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# Newly Developed Experimental Facilities

## 1-1 Overview

The Photon Factory (PF) is a pioneering light source providing bright X-rays and outstanding user support. The PF produces extremely bright X-rays as a resource for researchers to study materials at the atomic and molecular level. Research at the PF also benefits many sectors of innovation in Japan and leads to major advances in energy production, environmental remediation, nanotechnology, new materials and medicine. The PF provides unique educational experience and serves as a vital training ground for future generations of scientists and engineers.

The PF has been carrying out a beamline refurbishment program since 2006, in which the main strategy is to concentrate investments on insertion-device (ID) beamlines as reported in previous activity reports [1-3]. With the upgrade of the 2.5-GeV PF ring for extending straight sections, the long and medium straight sections have been lengthened so that state-of-the-art insertion devices can be installed to cover the vacuum ultraviolet and soft X-ray regions. Four new straight sections have been newly produced for allowing the installation of short-period and small-gap undulators (SGUs) to supply well-focused hard X-rays (HX). 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. As the last straight section, we are commissioning a new beamline for both small-angle X-ray scattering (SAXS/GI-SAXS) and X-ray spectroscopy (XAFS/XRF/XRD) at BL-15. The details are described below.

BL-2 was the first undulator-based vacuum ultraviolet/soft X-ray (VSX) beamline constructed in the early 1980s, and has been operating for over 25 years. The BL-2 undulator is 3.6-m long, while the length of the straight section is 9 m after the PF upgrade in 2005. A new tandem undulator with 16-cm period was installed in 2014 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 in 30–4000 eV. 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 in 2006 as a high-performance spectroscopic beamline dedicated to photoelectron spectroscopy in the VSX region. 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. BL-13 was recently reconstructed as a VSX spectroscopic beamline for studying organic thin films adsorbed on well-defined surfaces using angle-resolved photoelectron spectroscopy and X-ray absorption spectroscopy. The detailed performance is described below.

In addition to the upgrade of the ID beamlines, competent bending-magnet stations are also being upgraded for supporting active users. The current status of SAXS stations at BL-6A and BL-10C, soft X-ray spectroscopy station at BL-11A, and X-ray topography and related X-ray diffraction station at BL-20B are also described in this section.

## REFERENCES

- [1] *Photon Factory Activity Report 2010 #28*, (2012).
- [2] *Photon Factory Activity Report 2011 #29*, (2013).
- [3] *Photon Factory Activity Report 2012 #30*, (2014).

## 1-2 BL-15A, Dedicated to Semi-Microbeam XAFS/XRF/XRD and High-Brilliance SAXS/GI-SAXS Studies

As reported previously [1], a new beamline, BL-15A, was completed at the BL-15 section of the PF ring in FY2013. This new beamline has a short-gap undulator which produces high-brilliance X-rays ranging from 2.1 keV to 15 keV. The beamline will be dedicated to both XAFS/XRF/XRD studies using semi-micro focus beams (A1 station) and SAXS/GI-SAXS experiments using collimated soft and hard X-rays (A2 station).

In the XAFS/XRF/XRD studies, the semi-micro focus beam available in a wide range of photon energies allows analyzing the local structures of the elements and valence of inhomogeneous samples in the fields of environmental science and new energy source science. The soft X-rays up to 2.1 keV will provide access to the absorption edges of phosphorus and sulfur, which are very important targets for those fields.

The SAXS scientific programs include structural studies of functional membranes, time-resolved X-ray scattering and large hierarchical structure analysis. All three programs require a high-brilliance light source. In particular, grazing-incidence SAXS (GI-SAXS) using vertically small-size soft X-rays ranging between 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.

Furthermore, the combination of XAFS/XRF and SAXS experiments yields wide structural information from fine atomic structures to low and medium resolution. It is beneficial to build these instruments as two stations (A1 and A2) on the same beamline. BL-15A is oriented toward joint advanced studies using the two techniques.

The old BL-15 beamlines were scrapped and new construction work started in the spring of 2013. The construction was completed in the summer shutdown of 2013 (Fig. 1) and the first undulator beam was introduced on October 17, 2013 (Fig. 2). We subsequently started tuning the optics, and the first beams into the A1 and A2 stations were delivered on November 7, 2013

and February 19, 2014, respectively. After the optics tuning, preliminary beamline characterizations were carried out. The FWHM beam sizes at three focal points, 32.8 m, 36.75 m and 42.75 m from the source, were  $20\ \mu\text{m}$  (H)  $\times$   $21\ \mu\text{m}$  (V),  $289\ \mu\text{m}$  (H)  $\times$   $36\ \mu\text{m}$  (V) and  $651\ \mu\text{m}$  (H)  $\times$   $44\ \mu\text{m}$  (V), respectively. The undulator spectra and the photon flux were measured and the results are consistent with the ray-tracing simulation. These beam performances are promising for both research activities.

