1 Newly Developed Experimental Facilities

1-1 Overview

The Photon Factory (PF) started user operations in 1982, and has been providing valuable resources for X-ray research for more than 3,000 users annually from universities, industries, and government institutions. The PF has been carrying out a beamline refurbishment program since 2006, in which the main strategy is to concentrate investments on competent beamlines which use insertion devices as light sources. With the upgrade of the 2.5-GeV PF ring for lengthening the straight sections, the lengths of the long and medium straight sections have been increased so that state-of-the-art insertion devices can be installed to cover the vacuum ultraviolet and soft X-ray region. Four new straight sections have also been created to allow the installation of short period and small gap undulators (SGU) to supply well-focused hard X-rays. We have already constructed three HX beamlines at the short straight sections: BL-3 for materials science, and BL-1 and -17 for macromolecular crystallography. At the fourth straight section, we are constructing a new beamline for both small-angle X-ray scattering (SAXS) and X-ray absorption spectroscopy (XAFS/XRF) at BL-15. The details are described below.

Beamlines BL-13, -16, and -28 were originally constructed for sharing the photon beam between VSX and HX users by operating the insertion device in the undulator and multipole wiggler mode. Among the three beamlines, BL-28 was renewed first as a high-performance spectroscopic beamline dedicated to photoelectron spectroscopy in the VSX region in 2006. The second branch of BL-28 has been open as a free port. BL-16 was completely upgraded as a soft X-ray spectroscopic beamline with fast polarization switching capabilities. Two tandem APPLE-II type undulators are installed to generate different polarizations such as right- and left-hand circular polarizations, and the polarization is switched by modulating the electron orbit through the undulators, as reported in the previous activity report [1,2]. BL-13 was recently reconstructed as a VSX spectroscopic beamline for studying organic thin films absorbed on well-defined surfaces using angle-resolved photoelectron spectroscopy and X-ray absorption spectroscopy; the detailed performance is also described below.

The VSX beamline refurbishment program has required the replacement of the insertion devices at BL-13 and BL-28, which were constructed in the 1980s for supplying photon beams in the VSX and HX regions. These old insertion devices do not always meet the needs of VSX users and are not adequate for utilizing the full performance of the newly constructed beamlines. Furthermore, during the test operation of the PF ring and the beamlines in June 2011, after the Great East Japan Earthquake on March 11, 2011, we found some troubles caused by earthquake damage to these old insertion devices. Therefore, these two old undulators should be replaced by appropriate new ones for the renewed beamlines. BL-2 was the first undulator based VSX beamline constructed in the early 1980s, and has been operated for over 25 years. The BL-2 undulator is 3.6-m long, while the length of the straight section is 9 m following the upgrade in 2005. We have decided to add a 16-cm period undulator, and to construct a new beamline in collaboration with Hitachi, Ltd. The new beamline optics consist of a grazing-incidence grating monochromator and a double-crystal monochromator, and will supply VUV and soft X-rays of 30–4000 eV. Construction of the new beamline started in February 2013, and the new undulator will be installed in the spring of 2014.

The Slow Positron Facility (SPF) is affiliated with the PF, and three stations are currently available: (i) one for the photodetachment of the positronium negative ion (Ps-) and its application to the production of an energy-tunable positronium beam, (ii) one for positronium time-of-flight (Ps-TOF) spectroscopy, and (iii) one for reflection high-energy positron diffraction (RHEPD).

REFERENCES

[1] *Photon Factory Activity Report 2010* **#28**, (2012).
[2] *Photon Factory Activity Report 2011* **#29**, (2013).

1-2 Construction of New High-Brilliance XAFS/XRF/SAXS Beamline, BL-15A

The last SGU section, BL-15A, is being built for SAXS and XAFS/XRF activities. The SAXS scientific programs at BL-15A are structural studies of functional membranes, time-resolved X-ray scattering and large hierarchical structure analysis, using highly collimated intense X-ray photons derived from the SGU#15. In particular, grazing incidence SAXS (GI-SAXS) using vertically small-size softer beams in the range 2.1–3.0 keV will help to control the depth of the membrane structure analysis and reduce the roughness defects of an imperfect membrane. For the XAFS/XRF studies, the semi-micro focus beam available in a wide range of photon energies is used for analyzing the local structures of the elements and valence of inhomogeneous samples in the fields of environmental science and new energy source science. The 2.1-keV X-rays provide access to the absorption edges of phosphor and sulfur, which are very important targets for those fields. The combination of SAXS and XAFS yields diverse structural information from fine atomic structures to low and medium resolution. It can be beneficial to build these instruments as two stations on the same beamline. BL-15A is oriented toward joint advanced studies by using the two techniques.

