

## 4

# Research Groups and Beamline Updates

## 4-1 Overview

The Photon Factory (PF) is an outstanding research facility that harnesses accelerator technology to provide bright light for users. The facility utilizes the light so researchers can see the fundamental structure, composition and behavior of materials, on scales ranging from the atomic to the macroscopic with a level of detail, speed and accuracy not possible using conventional laboratory-based equipment. The PF supports a broad range of high-quality research and leads to major advances in energy production, environmental remediation, nanotechnology, new materials and medicine. Our advanced techniques and dedicated staff contribute directly to scientific advances and industrial innovations.

The PF has carried out the beamline refurbishment program since 2006, in which the main strategy is to concentrate investments mainly on insertion-device beamlines. 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. In addition,

four short straight sections have been newly produced for allowing the installation of short-period and small-gap undulators to supply well-focused hard X-rays.

Beamline developments over the past year have mainly focused on the above-mentioned beamlines for improving the reliability, speed of operation, quality of data acquired, and the ability to reach new areas of scientific impact. Despite continuous improvements in efficiency, demand for beamtime exceeds supply on some beamlines, which reflects the continued expansion of our researcher community. There is a great need to expand the range of techniques to meet the oversubscribing demands of the user community.

At our facility, it is the expertise and dedication of all of our staff that maximizes the productivity from highly specialized equipment. Our people are a diverse group with expert skills in such areas as high-level research and development in materials and biological sciences including R&D projects for future light sources, beamline engineering and technical support, computing and control systems, user safety, user liaison and public relations (Table 1). The current status and future prospects of the groups at the PF are summarized below.

Table 1: Research groups in synchrotron radiation I and II (see Fig. 2 in p.114).

Group Name	Group Leader	Group Description
Electronic Structure	H. Kumigashira	This Group is studying the electronic structures of materials to reveal the origins of the properties of those materials and to design their functionalities by using photoelectron spectroscopic techniques such as photoemission spectroscopy and X-ray absorption spectroscopy. In order to conduct state-of-the-art research, we are developing high-performance beamlines and instruments with high brightness insertion devices in the VUV and soft X-ray regions.
Condensed Matter	H. Nakao	This Group is investigating the static structure of materials by means of precise X-ray crystal structural analysis, resonant X-ray scattering, magnetic X-ray scattering, and X-ray imaging. In addition, we are studying the dynamic structure of materials via a pump-and-probe experiment that uses a synchrotron radiation pulse. Our research has expanded from fundamental high-pressure properties to Earth and planetary science by a combination of high-pressure X-ray measurements. In addition to our research, we are optimizing beamlines and experimental devices to promote our research and are developing research methods that use next-generation light sources

Group Name	Group Leader	Group Description
Materials Chemistry	M. Kimura	This Group is developing and providing the observational technology required for chemistry that contributes to the production of materials and substances that improve daily life, and expanding the research and use of this technology in other fields. Taking into consideration next-generation light sources, we are conducting research that uses observational technology, including X-ray absorption fine structure (XAFS) spectroscopy and scattering and chemical-state imaging, in collaboration with industry, academia, and the government. Currently, we are involved in various projects such as “structural materials for innovation” under the Cross-ministerial Strategic Innovation Promotion Program (SIP) by the Cabinet Office and Accelerated Innovation Research Initiative Turning Top Science and Ideas into High-Impact Values (ACCEL) of Japan Science and Technology Agency (JST).
Life Sciences	T. Senda	This Group is conducting life science researches with synchrotron radiation in the fields of 1) structural biology, 2) X-ray imaging, and 3) radiology. These activities range from atomic to organ level researches and reveal connections between the structures and functions of biological components.
Slow Positron	T. Hyodo	The positron is the anti-particle of the electron. This Group uses a slow positron beam created by a dedicated 55-MeV linac for researches in solid state, surface, and atomic and molecular physics. The stations in operation are for positronium negative ion research, positronium-time-of-flight spectroscopy, and total-reflection high-energy positron diffraction (TRHEPD) for the surface structure
Beamline Engineering, Technical Service & Safety	N. Igarashi	The mission of this Group is to develop and construct advanced synchrotron radiation instrumentation and use it safely. Specific tasks are: 1) Development and construction of synchrotron radiation beamlines 2) R&D Study of experimental apparatus and detectors 3) R&D Study of beamline control system 4) Safety control for synchrotron radiation experiment
User Support and Dissemination	K. Hyodo	This Group works as the liaison to users and public relations. The major missions are: 1) Management of the whole peer-review process of the research proposals. 2) Analysis of the statistics related to user activities. 3) Dissemination of research outputs to public.
Advanced Detector R&D Working Group	S. Kishimoto	This Group aims to develop new detectors for materials structure science: 1) a high spatial-resolution and high-speed readout area detector for synchrotron X-rays, 2) an ultra-high-speed high-time-resolution X-ray detector, and 3) a pulse-counting detector with a fast response in the soft X-ray region with position and energy resolutions.
Ultrafast Dynamics Working Group	J. Adachi	This Group is pursuing the possibility of utilizing short-pulse X-rays which will be produced by the future light source, and is conducting R&D on establishing the technical platform for ultrafast dynamics experiments in the femtosecond time domain. To achieve this purpose, we are conducting time-resolved experiments at the PF and PF-AR, and carrying out R&D.