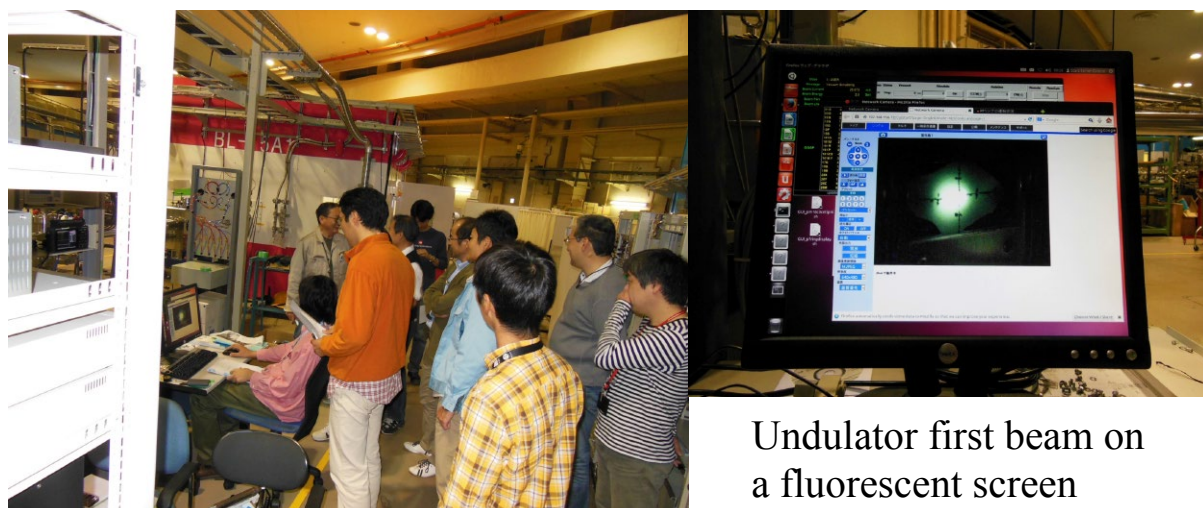
We will continue the optics tuning and commissioning experiments, and then start beamline operation for users in the autumn of 2014.

## REFERENCE

- [1] *Photon Factory Activity Report 2012 #30*, A 73 (2013)



Figure 1: Bird's-eye view of the BL-15A.



Undulator first beam on a fluorescent screen

Figure 2: First beam introduction on October 17, 2013.

### 1-3 Construction of New Wide-Energy Range VUV & SX Beamline BL-2 “MUSASHI”

The new beamline BL-2 (MUSASHI: Multiple Undulator beamline for Spectroscopic Analysis on Surface and Hetero Interface) is designed for wide-energy-range spectroscopic analysis including X-ray photoelectron spectroscopy, angle-resolved photoemission spectroscopy, and X-ray absorption spectroscopy. The conceptual scheme of the new BL-2 is shown in Fig. 3. The new BL-2 has two types of undulators in tandem alignment; one is designed for the vacuum ultraviolet (VUV) region (30–300 eV), and the other for the soft X-ray (SX) region (250–2000 eV). Consequently, light with a relatively wide energy range while maintaining high brilliance and high energy resolution will be available in this beamline by the combination of the two undulators and a variable-included-angle varied-line-spacing plane-grating monochromator (VLS-PGM). Moreover, in BL-

2B, an additional double-crystal monochromator (DXM) is built into this branch beamline to make the energy range of 2000–4000 eV available using the wiggler mode of the SX undulator.

In the summer of 2013, the old BL-2A and BL-2C beamlines were scrapped and the new BL-2A and BL-2B beamlines were constructed (Fig. 4). Figure 5 shows a photoabsorption spectrum of  $N_2$  molecules obtained using the new beamline optics with SX undulator. The fitting result by Voigt functions, Gaussian width of 31 meV with the Lorentzian width of 117 meV, indicates that the SX optics of the new BL-2 beamline achieve a resolving power of more than 13,000, which is much better than that of the old BL-2C beamline [1].

The installation of the new VUV undulator and the operation of VUV optics will start in the spring of 2014, and the completed version of the beamline will be opened for users in FY2015.

#### REFERENCE

- [1] M. Watanabe, A. Toyoshima, Y. Azuma, T. Hayaishi, Y. Yan and A. Yagishita, *Proc. SPIE*, **58**, 3150 (1997).

