Development of SAXS Apparatus Equipped at Hyogo Prefectural Beamline BL08B2

Hyogo prefectural beamline, **BL08B2**, has been constructed for industrial research [1]. The experimental hutches have been designed for XAFS, small-angle X-ray scattering (SAXS), monochromatic X-ray topography, and high-resolution powder X-ray diffraction measurements, which are utilized in key industries such as the semiconductor, catalyst, pharmaceutical and polymer industries.

SAXS has been particularly utilized for the study of hard and soft condensed materials in many industrial applications to survey the variation in density or the distribution of dispersed particles in nanocomposite materials with the aim of revealing the relation between the function or performance of the material and the nano-order structure (morphology and/or distribution). To satisfy these demands, standard SAXS and ultra-SAXS (U-SAXS) apparatuses have been installed in BL08B2 experimental hutch, and the system is now open for such industrial research.

The beamline optics was designed after consultation with reference to beamline BL01B1. A schematic view of the arrangement of the optics is shown in Fig. 1. Using a bending magnet source and a silicon double-crystal monochromator, photon energy is tunable from 6 to 70 keV. For the SAXS experiments, an energy range from 8 to 20 keV is mostly used with the aim of overcoming the limitation of SAXS resolution. A Rh-coated double mirror has the role of rejecting the higher harmonics and focusing the beam both vertically and horizontally. The monochromator and the upstream mirror are used to achieve a high energy resolution ($dE/E < 10^{-4}$). Using another mirror equipped downstream of the monochromator, the X-ray beam of a single wavelength can be focused at the SAXS detector position.

In the SAXS configuration, the focusing mirror is a source of diffuse scattering. Therefore, 5 sets of quadrant slits have been arranged to eliminate parasitic scattering. The slits were designed with blade to improve the efficiency of the suppression of diffuse scattering.

Owing to the very long focal length of the focusing mirror (placed about 30 m from the light source) and the effective use of the guard slits, highly parallel and small X-ray beams are available for SAXS experiments. The typical beam formation conditions are as follows. The beam size at the sample position is 200 μm × 200 μm, and the beam flux is about 10^{10} photons/sec.

A SAXS camera for BL08B2 (KOHZU and Daitoseiki, as shown in Fig. 2) has been designed to cover the wide range of surveying scales. The sample-to-detector distance (camera length) can be varied from 300 to 6000 mm depending on the SAXS resolution. A mechanical adjustment system consisting of several pre-aligned vacuum tubes was realized using long guide rails. The system enables

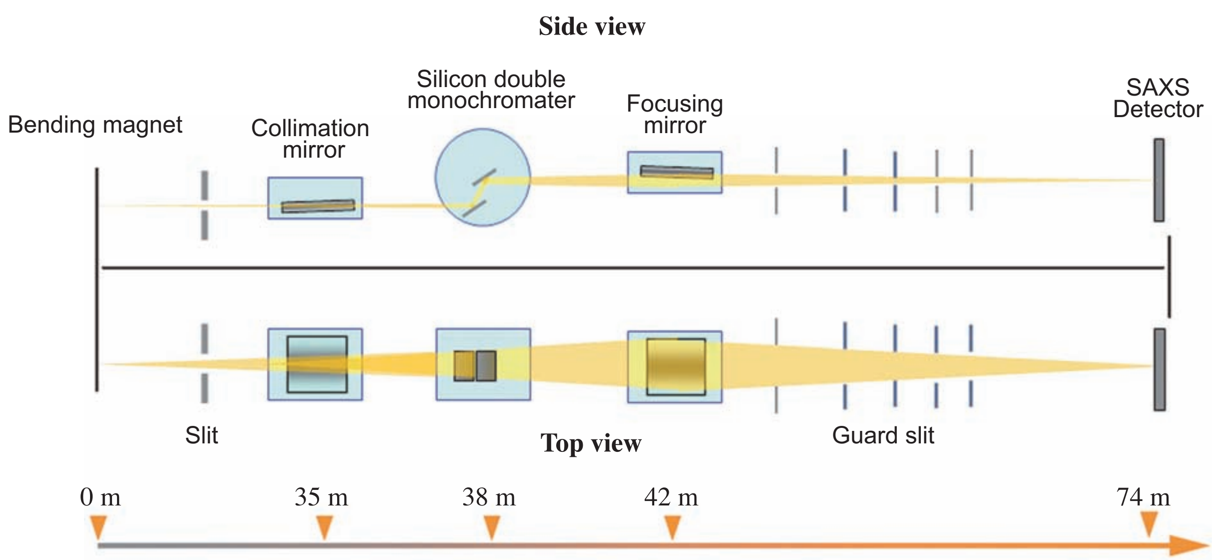

Fig. 1. Schematic view of BL08B2 optics.