## 4-2 Electronic Structure Group

### 4-2-1. 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 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, relatively wide energy-range light 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 have the energy range of 2000–4000 eV available using the wiggler mode of the SX undulator.

At the end of FY2013, a new VUV undulator was installed (Fig. 1) and the operation of both SX and VUV optics was available from FY2014. Figure 2(a) shows a photoabsorption spectrum of  $N_2$  molecules obtained with the SX undulator and optics. The fitting result by Voigt functions, with Gaussian width of 29 meV and Lorentzian width of 117 meV, indicates that the SX optics of the new BL-2 beamline achieve a resolving power of more than 10,000 that of the old BL-2C beamline [1]. In addition, Fig. 2(b) shows a photoionization spectrum of double excited He obtained with the VUV undulator and optics. The minimum peak width of 3.0 meV in the  $(2,0)_{18}$  resonance peak is obtained, indicating that the VUV optics of the BL-2 beamline achieve a resolving power of more than 20,000, which is comparable to VUV undulator beamline BL-28 [2]. Thus, we can perform spectroscopic experiments using both VUV and SX light with high energy resolution at the new BL-2 MUSASHI. The completed version of this beamline will be opened for users in FY2015.

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Figure 1: Photograph of two undulators U#02-1 (for SX) and U#02-2 (for VUV) for BL-2.

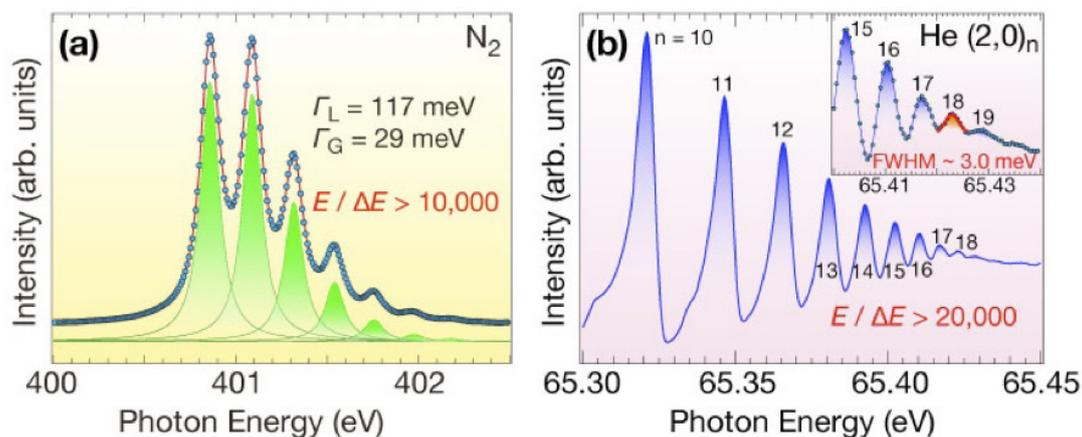


Figure 2: (a)  $1s\sigma \rightarrow \pi^*$  photoabsorption spectrum of  $N_2$  molecules and the fitting result by Voigt functions. (b) The photoionization spectrum of autoionizing resonance state  $(2,0)_n$  of double excited He.

#### 4-2-2. BL-13A/B: Variable polarization beamlines for VSX spectroscopy

An APPLE-II undulator was installed for BL-13A/B in February 2015. User experiments using horizontally linear and elliptically polarized SR began in May 2015. Vertically linear and circular polarization modes will be available in the near future. Figure 3 shows typical undulator spectra in the horizontally linear polarization mode with the  $\rho/2$  parameter of 0 mm measured at BL-13A. Photon intensity increases by about one order of magnitude in comparison with the BL-13A with the previous undulator.

