Upgrade of the small angle X-ray scattering beamlines at the Photon Factory

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Abstract. BL-10C and BL-15A at the Photon Factory, which became operational in 1982, are some of the oldest small angle X-ray scattering beamlines in the world. Recently, both beamlines were upgraded for two-dimensional (2D) SAXS-WAXS experiments. A wide-area imaging plate (IP) detector and a fast-readout flat panel (FP) detector were installed at BL-10C and BL-15A, respectively. Preliminary experiments of both systems showed promising results.

1. Introduction

Small angle X-ray scattering (SAXS) provides information on the structure of matter on the nanometer to micrometer scales. Thus, for years it has been commonplace to investigate complicated hierarchical structures like soft matters and biological systems [1]. The Photon Factory (PF) operates two SAXS beamlines, BL-10C and BL-15A, and partly uses BL-9C for SAXS. Currently, beamtime management and beamline maintenance of BL-10C and BL-15A are performed in cooperation with user groups (Chairpersons are Prof. Yoji Inoko of Osaka Univ. and Prof. Mitsuhiro Hirai of Gunma Univ.). BL-10C and BL-15A were initially installed to provide high X-ray flux for kinetic solution scattering experiments and time resolved muscle fiber diffraction [2]. Both beamlines have been operating for about 30 years, and the last major upgrades were done in the 1990s; thus, much of the hardware and software has since become obsolete.

Soft matter shows a hierarchical structure from the sub-nm to several hundreds µm scale. Hence, the ability to measure scattering over a wide scale is of great importance in the structural analysis of soft matter [3]. To date, we have had many papers on WAXS and SAXS experiments at the both beamlines, but they have been performed separately by assuming the reproducibility of the
experimental conditions. However, irradiation, thermal, or mechanical treatment can easily damage soft or biological samples. Although several users simultaneously performed SAXS-WAXS measurements at the PF, most were limited to one-dimensional measurements using a position-sensitive proportional counter (PSPC). To implement 2D-SAXS-WAXS measurement systems, we recently installed an IP detector system at BL-10C and a FP detector at BL-15A. Herein we describe the development and the preliminary experiment of the 2D-SAXS-WAXS measurement system at each beamline.

2. Wide-area imaging plate system at BL-10C

BL-10C was designed and constructed mainly to investigate non-crystalline materials such as aqueous solutions of biological macromolecules and synthetic polymers. Due to the low sample concentration and low-scattering contrast of such samples, a low-noise signal detection system is required, especially for higher-angle scattering data. For years, only a one-dimensional PSPC (RIGAKU) was used as a detector. However, an IP detector (R-AXIS 7, RIGAKU) equipped with a synchronized X-ray shutter was recently installed to measure wide 2D scattering images (figure. 1).

Preliminary experiments were performed using horse spleen apoferritin and a wavelength ($\lambda$) of 0.1488 nm (figure. 2). The camera distance and the exposure time were 1980 mm and 600 sec, respectively. The temperature of the cell, which had a 1 mm beam path and a pair of 20 μm quartz windows, was kept constant at 24°C using a metallic cell holder through which water at a constant temperature was circulated. The standard cell volume was 45 μl ($3 \times 15$ $\text{mm}^2$). Variations in the scattering data for different solutions were corrected by monitoring the beam intensities with an ionization chamber placed in front of the sample cell holder. Two-dimensional scattering data were corrected.

![Figure 1](image1.png)

Figure 1. Long camera setting with R-AXIS 7 at BL-10C. The camera distance is about 2000 mm (The middle setting: about 900 mm.). The X-ray beam passes from left to right.

![Figure 2](image2.png)

Figure 2. (a) Typical 2D pattern and (b) Scattering curve ($q$ vs. $\ln I$) of horse spleen apoferritin. Cp is protein concentration and S-D is sample-to-detector distance.
circle-averaged about the beam center. The obtained signals were corrected for both solvent scattering and sample concentration to yield the net scattering intensity \( I(q) \). The \( q \)-value was calibrated by the diffraction pattern of the dried chicken collagen, and the observed \( q \)-range was between \( \sim 0.07 \) and \( 2.5 \) nm\(^{-1}\).

Figure 2b shows a typical scattering curve for the horse spleen apoferritin. Apoferritin is a hollow sphere molecule composed of 24 subunits and each subunit has \( \sim 19,800 \) molecular weight [4]. The scattering pattern indicates peaks at \( q = 0.8, 1.4, \) and about \( 1.9 \) nm\(^{-1}\), and the resolution is sufficient for detailed structural characterization of the 24-mer molecule. This result reveals the benefit of the new IP detector.

3. 2D-SAXS-WAXS system at BL-15A
BL-15A was originally built about 30 years ago for time-resolved studies of small-angle diffraction from contracting muscles [2]. The optics consists of a bent flat mirror and a triangle bent asymmetric-cut monochromator for vertical and horizontal focusing, respectively. It can produce a point focus spot with a high spatial resolution and the intensity necessary for 2D-SAXS-WAXS experiments. Due to such a good point focus beam, the beamline is still widely used for 2D-SAXS and 2D-WAXS. However, the combined measurements of SAXS-WAXS were limited to one-dimensional measurements; thus, we installed a FP detector (Hamamatsu C9728DK-10 [5]) as a WAXS detector for 2D-SAXS-WAXS. Figure 3 depicts the 2D-SAXS-WAXS system. For 2D-SAXS image detection, several detectors are available: two types of CCD detectors (Hamamatsu C4880-10 and C7300) with an X-ray image intensifier [6], pixel array detector (Dectris PILATUS 100K), and off-line IPs (Fujifilm BAS2500).

Figure 4 shows the 2D-SAXS-WAXS patterns for dry chicken collagen. The PILATUS 100K was used for the SAXS detector. The X-ray wavelength and the exposure time were 0.15 nm and 10 sec, respectively. The camera distances of the SAXS and WAXS were approximately 2,000 mm and 50 mm, respectively. Electric pulses generated by an electronic stimulator (Nihon Koden SEN-7103) were used to synchronize the SAXS and WAXS detectors. The SAXS-WAXS images demonstrate that the first order peak is clearly separated from the beam stop and that higher order scattering is observed simultaneously. Although the active area is relatively small, the SAXS-WAXS data can be observed by rotating the sample.

Figure 3. Schematic illustration and photograph of the 2D-SAXS-WAXS simultaneous measurement system at BL-15A in the PF.
4. Further upgrades

Herein we report the successful developments of the 2D-SAXS-WAXS systems at BL-10C and BL-15A at the PF. Additionally, we are pursuing further upgrade programs for both beamlines. The PSPC electronics will be changed from CAMAC to a high-speed data acquisition system based on Si-TCP technology [7]. A GI-SAXS data collection system has been implemented at BL-15A for common use. Moreover, we have proposed a new high-brilliance SAXS beamline at the BL-15 site using a short-gap undulator. This improved BL-15 should be capable of structurally analyzing heterogeneous materials, and then some of the current BL-15A activities will be transferred to BL-6A. BL-6A was closed in the spring of 2010 and the beamline geometry is similar to the current BL-15A. Hence, the current BL-15A settings can be transferred to BL-6A. Then, two bending magnet beamlines (BL-6A and BL-10C) and one undulator beamline (new BL-15A) will be dedicated to the extensive SAXS activities at the PF.

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