We have completed the beamline design of BL-15A (Fig. 1). The SGU#15 covers the wide continuous energy range required by XAFS/XRF studies; the undulator has a periodic length of 17.6 mm and the number of periods is 27. The minimum gap is 4.0 mm, giving a Kmax of 1.61. We will use the 1st to 9th harmonics including the 2nd harmonic to cover the large energy gap between the 1st and 3rd harmonics. Synchronization between the ID gap and energy setting of a monochromator will be developed in the control system for quick energy scanning, by a configuration similar to the one proposed at NSLS-II [1]. Encoder reading of the updated ID gap is directly fed to the monochromator controller. The closed loop adjustments of the monochromator axes follow the gap in real time. The main optics are a horizontal collimating mirror, a liquid nitrogen cooling double-crystal monochromator, a vertical focusing mirror and a pair of asymmetrically horizontal focusing mirrors which employ a secondary source. These deliver a stable, semi-micro focus or collimated beam. The monochromator crystals are Si(111), and their lowest energy is limited to 2.1 keV. The two horizontal focusing mirrors are bimorph ones [2]. The secondary source aperture is controlled with a set of slits. Tuning the mirror surfaces and setting the size of the slits adjusts the size of the beam at the sample position. A vertical double-mirror system is also installed in the last section in order to eliminate higher-order reflections. The experimental hutch is separated into two tandem stations because of the very different beam requirements of SAXS and XAFS/XRF experiments. The upstream and downstream stations are BL-15A1 for the XAFS/XRF activity and BL-15A2 for the SAXS activity, respectively. The second horizontal focusing mirror has double-mirror surfaces that provide a highly focused and collimated X-ray beam for A1 and A2 stations, respectively. The demagnification ratios for XAFS/XRF/XRD and SAXS are approximately 16.7:1 and 1:2 (or 1:1), respectively. The front-end is retrofitted with several differential pumps for windowless operation. Windowless operation allows the beamline to utilize softer X-ray beams of up to 2.1 keV.

In order to check the beam performance, ray-tracing simulations were performed using the programs XOP and SHADOW [3,4]. Table 1 shows some results of these calculations at different focal points at different energies. Thanks to the high-brilliance light source, the windowless beamline design and the double surface bimorph mirror, sufficient beam performance for both techniques can be obtained in the entire energy range of interest. The photon flux of the 2nd harmonic range, which is used only for XAFS studies, is almost the same as those in the adjacent range.

We started the construction just after the end of the PF-ring operation of FY2012 (Fig. 2). The first beam will be delivered in October 2013. After the commissioning of the beamline components and the experimental apparatus, we will start the user beamline operation at the end of FY2013.

REFERENCES

[1] T. Tanabe, D. Chabot, O. V. Chubar, L. R. Dalesio, D. A. Harder and J. Rank, *Proc. of ICALEPCS* 776 (2009).

[2] R. Signorato, O. Hignette and J. Goulon, *J. Synchrotron Rad.* **5**, 797 (1998).

[3] M. Sanchez del Rio, N. Canestrari, F. Jiang and F. Cerrina, *J. Synchrotron Rad.* **18**, 708 (2011).

[4] M. Sanchez del Rio and R. J. Dejus, *AIP Conf. Proc.* **705**, 784 (2004).

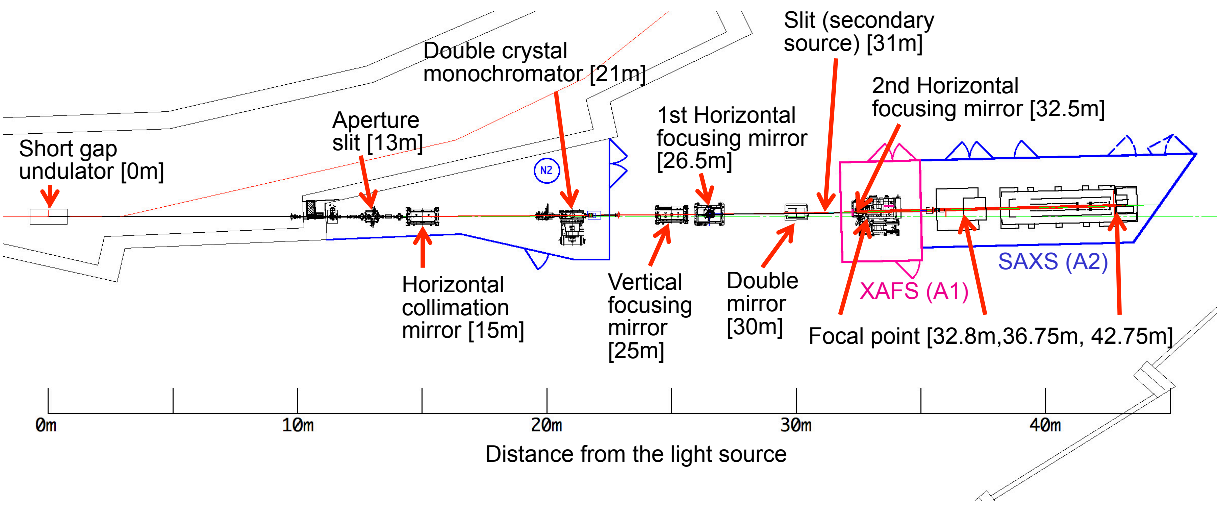

Figure 1: Layout of new BL-15A.