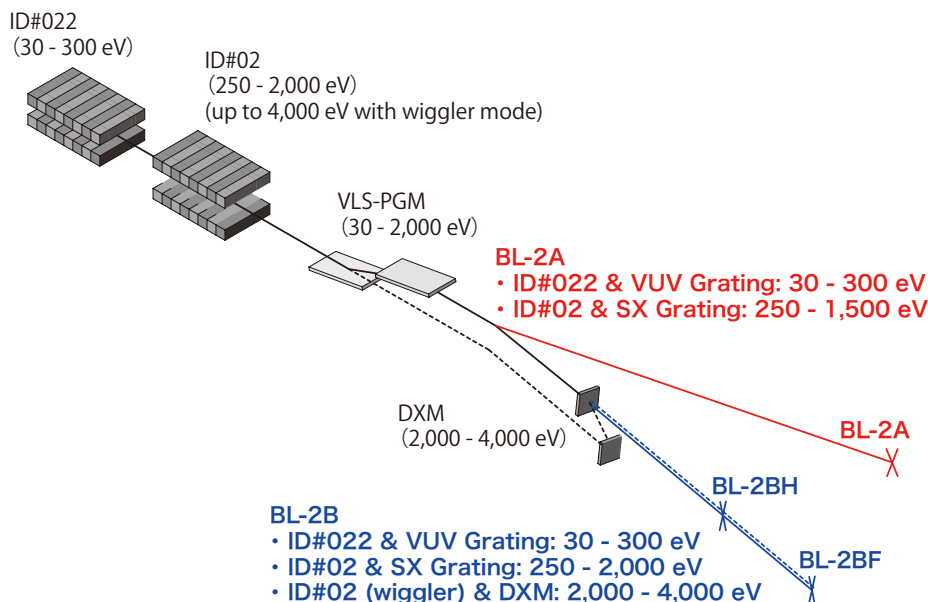


Figure 3: The conceptual scheme of the new BL-2 beamline.

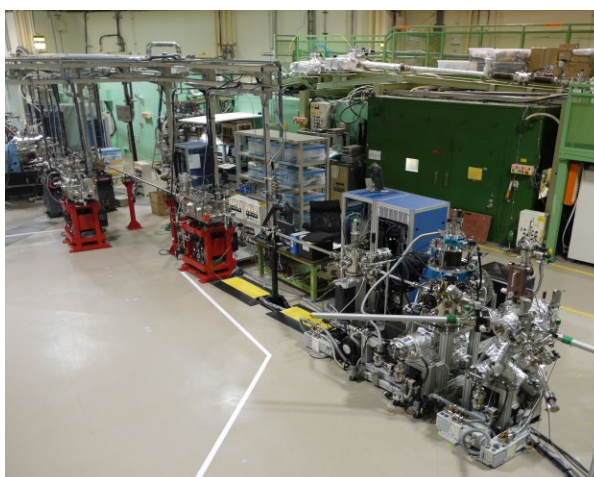


Figure 4: Photograph of the constructed BL-2A and BL-2B beamlines.

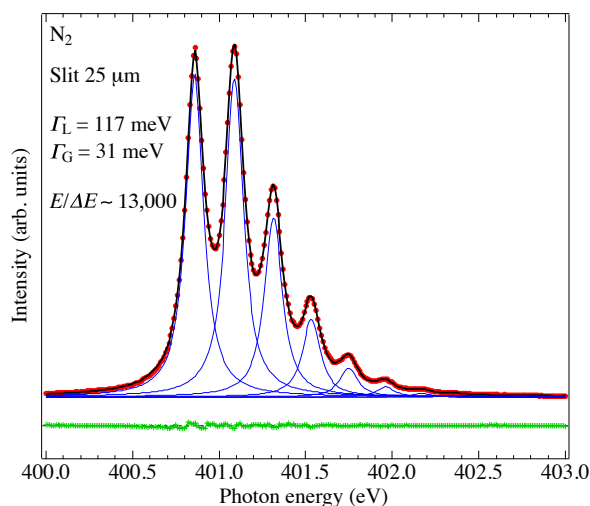


Figure 5: Photoabsorption spectrum of  $N_2$  molecules.

## 1-4 Construction of BL-13B, Optics for Photoelectron Spectroscopy

Optics for photoelectron spectroscopy for the study of surface chemistry (BL-13B) was opened for users in October, 2013. A typical photon intensity and photon energy resolution ( $E/\Delta E$ ) of BL-13B are shown in Fig. 6. Carbon contamination on the Au-coated plane mirror (Mp) for suppressing higher harmonics was removed by using oxygen activated by non-monochromatized synchrotron radiation (SR) [1] as shown in Fig. 7. Since the removal of the carbon contamination, the photon intensity in the carbon K-edge region is improved while the change in intensity in the oxygen K-edge region is negligible.