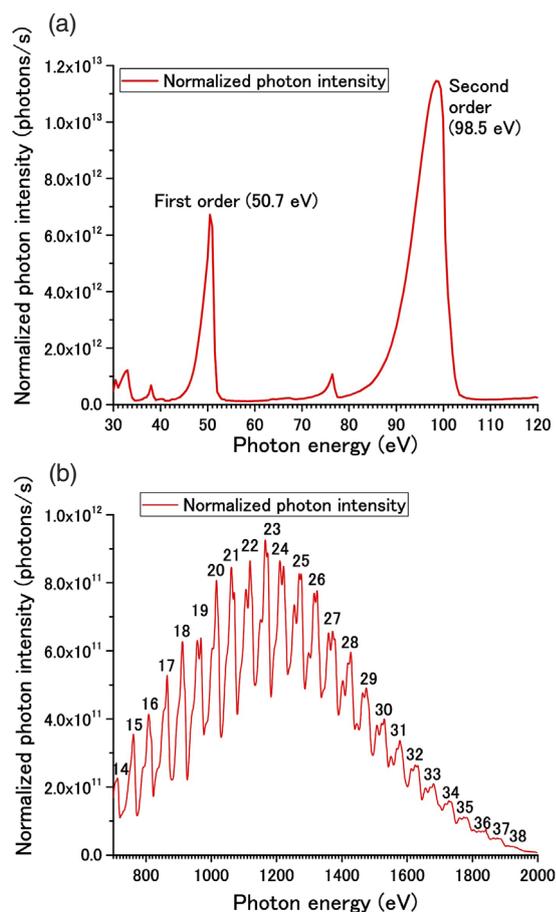


Figure 3 (a), (b): Typical undulator spectra in the horizontally linear polarization mode with the  $\rho/2$  parameter of 0 mm measured at BL-13A. The small peaks at 33, 38, and 76.5 eV derive from higher order harmonics.

#### 4-2-3. Upgrade of an undulator in beamline BL-28 for polarization-dependent angle-resolved photoemission spectroscopy

The optics of BL-28 were reconstructed as a high-performance spectroscopic beamline dedicated to angle-resolved photoelectron spectroscopy (ARPES) in the vacuum ultraviolet (VUV) region in 2006. At the end of FY2014, the undulator of BL-28 was replaced with a new one having six variable rows of magnetic arrays. The new undulator provides linear-horizontal, linear-vertical, and circular polarization states of radiation in



Figure 4: Photograph of the new undulator installed at BL-28.

the VUV energy region from 30 eV to 300 eV (Fig. 4). These variable polarization modes enable us to perform a state-of-the-art polarization-dependent ARPES, which will be available step-by-step for users from FY2015.

### 4-3 Condensed Matter Group

#### 4-3-1. Development of an in-vacuum diffractometer for resonant soft X-ray scattering

The strong correlation between electrons in solids plays very important roles in the electric and magnetic properties of the ordering states of orbital, charge, and spin degrees of freedom. The study of these electronic ordering states is essential to understand phenomena microscopically. Resonant X-ray scattering (RXS) at the  $K$ -edge is a powerful tool for observing the spatial ordering of charge and orbital degrees of freedom in  $3d$  transition metal oxides. The RXS signal at the  $K$ -edge reflects the  $4p$  electronic state. On the other hand, the RXS at the  $L_{2,3}$ -edge can probe the  $3d$  electronic state directly, and the signal of resonant magnetic scattering is strongly observed at the  $L_{2,3}$ -edge in  $d$  electron systems. Moreover, the RXS at the  $K$ -edges of O, S, and P ions, which play a key role in itinerancy through hybridization with the metal ion, also becomes observable using soft X-rays.

We have developed a new in-vacuum diffractometer as shown in Fig. 5 to carry out Resonant Soft X-ray Scattering (RSXS) experiments. We have succeeded in drastically improving the signal-to-noise ratio of the RSXS signals using a slit and silicon drift detector with good energy resolution in a large vacuum chamber as shown in Fig. 5(b). The diffractometer was installed in the beamlines BL-16A and BL-11B at the Photon Factory, and was commissioned. Utilizing the dif-