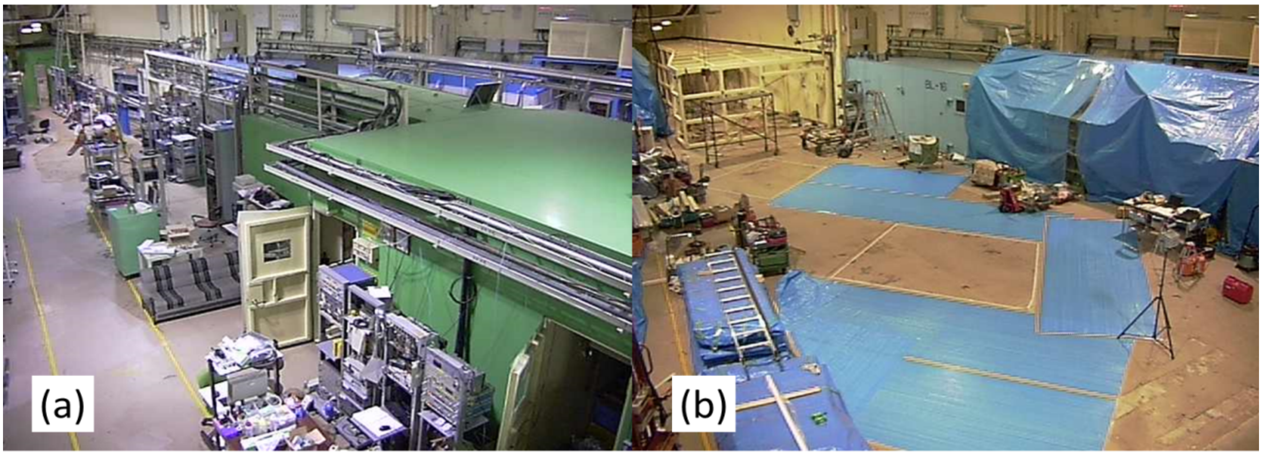

Figure 2: Photos of BL-15 site (a) before the end of the PF-ring operation of FY2012 and (b) at the end of FY2012.

Table 1: Ray-tracing simulation using the programs SHADOW and XOP.

Energy (ev)	2101	2800	4406	7344	10281	13218
Harmonics	1st	2nd	3rd	5th	7th	9th
K value	1.10	1.61	1.61	1.61	1.61	1.61
XAFS at 32.8 m [secondary source size: 0.1 mm (H) x 0.5 mm (V)]						
Beam Size (mm)	0.017 (H) x 0.009 (V)					
Photon Flux (phs/s)	1.3x10^{11}	1.2x10^{11}	6.3x10^{11}	4.7x10^{11}	2.4x10^{11}	1.7x10^{11}
SAXS at 42.75 m [secondary source size: 0.05 mm (H) x 3.0 mm (V)]						
Beam Size (mm)	0.339 (H) x 0.018 (V)					
Divergence (mrad)	0.058 (H) x 0.109 (V)					
Photon Flux (phs/s)	9.1x10^{10}		3.0x10^{11}	2.3x10^{11}	1.5x10^{11}	9.4x10^{10}
GI-SAXS at 36.75 m [secondary source size: 0.1 mm (H) x 3.0 mm (V)]						
Beam Size (mm)	0.275 (H) x 0.012 (V)					
Divergence (mrad)	0.157 (H) x 0.160 (V)					
Photon Flux (phs/s) w/o terminal window	1.9x10^{11} 6.1x10^{11}		6.1x10^{11} 6.8x10^{11}	4.5x10^{11}	2.7x10^{11}	2.0x10^{11}

1-3 Construction of BL-13B, Optics for Photoelectron Spectroscopy

Optics for photoelectron spectroscopy for the study of surface chemistry (BL-13B, Fig. 3 and Fig. 4) were constructed in March 2012. BL-13B involves a plane mirror for suppressing higher harmonics with a grazing angle of 2° (Mp, Fig. 5), an exit slit, two post-focusing mirrors, and an apparatus to monitor photon intensity and resolution [1]. The upper, middle, and lower areas of Mp are coated with nickel, gold, and chrome, respectively. The calculated reflectivity of Mp is also shown in Fig. 5 for the grazing angle of 2° and p-polarization. Nickel- and chrome-coated mirrors will be used mainly for X-ray absorption measurements in the nitrogen and carbon K-edge regions, respectively. The photon energy region and photon energy resolution of BL-13B are almost the same as those of BL-13A (Fig. 6). BL-13B will be opened for users in October 2013.