### REFERENCE

- [1] A. Toyoshima, T. Kikuchi, H. Tanaka, J. Adachi, K. Mase and K. Amemiya, *J. Synchrotron Rad.* **19**, 722 (2012).

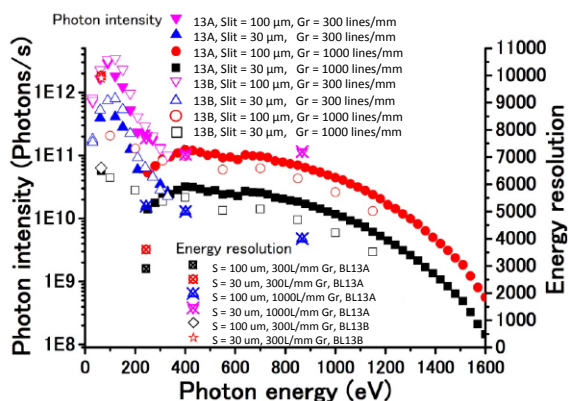


Figure 6: Typical photon intensity and photon energy resolution ( $E/\Delta E$ ) of BL-13A and BL-13B in the photon energy region of 30–1600 eV.

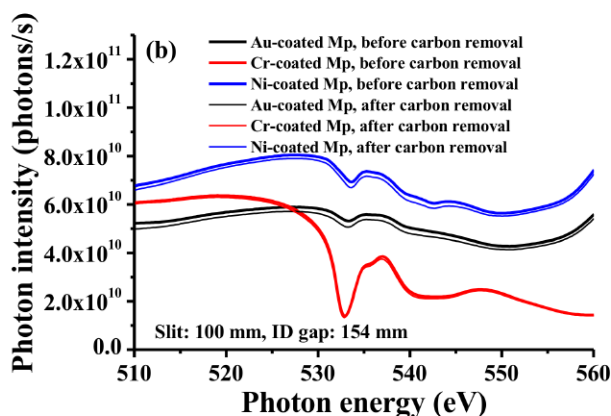
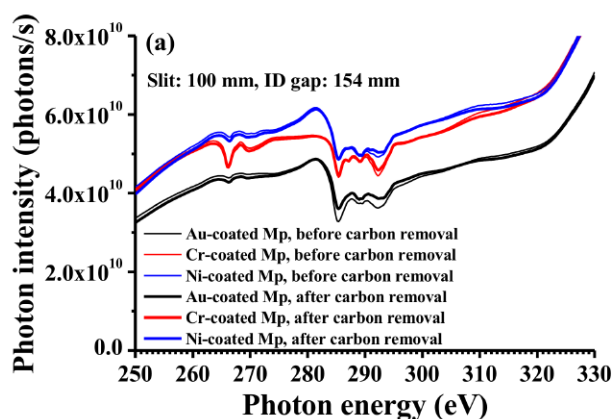


Figure 7: Photon intensity spectra of BL-13B in the (a) carbon and (b) oxygen K-edge regions measured with the 1000-lines/mm grating before and after in situ non-monochromatized-SR-activated oxygen cleaning of the Au-coated Mp.

## 1-5 Upgrade of PF SAXS Beamlines: Replacement of the Experimental Stage and the Detector at BL-6A, and Refurbishment of BL-10C

BL-6A is a small-angle X-ray scattering (SAXS) beamline for mainly the structural study of hard and soft materials. In BL-6A, the experimental stage and the SAXS detector were replaced in fiscal 2013 in order to improve the user operability and the detectable angle range (Fig. 8). The camera length can be changed semi-automatically from 0.25 to 2.5 m in this stage. The hybrid pixel detector, PILATUS 300K, had been used at BL-6A. However, its detectable area of  $83.8 \times 106.5 \text{ mm}^2$  was narrow in order to enable structural analysis with large spatial resolution. PILATUS3 1M was newly installed as a SAXS detector, thus enabling measurement over a broad range of angle in combination with a WAXS detector, PILATUS 100K, since its area of  $168.7 \times 179.4 \text{ mm}^2$  is sufficiently larger than that of the 300K. We also installed a downstream shutter (DSS) in BL-6A to improve the safety of BL-6A and BL-6C.

