}$  (ver.).

For monochromatic operation in the 8–12.4 keV range, a horizontal bendable mirror and a Wolter mirror [Fig. 2(a)] in EH1 can be inserted to deliver a microfocused beam to the sample position. At 12.4 keV, a beam focus of  $5\ \mu\text{m}$  (hor.)  $\times$   $2\ \mu\text{m}$  (ver.) with a flux of  $\sim 10^{12}$  photons/s is expected. Such microbeams will be available for WAXS and SAXS measurements with camera lengths of 2 and 4 m.

After the refurbishment, the sample position is fixed at 52 m from the source in EH1, and a vacuum path switcher [Fig. 2(b)] allows SAXS measurements with a camera length of 2, 4, or 10 m [Fig. 2(c)] at EH2 and the EH2 detector booth located downstream of EH2. For the SAXS detector, a PILATUS 1M (Dectris) will be primarily installed, and for XPCS at the 10 m configuration, the installation of CITIUS 840k (vacuum type) is planned. Figure 3 shows the expected accessible Q-range after the restart in 2025B considering the incident photon energy (e.g., 8, 12.4 or 18 keV), the radius of the beam stopper (2 mm), and the dimension of the detector used at each camera length.

The sample experimental stage has the dimensions of 800 mm height, 1360 mm along the beam axis, and 1000 mm transverse to it, and is equipped with sliding rails for slits, sample holders, and detector carriers. The X-ray beam travels parallel to the floor at a height of  $\sim 1400$  mm, passing 600 mm above the stage surface and 465 mm above the carrier surface. The bellows expansion joint has been installed downstream of

the Wolter mirror [Fig. 2(a)] as well as downstream of the sample, allowing the adjustment of the upstream slit position and the vacuum path depending on the surrounding sample environment. This configuration ensures compatibility with all user-provided equipment previously employed for SAXS experiments at BL40XU.

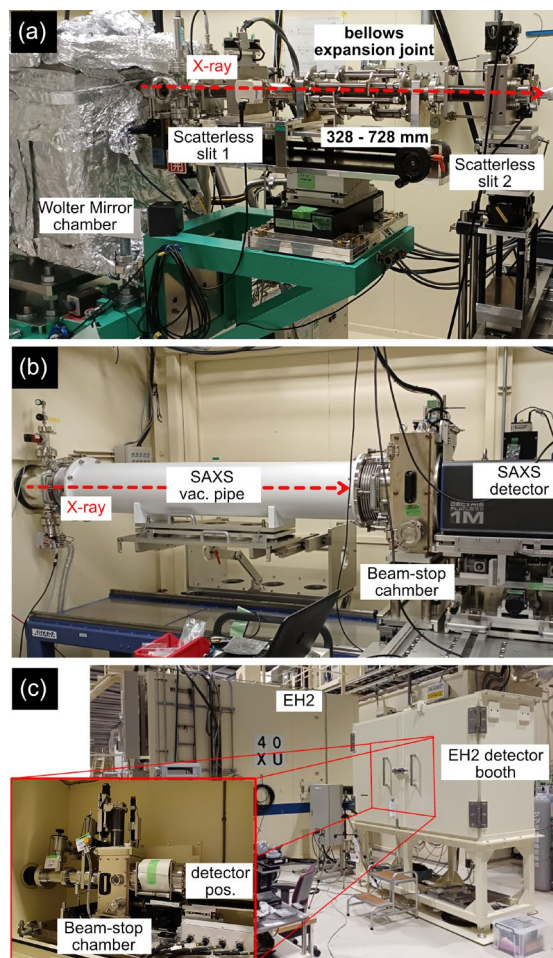


Fig. 2. (a) Bellows expansion joint installed downstream of Wolter mirror at EH1. (b) SAXS vacuum path set for 4 m SAXS at EH2. (c) EH2 detector booth located downstream of EH2.

In addition, the development of common-use sample environment equipment has been further advanced. A stopped-flow apparatus for the rapid mixing of solution samples (SFM-4000, BioLogic), a rheometer for applying shear to viscoelastic

samples (MCR302e, Anton Paar), and a temperature-controlled tensile stretching stage (TST350, Linkam) are available for users.

Beam tuning will be carried out during the 2025A operation period, followed by the stepwise preparation of control and data-processing software, in order to commence shared use in October 2025.

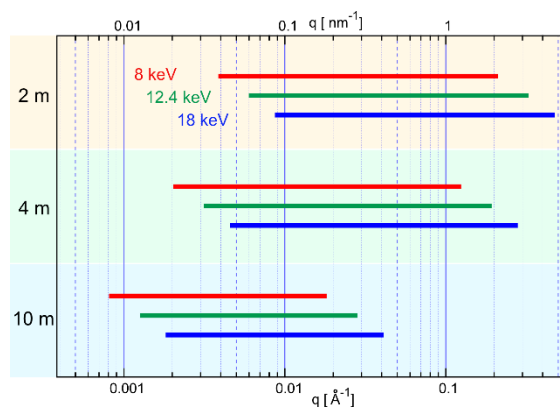


Fig. 3. Typical SAXS  $q$ -range in 2025B term at BL40XU SAXS-ID.

### 3. EH2: Introduction of a Heating Vacuum Drying System

We have installed a heating vacuum drying system, an apparatus that dries samples under vacuum with precise temperature control. This system enables the rapid and efficient removal of guest molecules from inclusion crystals, a process that was previously time-consuming. As a result, it is now possible to perform single-crystal X-ray structure analysis on the same crystal in both its guest-included state and its desolvated state (after guest removal). This provides an extremely powerful tool for elucidating the mechanisms of structural changes and function in inclusion compounds.

In parallel with these developments, the diffractometer previously installed in EH2 of BL40XU has been removed, and a new diffractometer is currently being installed and

commissioned at BL05XU.



Fig. 4. Heating vacuum drying system.

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References:

- [1] Inoue, K. (2001) *SPring-8 Annual Report 2000*, 82–83.