REFERENCES

[1] A. Toyoshima, H. Tanaka, T. Kikuchi, K. Amemiya and K. Mase, *J. Vac. Soc. Jpn.* **54**, 580 (2011).
[2] A. Toyoshima, T. Kikuchi, H. Tanaka, K. Mase, K. Amemiya and K. Ozawa, *J. Phys.: Conf. Ser.* **425**, 152019 (2013).

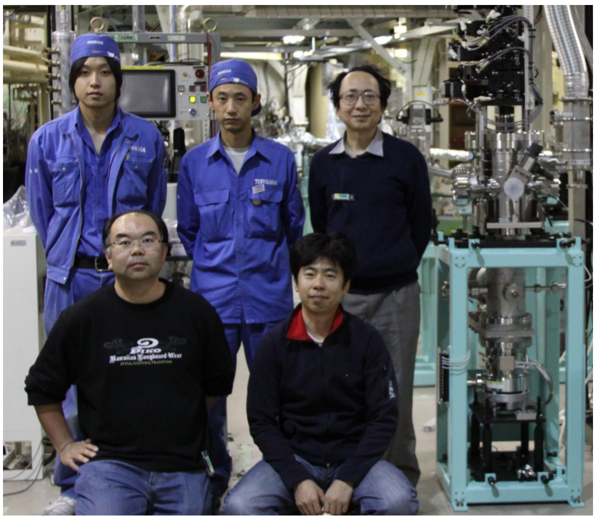

Figure 3: Photograph of BL-13B.

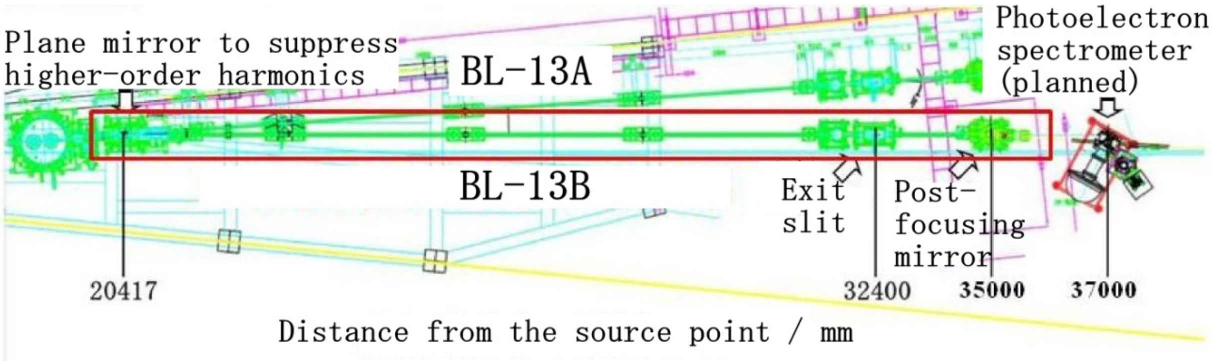

Figure 4: Floor layout of optics for photoelectron spectroscopy (BL-13B).