BL-10C is also a SAXS beamline and is one of the oldest beamlines at the Photon Factory. Therefore, in order to renew the optical components and the measurement system, scrap-and-rebuild of BL-10C was performed in fiscal 2013. The experimental hutch was enlarged in the summer of 2013 to prepare for the installation of the longer experimental stage. All other reconstruction work was conducted in March 2014. All the components from the end of the main hutch to the experimental hutch were removed first, then the main

components, the fixed-exit double-crystal monochromator, a two-dimensional focusing mirror (bent cylinder mirror), a long experimental stage, WAXD (Wide-angle X-ray diffraction) chambers and two hybrid pixel detectors, PILATUS3 2M for SAXS and 200K for WAXD, were newly installed in BL-10C (Fig. 9). As a result of these upgrades, users will be able to change the measurement energy from 6 to 14 keV and to use a higher-brilliance beam by changing the focusing ratio. The maximum camera length is extended from 2 m to 3 m in this experimental stage, and PILATUS3 2M

with a large area and high-speed readout will help to perform high-throughput structural analysis with a large spatial resolution. The simultaneous SAXS(GI-SAXS)/WAXD measurement system was newly constructed in BL-10C. In order to improve beamline safety, we also replaced the old electric wirings and distribution boards which had been used for over 30 years with new ones. A chain block and a cable ladder were also installed in the experimental hutch. The user time will restart from the end of May 2014 after commissioning.



Figure 8: New experimental stage and detector, PILATUS3 1M at BL-6A.

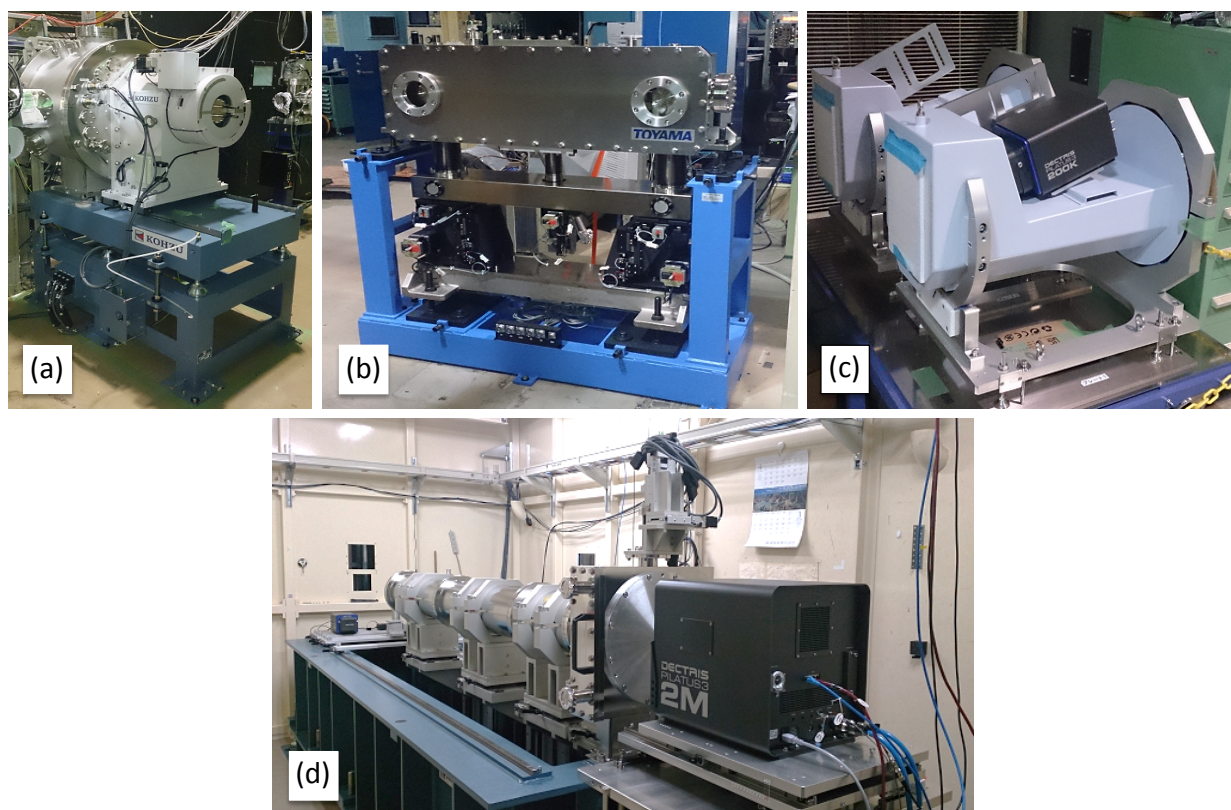


Figure 9: New beamline components at BL-10C. (a) A water-cooled fixed-exit Si(111) double-crystal monochromator. (b) A 1000-mm 2D-focusing mirror and a mirror bender. This mirror is coated with rhodium on the silicon substrate with a cylinder shape, and bent to focus the beam at the detector position with this bender. (c) Two kinds of chambers for WAXD experiments. One is for SAXS/WAXD, and the other is for GI-SAXS/WAXD. PILATUS3 200K can be exclusively installed in these chambers. (d) A new long experimental stage and a new PILATUS3 2M detector. Users can use the camera lengths of 0.25, 0.5, 1.0, 2.0 and 3.0 m, and change to these lengths semi-automatically.