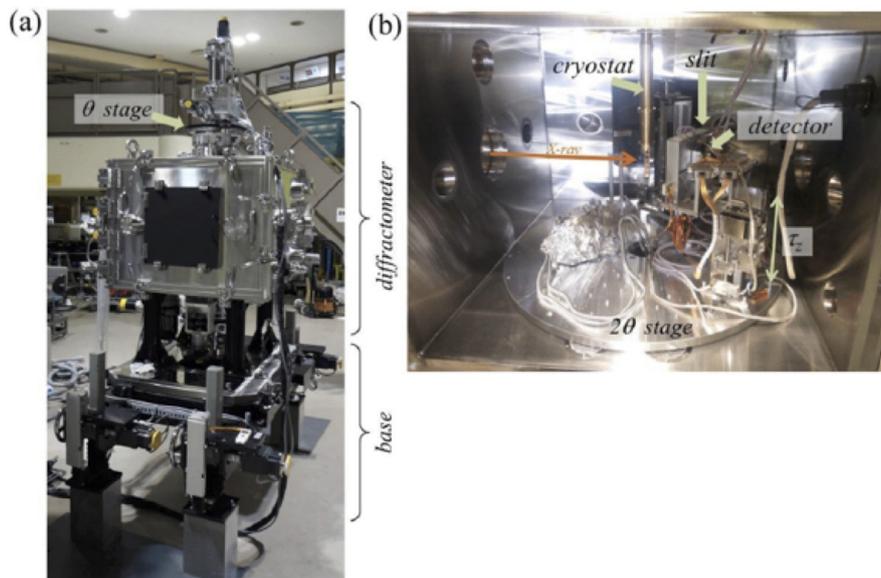


Figure 5: (a) New in-vacuum diffractometer and the alignment base. (b) Inside of the vacuum chamber. The stages, cryostat, and detector system are shown.

fractometer, not only RXS but also X-ray absorption spectroscopy measurements have been carried out to elucidate the electronic states, and the ordering of the charge, spin, and orbital states in strongly correlated electron systems such as transition metal oxides. We have also constructed a soft X-ray diffractometer with a 7.5-T superconducting magnet in order to study ordered electronic structures under high magnetic field. This diffractometer was used to detect the RSXS signals of  $(\text{LaMnO}_3)_3(\text{SrMnO}_3)_3$  superlattice with a remarkable negative magnetoresistance effect under a 7 T magnetic field. We have succeeded in observing the difference of RSXS spectra corresponding to reversal of the Mn  $3d$  magnetic moment.

#### 4-3-2. Condensed Matter Research Center (CMRC)

The CMRC pursues cutting-edge research on condensed matter science through the comprehensive use of multiple probes: synchrotron light, neutron, muon, and slow positron. The Condensed Matter Group is partly involved in the CMRC, and is producing crucial scientific outputs mainly using synchrotron light. The CMRC consists of four groups: the correlated electron matter group, the surface/interface group, the matter under extreme conditions group, and the soft matter group. These groups have been promoting nine bottom-up projects: 1. Hybridized Orbital Ordering, 2. Geometrical Correlation, 3. Molecular Crystals, 4. Oxide Heterostructures, 5. Surface/interface Magnetism, 6. Extreme Conditions, 7. Soft Matter, 8. Hydrogen, and 9. Surface Structure and Electronic States. In addition, the CMRC is conducting two types of Ministry of Education, Culture, Sports, Science and Technology (MEXT) project: the Element Strategy Initiative to Form a Core Research

Center, and the Photon and Quantum Basic Research Coordinated Development Program. In these national projects, the CMRC members are focusing on the Element Strategy Initiative for Electronic and Magnetic Materials, and Tribology with Muons and Neutrons.

## 4-4 Materials Chemistry Group

### 4-4-1. Status of beamlines managed by MCG

Our group operates the beamlines and experimental stations of BL-9A, BL-9C, BL-12C, BL-15A1, AR-NW10A, and AR-NW2A. These BLs are mainly dedicated to XAFS experiments, and are widely available for academic and industrial users. In addition, we support BL-4A for XRF experiments, which are operated by the microbeam XRF users group. The following experimental apparatuses have been recently installed at the BLs: an automated in situ experimental condition setup system (auto in situ system) at BL-9C, automatic 100 samples measurement system at BL-12C, and 21-element Ge pixel array detector (21Ge-PAD) at AR-NW10A. The auto in situ system at BL-9C enables us to set measurement conditions such as gas atmosphere, temperature and heating speed.  $\text{H}_2$ ,  $\text{O}_2$ ,  $\text{CO}$ , and  $\text{NO}$  gases are available with  $\text{N}_2$  and  $\text{He}$  as balance gases. With the automatic 100 samples measurement system at BL-12C, we can measure up to 100 samples sequentially without sending any commands. Fluorescence yield measurements with the 21Ge-PAD at AR-NW10A enable us to study samples containing diluted elements of relatively high-energy absorption edges, especially higher than 18 keV. These apparatuses will attract researchers at corporations, not only in academia.