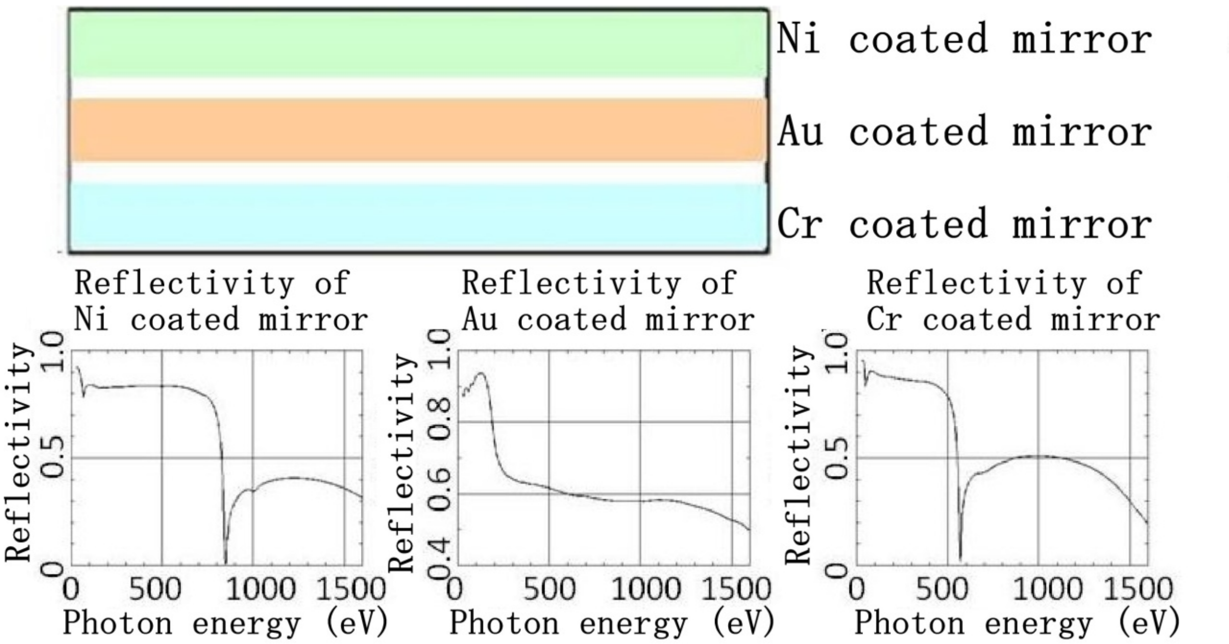

Figure 5: Plane mirror for suppressing higher harmonics (Mp), and its reflectivity for the grazing angle of 2° and *p*-polarization calculated using the web page of the Center for X-Ray Optics (*http://henke.lbl.gov/optical_constants/layer2.html*).

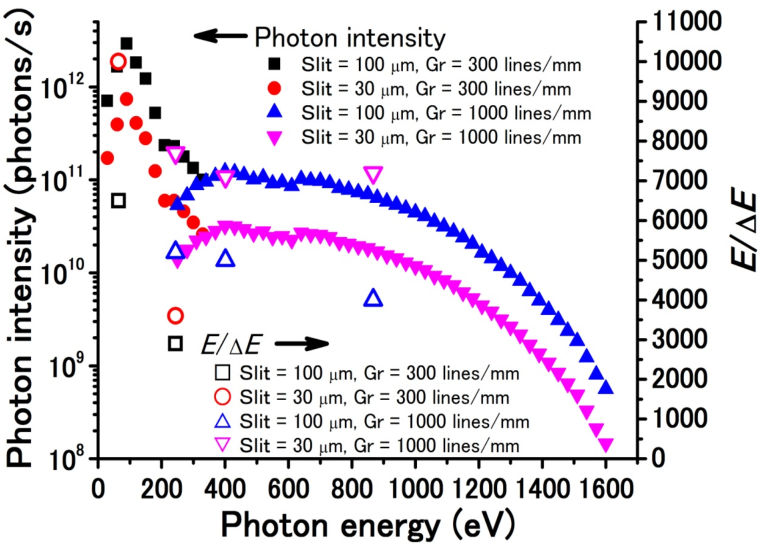

Figure 6: Typical photon intensity and photon energy resolution (E/ΔE) of BL-13A in the photon energy region of 30–1600 eV [2].

1-4 New Simultaneous SAXS/WAXD Measurement System Combining Two Pixel Detectors at BL-6A, and Improvement of Data Quality at BL-10C

BL-6A is used for the structural analysis of hard and soft materials studied by small-angle X-ray scattering. X-ray intensified CCD detectors and a flatpanel sensor have been installed and used in BL-6A for SAXS and WAXD simultaneous experiments in order to obtain structural information in both the nanoscale and the mesoscale. However, the dynamic ranges of these CCDs and the flatpanel sensor are quite narrow, perhaps around 10 bits, because of high noise level. Therefore, a new simultaneous SAXS/WAXD measurement system was constructed at BL-6A (Fig. 7), with the new installation of hybrid pixel detectors, PILATUS 300K and 100K, and an in-vacuum X-ray shutter which synchronizes with the detectors. Although the detectable area of these PILATUSs is somewhat small, they have high dynamic range, high-speed readout (7 and 2.3 msec) and quite a low noise level. Moreover, we redeveloped the measurement software and the image viewer of PILATUS with a GUI as standard software of the Photon Factory (Fig. 8). It has been used not only at SAXS beamlines but also at several XAFS beamlines.