## 1-6 Replacement of Main Optical Components of the Wide-Range Soft X-Ray Spectroscopy Station BL-11A

BL-11A provides soft X-rays in the energy range between 70 eV and 1900 eV with a grazing-incidence monochromator from a bending-magnet source, where all of the beamline components were reconstructed in 1996 [1-3]. After being used for 18 years, it was decided to replace the main part of the beamline optics to improve the performance. The purpose of this modification is to attain higher flux in the energy region higher than 1 keV and to provide a stable beam. To achieve this, the incident angle to the focusing mirrors (M0', M1 and

Mf) is changed from 2 degrees to 1 degree, except for the first mirror M0 in the shield wall, and a water-cooling system for the gratings is introduced. All the components have been set on the optical path and commissioning will start in May 2014. Details of the beamline optics and progress of the commissioning will be posted on the beamline website ([http://pfwww.kek.jp/sxs/spec/sx/bl11a\\_e.html](http://pfwww.kek.jp/sxs/spec/sx/bl11a_e.html)) together with the previous performance.

### REFERENCES

- [1] K. Amemiya, Y. Kitajima, T. Ohta and K. Ito, *J. Synchrotron Rad.* **3**, 282 (1996).
- [2] Y. Kitajima, K. Amemiya, Y. Yonamoto, T. Ohta, T. Kikuchi, T. Kosuge, A. Toyoshima and K. Ito, *J. Synchrotron Rad.* **5**, 729 (1998).
- [3] Y. Kitajima, Y. Yonamoto, K. Amemiya, H. Tsukabayashi, T. Ohta and K. Ito, *J. Elec. Spectrosc. Relat. Phenom.* **927**, 101 (1999).

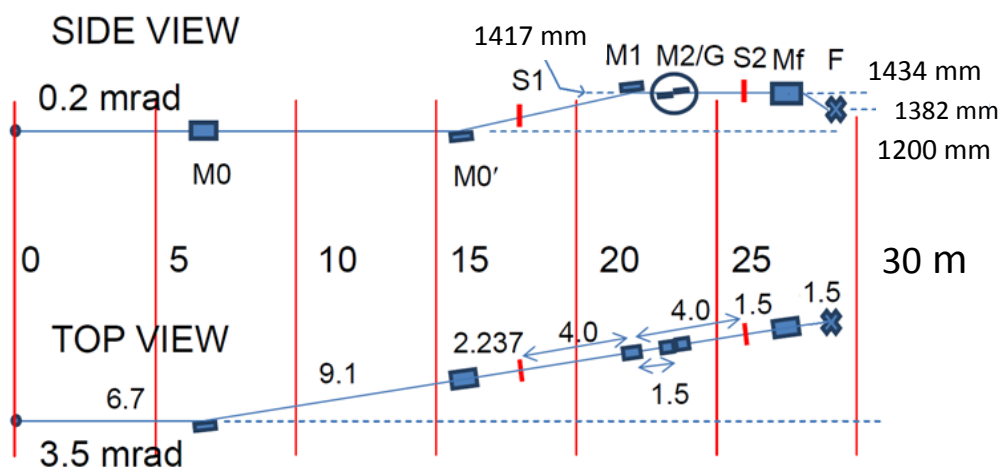


Figure 10: Schematic layout of BL-11A. Only M0 is left as before; the other components have been replaced.

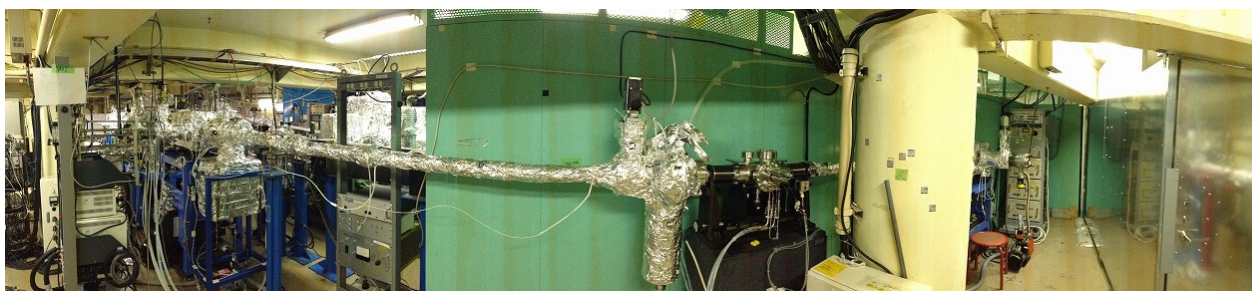


Figure 11: Photograph of M0', S1, M1 and M2/G chamber (from right to left).