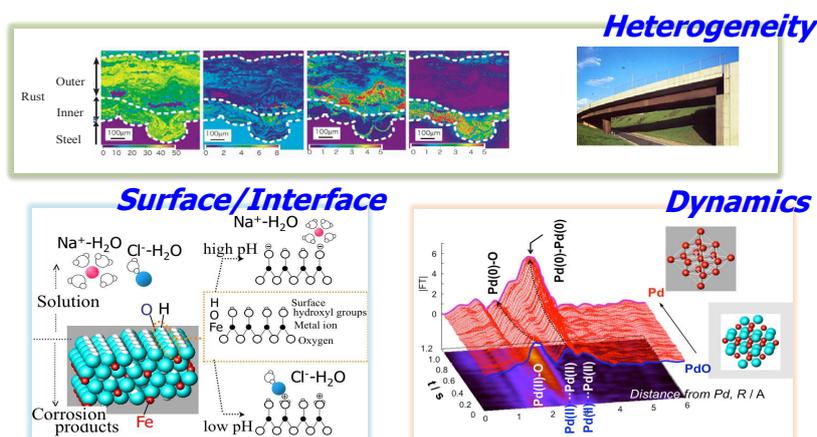


Figure 6: Target of the Materials Chemistry Group.

One of our most important missions is to develop experimental methods and to perform cutting-edge science, in addition to the maintenance and improvement of BLs as outlined above. Recently, we designated three important fields of science: heterogeneity, dynamics, and surface/interface (Fig. 6). BL-15A1 creates a semi-micro beam (ca. 20  $\mu\text{m}$ ) for mapping of chemical states using X-ray Absorption Spectroscopy (XAS) and crystal structures using XRD, and is suitable for studying the heterogeneity of various samples. Also, we plan to install a scanning transmission X-ray microscope (STXM). STXM at BL-15A1 enables us to observe samples with a spatial resolution of ca. 30–200 nm for elements with edges of between  $\sim 2$ –15 keV. Quick XAFS (QXAFS) measurement systems are available at each BL except AR-NW2A. The dispersive XAFS (DXAFS) measurement system is available at AR-NW2A, and we intend to promote dynamic studies by improving the DXAFS system. We have developed the laser pump–DXAFS probe method, paving the way for a new field of dynamics research. We have also developed a new surface-sensitive XAFS measurement method, the named Kramers-Kronig reflection XAFS (KK-XAFS) method. This is a relatively easy way to obtain surface-sensitive XAFS spectra by detecting signals of total reflection. The reflection spectra are transformed to “absorption” spectra based on Kramers-Kronig relations. The method is useful for studying the evolution of surface states during chemical reactions. The method can be combined with the DXAFS system, and thus fast dynamics studies of surfaces in the hard X-ray energy region will emerge.

## 4-5 Life Sciences Group

### 4-5-1. Structural Biology Research Center (SBRC)

The SBRC is playing a major role in a national project of structural biology, Platform for Drug Discovery, Informatics, and Structural Life Science (PDIS), which was launched with the support of MEXT in FY2012. The platform consists of three cores, “Structural Analysis”,

“Regulation”, and “Informatics”. The SBRC has been leading the Structural Analysis core. With the support of the PDIS project, the SBRC has improved BL-1A for sulfur-based single-wavelength anomalous diffraction (S-SAD), which is a new phasing method in protein crystallography. We are also developing a standard experimental protocol for S-SAD data collection. For small-angle X-ray scattering (SAXS) experiments, a solution sample changer was installed in BL-15A2 and a new program for SAXS experiments, SAngler, has been developed. BL-17A for protein crystallography was extensively upgraded in FY2014 with supplementary budget. The optical layout was modified and a new X-ray detector, Pilatus S6M, was installed. Pilatus S6M, which is a pixel array detector with a large active area (423.6  $\times$  434.6  $\text{mm}^2$ ), has short readout time (2.03 msec) and a high frame rate (25 Hz at maximum), enabling us to perform shutterless data collection and fine-phi slice data collection.