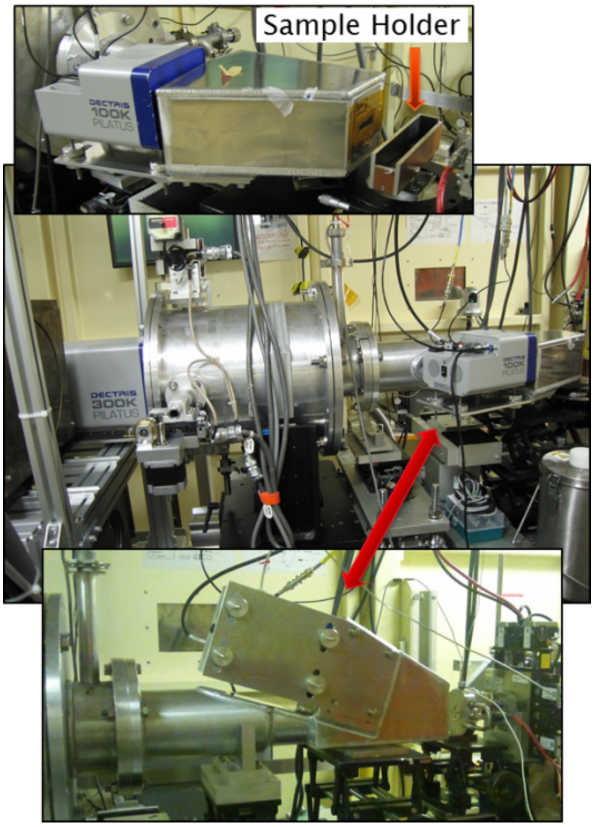

Figure 7: New SAXS/WAXD measurement system with two PILATUS detectors. The WAXD detector, PILATUS 100K, is fixed to the vacuum chamber, and the angle of this detector can be changed from the horizontal direction to the vertical direction.

BL-10C is also dedicated to small-angle X-ray scattering mainly for measuring solution samples such as biomolecule solutions. We aimed to improve the data quality of BL-10C in this fiscal year (Fig. 9). Although there was a long air section around the sample in the previous settings, we replaced the out-vacuum X-ray shutter and scattering guard slit to in-vacuum ones in the summer of 2012 in order to minimize the effects of air scattering and absorbance on the data. Moreover, we newly installed a pinhole as a scattering guard just before the sample. In addition to these installations, we also optimized the settings of the monochromator and the focusing mirror. As a result, the beam size at the focal point became smaller, and the beam stopper size also became smaller since the background level of image data decreased remarkably. Finally, the small angle resolution improved from 800 Å to 1100 Å at the camera length of 2 m.

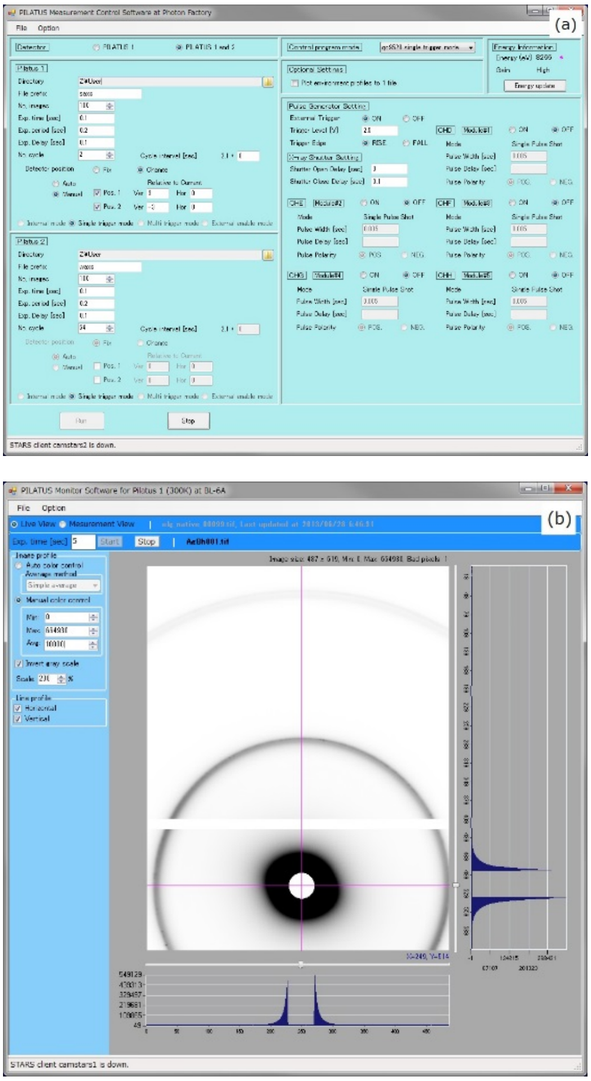

Figure 8: (a) The GUI measurement software of PILATUS. Users can collect data by using two PILATUSs, controlling an X-ray shutter and a pulse generator. (b) Image viewer of PILATUS. It has two modes: live view and measurement view. Live view is a video-like viewer mode, and measurement is executed repeatedly with the inputted exposure time. Measurement view is the automatic loader mode; the latest image is displayed automatically every second.