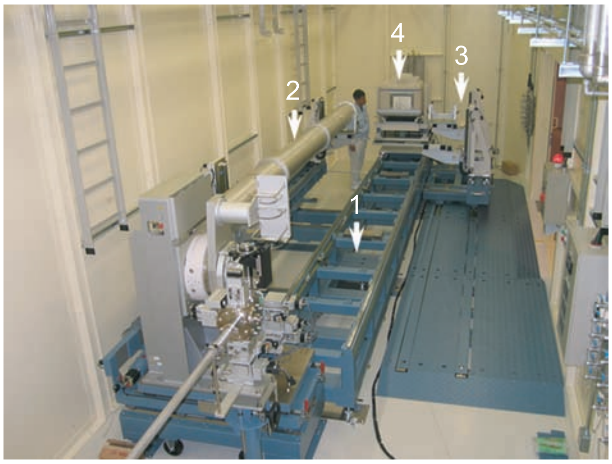

Fig. 2. Photograph of the SAXS camera in BL08B2 experimental hutch 2: (1) guide rail, (2) long vacuum tube (6 m camera length), (3) carrier stage for middle- or short-length vacuum path, (4) SAXS detector.

us to furnish any tube between the sample and the SAXS detector depending upon the desired q value (Fig. 3). Sample stages are also loaded on the same guide rail to allow fine adjustment of the camera length. The sample can be set on the stages under a vacuum or ambient condition, and both transmitted and total-reflection (grazing incident) X-ray configurations are possible for the purpose of measuring bulk, liquid, and thin films. For *in situ* experiments, large sample preparation systems such as a high-shear mixer can be used.

Two types of two-dimensional detectors are available for signal recording. One is an imaging plate system (Rigaku R-AXIS IV++) with a detecting area of 300 mm × 300 mm and high space resolution (100 μm). A 14-bit cooled CCD camera combined with an X-ray image intensifier (4 inch diameter) allows us to measure high-speed phenomena (37 msec/frame) with a space resolution of about 90 μm.

To meet the demand for higher resolution SAXS experiments (U-SAXS), a special configuration with long camera length ranging from 15,000 to 17,000 mm, is available using two experimental hutches simultaneously.

We have evaluated the SAXS resolution, the q range of the standard SAXS, and that of U-SAXS at the BL08B2 SAXS station using some standard samples such as collagen, polystyrene latex, and silica particles in solution. For each sample measurement, the SAXS camera length was adjusted according to the sample size. The scattering profile for silica particles is shown in Fig. 4 and the performance of the q resolution is shown in Table 1.