### 4-5-2. Upgrade of macromolecular crystallography beamline, BL-17A

BL-17A is one of the macromolecular crystallography beamlines at the Photon Factory. The beamline was constructed as the first short-gap undulator beamline at the Photon Factory in 2006, and thanks to this light source and dedicated optics, BL-17A was characterized as a beamline for diffraction experiments with small crystals [1]. Since the beamline was opened to general users, it has contributed to a large number of significant achievements on important biological problems. However, the recent trend in macromolecular crystallography is moving to much more difficult targets, and so users’ demands for the beamline have become much higher. In 2014, we upgraded the beamline to deliver a much more highly focused beam at the sample position and installed a new diffractometer and detector in the end station.

For this upgrade, the optical layout needed to be changed in order to achieve a more focused beam at the sample position. Several layout plans were con-

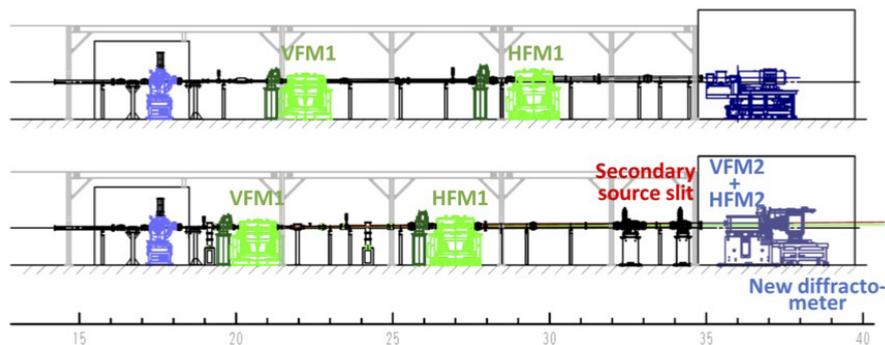


Figure 7: Optical layout of BL-17A before (upper) and after (lower) the upgrade.

sidered and we finally decided to employ a secondary source approach (Fig. 7) [2]. In this plan, the existing two mirrors which were used to focus the X-ray beam to the sample position vertically and horizontally, respectively, are moved upstream to make the secondary sources upstream of the end station. New bimorph KB mirrors are installed just upstream of the sample position to focus the beam from the secondary source to the sample position. Ray-trace simulation suggested that the beam size becomes much smaller with reasonable divergence (Fig. 8).

As the beam size decreases, the stability of each optical component becomes more important. Any small movements in the optical components may affect the beam intensity at the sample position, and therefore great care was taken to ensure the stability of the optical components. All newly installed devices have heavy granite base tables to damp the vibration transmitted from the floor. Particular sections of the experimental floor where the monochromator and mirrors are installed, were dug up and refilled with thicker reinforced concrete (Fig. 9).

The changing of optical layouts and installation of new devices will be finished in April 2015. After the

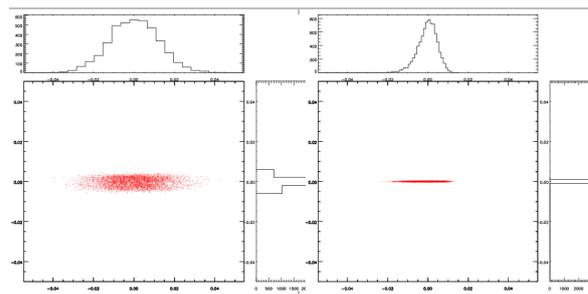


Figure 8: Ray-trace simulation of BL-17A with optical layouts (a) before and (b) after the upgrade. The beam sizes and divergences (H × V) are  $284 \times 52 \mu\text{m}^2$  and  $0.39 \times 0.096 \text{ mrad}^2$ , respectively, before the upgrade and  $86 \times 8.4 \mu\text{m}^2$  and  $1.47 \times 0.7 \text{ mrad}^2$ , respectively, after the upgrade.

beamline is commissioned, which is scheduled to be performed in May 2015, the beamline will be opened for general academic and industrial users in June 2015.