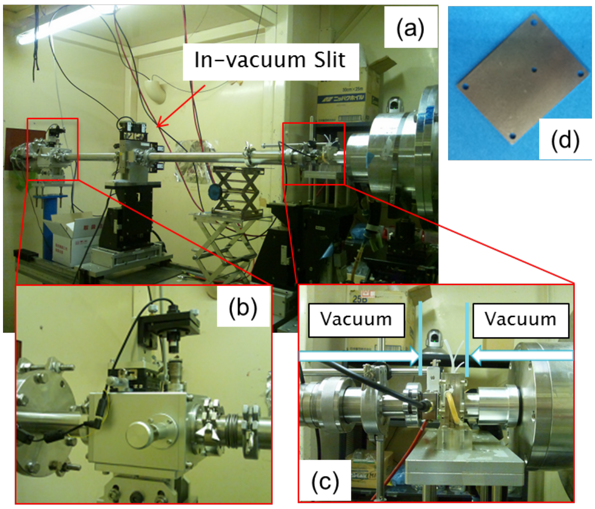

Figure 9: (a) New beam collimation system at BL-10C. (b) In-vacuum X-ray shutter and laser system (Rigaku Aihara Seiki). (c) Current status around the sample position. (d) A pinhole (1.6–1.8 mm) installed as a scattering guard in the micro ion chamber.

1-5 The Multipurpose X-ray Scattering Facility at the Indian Beamline, BL-18B, PF

The Indian beamline has been developed as a multipurpose beamline using beamport 18B of the Photon Factory ring. This beamline uses the existing beamline optics, mirrors, monochromators, slits, and so on, and we developed the experimental facilities in an existing experimental hutch at BL-18B. Presently, the available facilities are: (a) powder diffraction under ambient conditions, at low temperatures down to 10 K and at high temperatures up to 1200 K, (b) single-crystal diffraction from epitaxial multilayers and other nano-structures, (c) X-ray reflectivity and diffuse scattering from solid surfaces and interfaces, and (d) X-ray reflectivity, diffuse scattering and grazing incidence diffraction from liquid surface and liquid-liquid interfaces. For carrying out all these different types of X-ray scattering experiments, uniquely designed coupled goniometers have been installed in the experimental hutch of 18B (Fig. 10). The first goniometer is a four-circle goniometer and is used

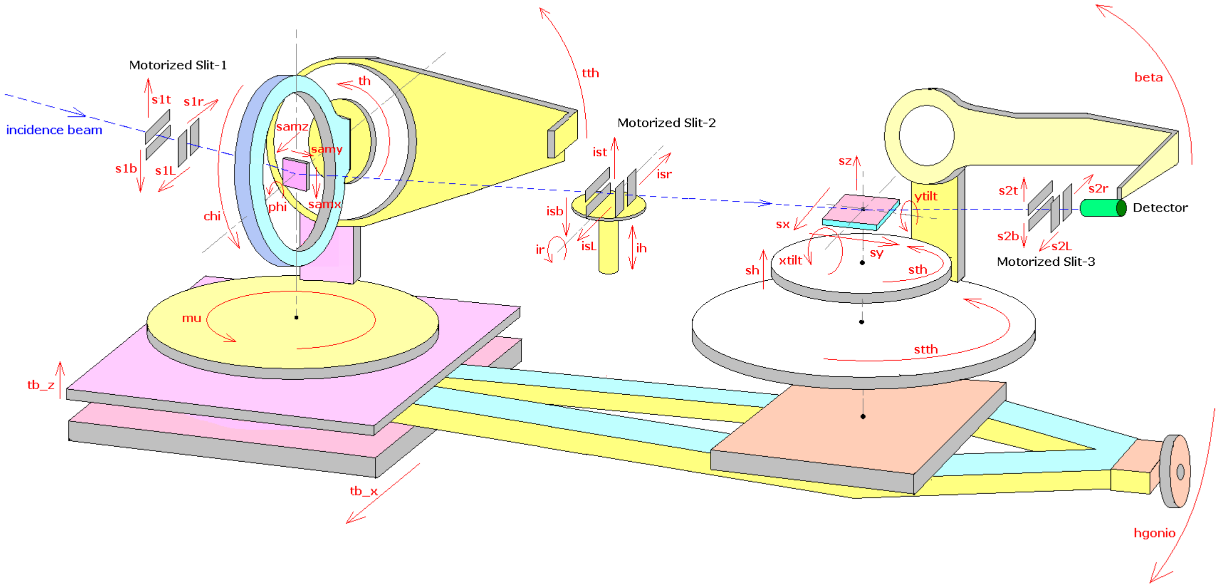

Figure 10: Schematic diagram of the experimental setup at the Indian beamline, BL-18B indicating all the motor controlled elements.