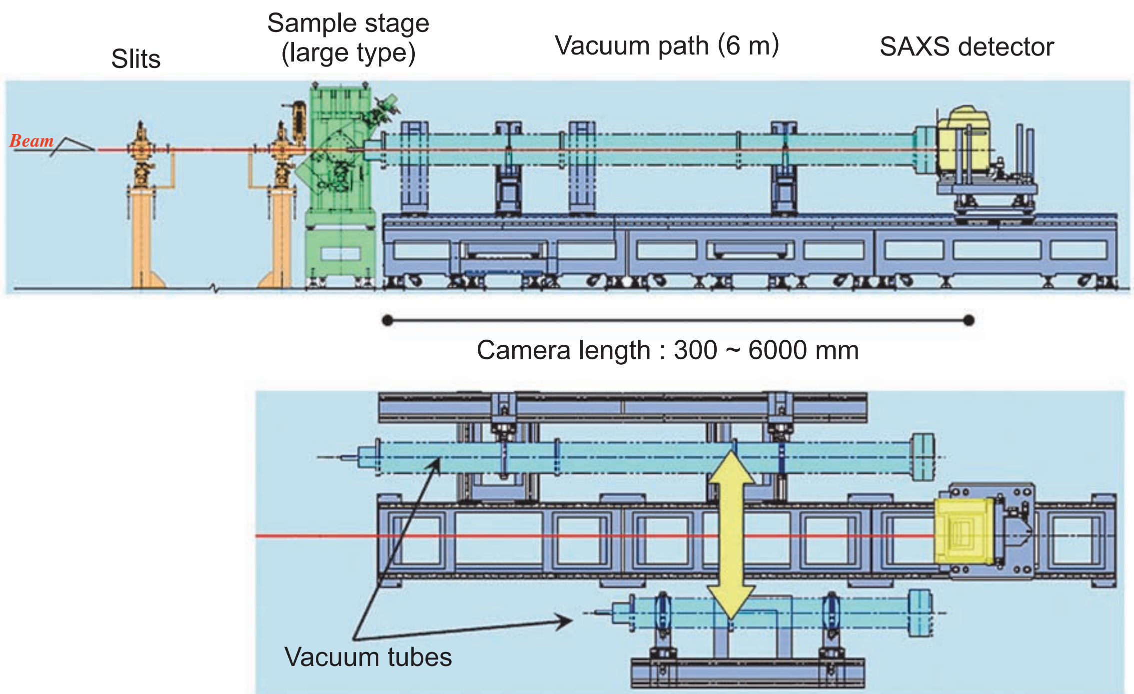

Fig. 3. Top view of guide rail system in BL08B2 SAXS camera.

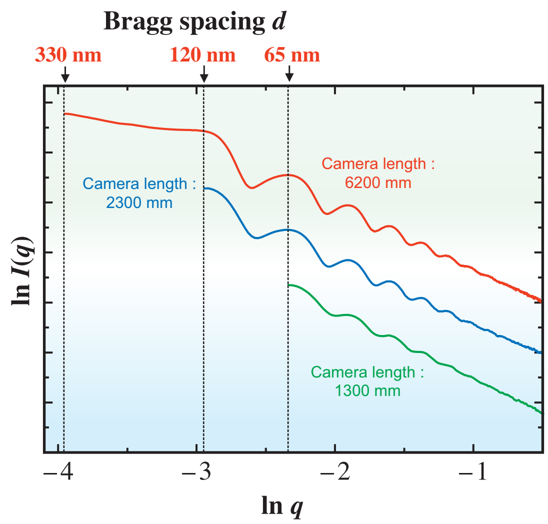

Fig. 4. Scattering profiles obtained from SiO_2 particles. The measurements were performed using a wavelength of 0.15 nm and the exposure time was 1.0 sec.

The results reveal that the performance of BL08B2 SAXS allow the observation of several hundred nm in a real space.

To perform valuable SAXS measurements of dispersion in solution, special sample cells are now being prepared, that can apply a shear stress to solution samples and induce the collapse of cluster structures.

For nanocomposite material applications, some experiments are planned to evaluate the dynamics of material structures such as the structural and morphological changes in materials caused by a catalyst and the simultaneous measurement of X-ray absorption fine structure (XAFS) and SAXS by the tuning and quick scanning of the photon energy near the absorption edge of the target element. This will enable us to realize the time-resolved observation of nanoscale structures.

Table 1. *q* range table of each camera length with IP detector

Camera length (mm)	*q* range (nm^{-1}) wave length = 0.15 nm	*d* spacing (nm)
500	0.260 ~ 11.4	0.55 ~ 24.2
1300	0.0966 ~ 4.30	1.46 ~ 65.0
2300	0.0527 ~ 1.50	4.18 ~ 119
6200	0.0189 ~ 0.963	6.52 ~ 332
15600	0.0081 ~ 0.246	25.5 ~ 781

These *q* range were estimated from the SAXS profile of standard samples, PS latex or SiO_2 particles.

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References

[1] *K. Yokoyama, T. Miyashita, S. Hiyama, S. Nose, M. Komatsu, Y. Urushihara, L. Li, S. Takeda, S. Kuwamoto, K. Nakamae and J. Matsui: in preparation.*