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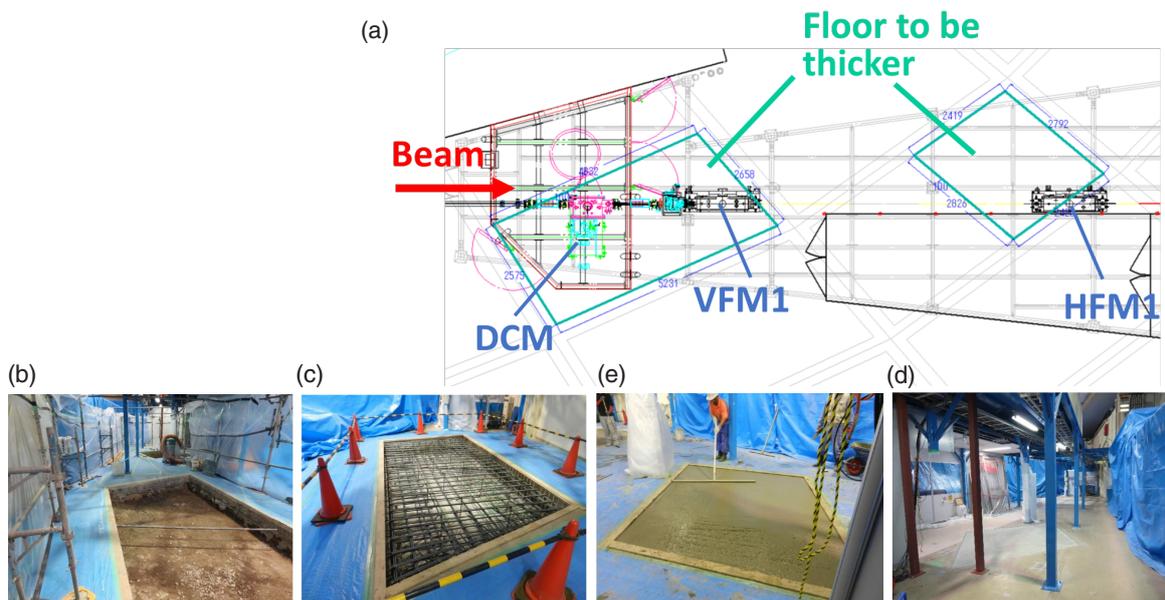


Figure 9: Reinforcement of experimental floor. (a) Sections of the experimental floor to be reinforced. (b)–(e) photographs of the reinforcement work.

### 4-5-3. New BL-10C: The result of commissioning

BL-10C is a small-angle X-ray scattering (SAXS) beamline. This beamline has been used for over 30 years from 1984. In FY2013, we scrapped and rebuilt it to replace almost all the components [1]. We then optimized and aligned the components in May 2015. We saw the first beam on the fluorescence screen monitor after the renewal on May 16 (Fig. 10). Figure 11(a) shows the photon flux at the sample position. Users can basically use X-ray energies from 6.5 to 14 keV. It is necessary to change the glancing angle of the focusing mirror to use energies below 6.5 keV because of the cutoff of the higher-order X-rays. Since the beam position moves only 0.03 mm in this energy range when changing the X-ray energy, users can easily change the energy without tuning by using the GUI software [Fig. 12(b)]. The energy is calibrated at 1.488 Å (Ni-K edge) as before. Figure 12 shows the beam shape at the focal position. The beam size is  $V0.18 \times H0.63$  mm (FWHM). The theoretical value of the beam size is  $V0.15 \times H0.59$  mm by raytracing. Therefore, the beam size is

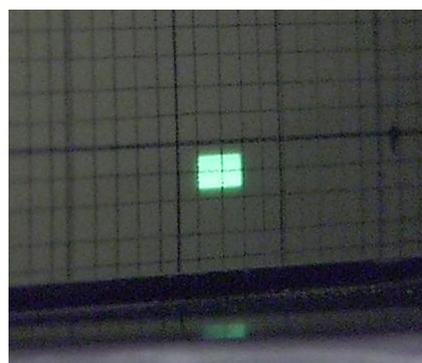


Figure 10: The first beam seen behind the monochromator.

almost identical to that calculated by raytracing. Figure 13 shows the scattering pattern of a standard sample, silver behenate, measured at the condition of 1.488 Å and 1 m camera length. BL-10C was opened to users in June 2014.

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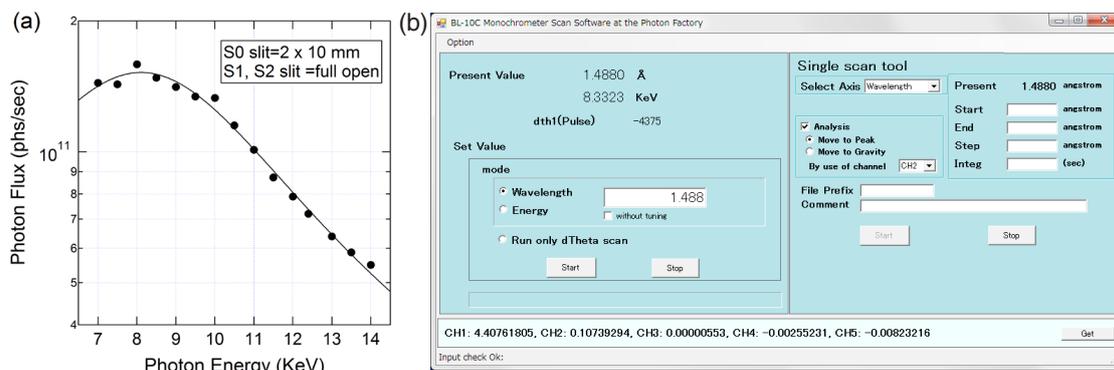


Figure 11: (a) Photon flux at the sample position, (b) GUI software for changing the measurement X-ray energy developed.