## 1-7 Reconstruction of BL-20B for X-Ray Topography and Related X-Ray Diffraction Experiments

BL-20B, which was the former Australian beamline, had been reconstructed for X-ray diffraction topography and related X-ray diffraction experiments with SR from bending magnets. The Australian activity at BL-20B ceased at the end of February 2013. X-ray topography and related activities at the former BL-15B and 15C were transferred to the new BL-20B. Figure 12 shows a plan view of the beamline structure after the reconstruction. The main modified parts are as follows.

1. The cam-type double-crystal monochromator which was used at the old BL-6C was installed instead of the channel-cut monochromator system. The monochromator crystals have also been changed from CZ-Si (111) to FZ-Si (111).

2. All auxiliary vacuum pumps for turbo-molecular pumps were changed from oil rotary pumps to oil-free scroll pumps to prevent carbon contamination for the monochromator crystal.
3. A white SR beam is practically available with safety.
4. A  $\gamma$ -ray mask with water-cooled mask and one  $\gamma$ -ray stopper were additionally installed to strengthen safety for  $\gamma$ -rays.

The topography goniometer with vertical rotation axis for the former BL-15B and the topography goniometer with horizontal rotation axis for the former BL-15C were moved to the new BL-20B. The equivalent control system of the old BL-15C was employed for the beamline. The horizontal acceptance angle of SR is 1.6 mrad in spite of the designed value of 2.0 mrad. This seems to be caused by the difference of the actual SR axis and the designed axis.

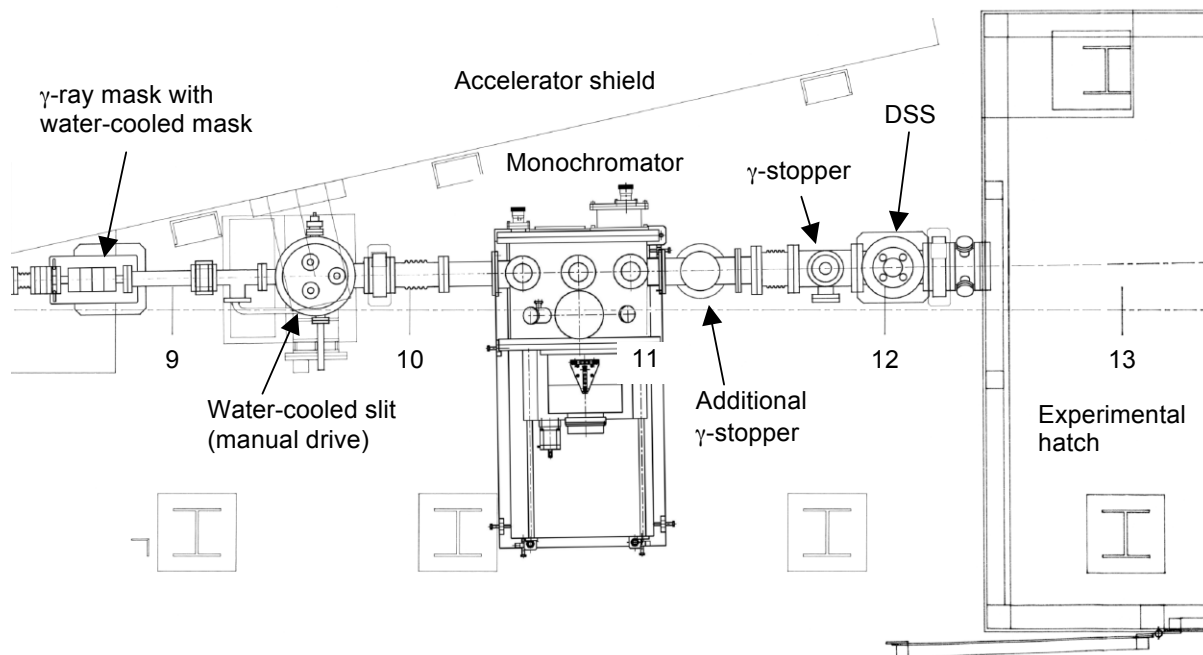


Figure 12: Plan view of the new BL-20B. The scale shows the distance from the light source along the BL-20A axis.

