Refurbishing of Small-Angle X-ray Scattering Beamline, BL-6A at the Photon Factory

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Abstract. BL-6A at the Photon Factory is new small-angle X-ray scattering beamline. The light source makes use of a bending magnet, and the available wavelength, monochromatized by a Johann type crystal monochromator, is fixed to 1.5 Å. Various kinds of detectors for SAXS and WAXS experiments are installed in BL-6A, and a SAXS/WAXS simultaneous measurement system was also constructed. This beamline is maintained to measure various samples, such as protein solutions, fibers, liquid crystals as well as solid state materials.

1. Introduction

Small-angle X-ray scattering (SAXS) is an established technique which makes use of the scattering intensity detected in the angle, 2θ < 10°. This technique is widely used in various fields from hard to soft matter, including the analysis of biomolecules, allowing the structural properties down to nanoscale order to be analyzed. Furthermore, the data in the wide-angle region of 2θ > 10° can also be measured, and SAXS/WAXS (Wide-angle X-ray scattering) experiments may be executed simultaneously. For example, one may obtain structural information on the effect of crystallization measured in a wide-angle region and the change of molecular size measured in a small-angle region, on the same time scale. Two SAXS beamlines, BL-10C and BL-15A [1] were available at the Photon Factory (PF). Both beamlines, constructed in 1982, are among some of the oldest SAXS beamlines in the world and have been used for around 30 years, consistently producing results of significance. We started upgrading these beamlines recently and have previously reported several improvements [2]. However a high brilliance beam generated by an insertion device is required in order to conduct up-to-date SAXS experiments over a broad range of spatio-temporal scales.

Four new short straight sections were constructed for insertion devices when the PF storage ring was upgraded in 2005, and the BL-15 site was one such beneficiary. With the aim of constructing a new short-gap undulator beamline for SAXS and XAFS activities at this site, we moved BL-15A to the BL-6A site in the summer of 2011. Almost all the experimental system was moved from BL-15A to BL-6A, and was installed using essentially the same previously utilized layout. However, we replaced old components and systems with new ones during this transfer in order to improve beamline safety and the ease of operation. New BL-6A was opened from October, 2011 after an initial tuning and commissioning period.
2. Beamline Layout of BL-6A

The schematic view of BL-6A is shown in Figure 1. The beam generated by a bending magnet is firstly focused in the vertical direction by a 110-cm-long non-coated bent ULE mirror after cutting into 2.3 mm (ver.) × 17.5 mm (hor.) with the first aperture slit. The glancing angle of this mirror is set to 3.0 mrad to eliminate higher harmonics. The vertically-focused beam is monochromatized to 1.5 Å and focused in the horizontal direction using a Johann type crystal monochromator, with a Ge (111) asymmetrically cut (8 degree) crystal. The second slit was used for setting the beam size at the sample position installed just behind the monochrometer, with a third slit used as a scattering guard to eliminate parasitic scattering just before the sample position on the diffractometer.

![Figure 1. Schematic view of BL-6A. Length is displayed in cm and indicates the distance from the light source.](image)

The camera length can be changed from 450 to 2400 mm on the diffractometer (Figure 2), and the position of the sample and the beam stopper can be also optimized using an x-y motorized stage. The guard slit was replaced by a newer one (Scatterless slits, Xenocs) because of age related deterioration. Two types of CCD detectors, C4880-10 and C7300 (Hamamatsu photonics), were installed for SAXS experiments. The CCDs record the scattering intensity amplified with an X-ray image intensifier of two sizes, 6 inch and 9 inch (Hamamatsu Photonics), respectively. A new detector stage was installed to control the SAXS detector position automatically. A flat-panel sensor, C9728DK-10 (Hamamatsu photonics), was also installed for WAXS experiments, and set just behind a sample, controlled with a motorized stage.

A system for simultaneous SAXS/WAXS experiments was constructed with a digital delay pulse generator; Model 500D (Berkeley Nucleonics Corporation). The PF standard control frame work, STARS [4] was applied to BL-6A in order to control all the motorized stages and a picoammeter remotely. Several new GUI programs were developed and installed for these components. The incident beam intensity was measured by a micro-ion chamber (MIC-R20, Repic) just before the sample. Moreover, a chamber can also be placed just behind a sample when measuring sample absorption.