for powder diffraction in various sample environments: reflectivity, diffuse scattering, and diffraction experiments from solid samples (Fig. 11). The second goniometer is an eight-circle goniometer and is mounted on a rotation stage by air-wheel motion whose axis of rotation passes through the center of the first goniometer. This design enables us to perform experiments from a liquid surface where the Ge(111) crystal mounted on the first goniometer bends the beam down to the liquid surface and the air-wheel rotation coupled with vertical downward motion of the whole goniometer assembly tracks the beam onto the liquid sample. The beamline has one point detector (Cyberstar) and a 100K Pilatus detector. Installation of the high-pressure powder diffraction facility will be completed soon and for this purpose we are procuring a focusing optics (compact refractive lens) and a bigger 2D detector (1M Pilatus). We also plan to install a facility to study the in-situ growth and structural characterization of thin films and nanostructures. The second eight-circle goniometer, focusing optics and the 1M Pilatus detector will be used for this set-up. The 1M Pilatus detector and the long distance between the detector arm of the second goniometer and the sample stage of the first goniometer will also allow us to collect small-angle scattering data in reflection and transmission geometry. The Indian beamline at the Photon Factory (PF) is sponsored by the Department of Science and Technology (DST), Government of India, with the Saha Institute of Nuclear Physics (SINP) as the main institute implementing the project.

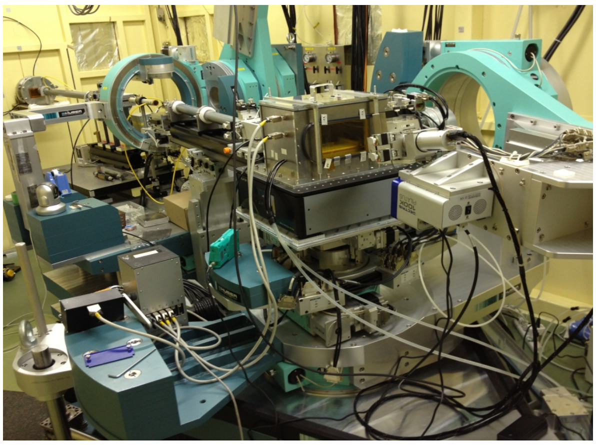

Figure 11: A picture of the experimental setup consisting of two goniometers at the Indian beamline, BL-18B. The first goniometer (in the back) can have the sample mounted for reflectivity and diffuse scattering experiments in the vertical plane, the second goniometer (at the front) has an active vibration table and Langmuir-Blodgett trough mounted on the sample stage for liquid surface experiments. The detectors (Cyberstar and 100K Pilatus) mounted on the second goniometer can be scanned in both the vertical and horizontal scattering planes.

1-6 Development of Beamlines for Structural Biology

A new diffractometer for in situ crystallography has been developed (Fig. 12). With this diffractometer, droplets in a crystallization plate can be directly exposed to an X-ray beam at room temperature. Combining the new diffractometer with the high-throughput crystallization screening described above enables a much more efficient evaluation of crystallization conditions.

A new VPN gateway dedicated for remote access to the structural biology beamlines in the PF has been installed at the KEK computing research center. This enables users to access beamline control from outside KEK. In addition, a remote access service has begun to be provided to limited users (industrial or project-involved users). In this service, a user sends cryo-cooled samples stored in a container for our sample exchange system and a beamline operator sets the samples in a beamline; the user then carries out experiments remotely. To operate this service correctly, PReMo (PF Remote Monitoring system) was updated to organize communication among operators and users. PReMo was also extended to manage all information derived from beamline operations, including experimental data, beam-time applications, beam-time schedule, and beamline-operation reports [1]. This enables the users and beamline staff to handle discursive data more efficiently.

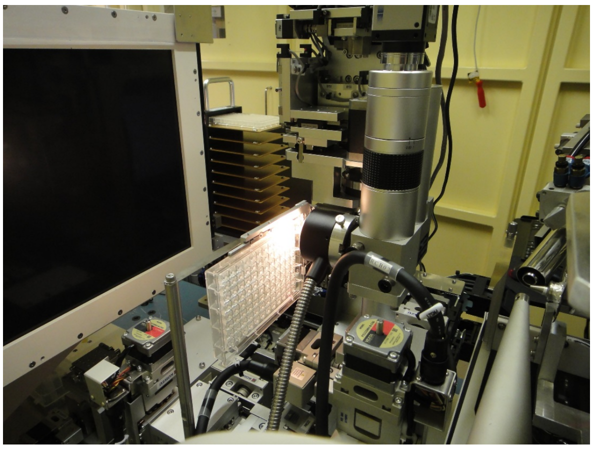

Figure 12: A new diffractometer for in situ crystallography, which consists of a plate holder, on-axis viewing system, and plate stacker. It is placed on an existing detector table.

REFERENCE

[1] Y. Yamada, N. Matsugaki, L. M. G. Chavas, M. Hiraki, N. Igarashi and S. Wakatsuki, J. Phys.: Conf. Ser. 425, 012017 (2013).