## 1-8 Upgrading of the BL-6C

BL-6C had been constructed as a double focusing X-ray beamline by means of a double-crystal X-ray monochromator and cylindrical bent X-ray mirror systems and used for almost ten years. The various scientific activities on material science used the beamline by means of resonant X-ray magnetic and non-magnetic Bragg scattering, X-ray MCD, X-ray fluorescent holography techniques, surface diffraction, and strain measurement systems. However, one of the problems was the limitation of relatively low energy usage for the focused X-ray beam because of the cut-off energy of the focusing mirror (~13 keV). The focusing mirror was located just upstream of the experimental hutch of BL-6C for geometrical reasons based on the interference of other beamlines.

In 2013, BL-15 was reconstructed based on the short-period undulator line, and the X-ray monochromator of the old BL-15C could be used for the present reconstruction of BL-6C. Figure 13 shows the plan views of the reconstruction. The upper and lower drawings

correspond to the new and old BL-6C, respectively. The mirror was located at 23.1 m from the light source before the reconstruction. After the reconstruction, the X-ray monochromator can be located beside the experimental hutch of BL-6A and the mirror can be shifted to the upstream of the beamline at the position of 19 m from the light source. Thus, the cut-off energy of the mirror can be increased up to about 20 keV.

The reconstruction was done during fiscal 2013. Figure 14 shows a photograph of the movement of the X-ray mirror. The focusing test of the beamline was successfully done in May 2014 and the FWHMs of the focused beam size are 0.5 mm (horizontal) and 0.3 mm (vertical), respectively. Figure 15 shows the energy spectra of the focused beam before and after reconstruction. It is possible for general users to use almost up to 19–20 keV monochromatic X-rays, enabling various atomic species such as Y, Zr, Nb, Mo and Ru to be examined by X-ray fluorescent holography and also resonant X-ray scattering techniques. Scientific results will emerge next fiscal year.

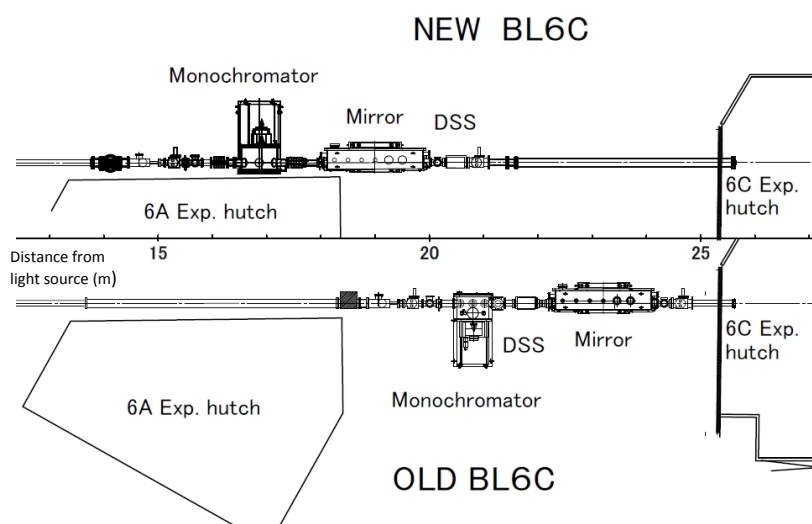


Figure 13: Plan views of the reconstruction. The upper and lower drawings correspond to the new and old BL-6C, respectively.



Figure 14: Photograph of the movement of the X-ray mirror.

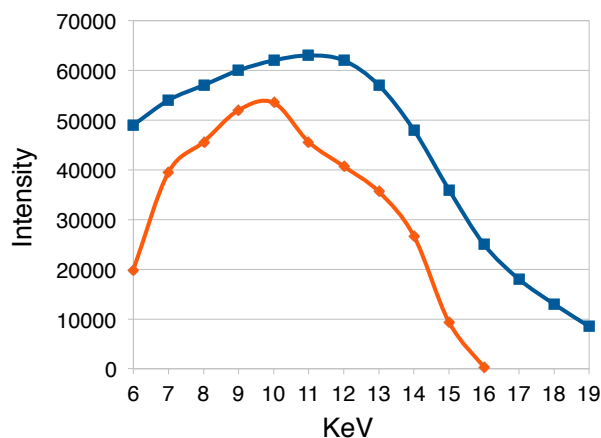


Figure 15: Energy spectra of the focused X-ray beam before and after reconstruction. The red (blue) line corresponds to the energy spectrum of the focused X-ray beam before (after) the reconstruction. It will be possible to use X-ray energy of up to 19 keV after the reconstruction.