3. Cleaning of the Mirror and Evaluation of the Surface Property after the Cleaning

A mirror was previously installed in the white beam section as shown in Figure 1. Over the course of many years of use, not only was the surface of the mirror polluted quite severely with carbon residue but also the color of the substrate material was changed to brown because of radiation damage. We transferred this mirror to SPring-8 for evaluation and to clean the surface. The surface was firstly washed with purified water to remove the carbon paste mixed with oil (Figure 2(a-b)). UV/Ozone ashing was performed with an UV/Ozone Asher (UA1000, Hitach) to remove the adhered carbon [5] after the washing. Although the carbon almost disappeared after ashing, the color of ULE did not change from brown (Figure 2(c-d)). The surface roughness, rms was measured by a 3D optical surface

![Figure 2. Photo of the experimental hutch.](image)
profiler (NewView 7300, Zygo). The rms values after cleaning were considerably worse than that at the time of purchase (Table 1).

|                          | rms (nm) |
|--------------------------|----------|
| At the time of purchase  | 0.2      |
| Inside of the footprint* | 7.1      |
| Outside of the footprint*| 1.2      |

*The surface profile was measured at three places: at the center and at either end of the mirror. Shown are the average values of the three.

4. Results of Beamline Tuning

4.1. Comparison of the beam size at the focal point found using Raytrace and measurement

We optimized the optics by monitoring the beam profile at focus with a flat-panel sensor (C10013SK, Hamamatsu Photonics). Although this sensor was originally covered with a 2.0 mm-thick layer of carbon fiber, we also used a 1 mm aluminum sheet as an attenuator to weaken the beam intensity further. Raytracing was calculated with the program SHADOW [3], to compare the measured result with the theoretical value. For the horizontal direction, the measured beam size was comparable to that calculated by the program. On the other hand, the measured beam size in the vertical direction seemed to be slightly larger than the raytraced value. It is reasonable to suppose that this result was caused by degradation of the surface of the mirror.

|                          | Raytrace | Measurement |
|--------------------------|----------|-------------|
| Beam size (mm, FWHM)     |          |             |
| Vertical                 | 0.188    | 0.245       |
| Horizontal               | 0.443    | 0.498       |
| Beam divergence (mrad)    |          |             |
| Vertical                 | 0.046    |             |
| Horizontal               | 0.963    |             |

4.2. Reflectance of the mirror and the photon flux at the sample position

The beam reflected by the mirror can be observed at the focal point only after passing through the monochromator. Therefore, we estimated the contamination ratio of higher-order X-rays at 1.5 Å to
evaluate the reflectance of the mirror correctly. The ratio of contaminated X-rays was estimated to be 0.07\% and thus we calculated the reflectance based on the beam intensity measured at the focal point using an ion chamber (S-1329A, OHYO KOKEN KOGYO) (Table 3). Unfortunately, since the reflectance before the ashing was not measured, we could not confirm the effect of the ashing. The observed reflectance was almost identical to the calculated value on the basis of the average rms inside of the footprint of the beam.

| Reflectance (%)                                      |
|------------------------------------------------------|
| Calculated using rms at the time of purchase         | 95.7 |
| Calculated using average rms after the ashing         | 90.7 |
| Measurement                                           | 89.2 |

The photon flux at the sample position was estimated using an ion chamber, S-1329A, filled with nitrogen gas. The flux behind the monochromator, in the fully open and with the size-definition slit set to 0.6 × 0.6 mm², was found to be \(1.0 \times 10^{12}\) phs/sec and \(3.6 \times 10^{10}\) phs/sec, respectively. Figure 4 shows the data diffracted by dry chicken collagen. The first order peak recorded is clearly separated from the beam stopper, indicating small-angle resolution of \(~800\) Å.

5. Conclusion and Future Plan
BL-6A is newly constructed SAXS beamline, updating and replacing the old components of BL-15A. We plan to replace the detector with a hybrid pixel detector for both SAXS and WAXS experiments. PILATUS 300K and 100K will be installed in October 2012 as SAXS and WAXS detectors, respectively, with control GUI software based on the originally developed PF standard software. It is expected such an upgrade for the beamline will enable further successful SAXS activities.

Acknowledgments
We would like to thank Dr. Haruhiko Ohashi and all the members of Light source and Optics Division in JASRI/SPring-8 for their valuable support and guidance during the mirror cleaning.

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Figure 4. Diffraction pattern of dry chicken collagen recorded by a 6 inch X-ray image intensified CCD (C4880). The size of the beam stopper is 6 mm. The bottom graph shows the profile along the dashed line.