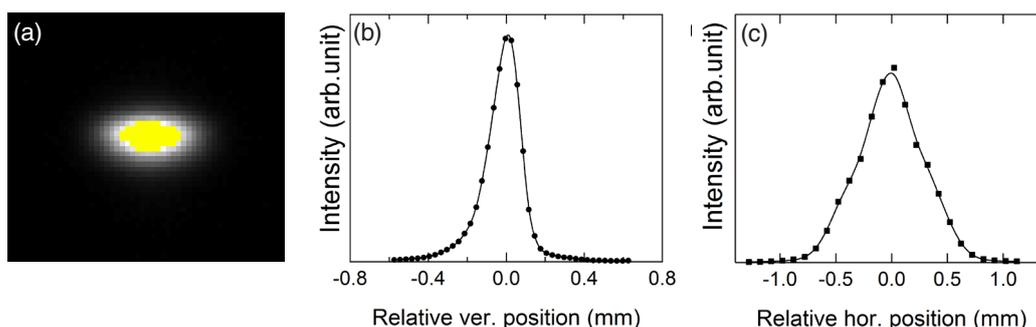


Figure 12: (a) Beam shape at the focal position recorded on the flat-panel detector (C10013SK, Hamamatsu). The X-ray beam was attenuated by 0.1 mm of Cu, (b) and (c) The line profiles in the vertical and horizontal directions, respectively. These profiles were calculated with the results of one-direction scanning of the slit blade.

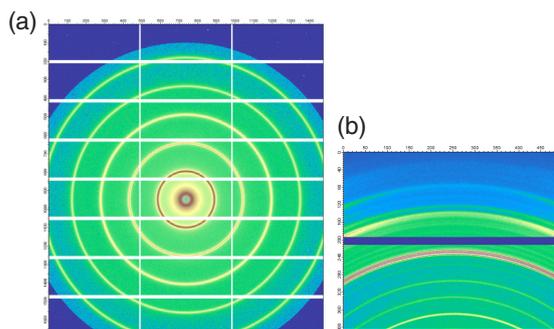


Figure 13: The scattering pattern of silver behenate ( $d = 58.38$  Å). (a) SAXS, (b) WAXD.

## 4-6 Operation Status of BL-15A

### 4-6-1. BL-15A, dedicated to semi-microbeam XAFS/XRF/XRD and high-brilliance SAXS/GISAXS studies

As reported previously, the construction of the new undulator beamline BL-15A was completed in FY2013. This beamline has a tandem beamline arrangement for both XAFS/XRF/XRD studies using semi-microfocus beams (station A1) and SAXS/GISAXS experiments using collimated softer and hard X-rays (station A2). At station A1, the semi-microfocus beams available in a wide range of photon energies (2.1–15 keV) allow analysis of the local structures of the elements and valence for inhomogeneous samples in the fields of environmental science and new energy source science. A new simultaneous XAFS/XRF/XRD measurement system was developed for grid-pattern element mapping and in situ experiments with the combined use of XAFS/XRF and XRD [Fig. 14(a)]. At station A2, the collimated beams are used for structural studies of functional membranes, large hierarchical structure analysis of soft matter and high-throughput solution structure determination of biological systems. Two experimental stages, a conventional SAXS/WAXS stage and a low-energy GISAXS stage, are placed in tandem in the station and a vacuum-compatible X-ray area detector, PI-

LATUS3 2M, is equipped [Fig. 14(b, c)]. The detector is directly connected to the GISAXS stage and low-energy GISAXS experiments of up to 2.1 keV can be performed without any filter in a vacuum environment.

The first beams were introduced into both stations in FY2013. We subsequently started the alignment of beamline optics and the characterization of beam performance. The beam profiles at the three focal points, 32.8 m, 36.75 m and 42.75 m from the source, are shown in Fig. 15. The undulator spectra and the photon flux were measured and the results are almost consistent with the ray-tracing simulation. Next, we conducted commissioning experiments in the spring of 2014. Figure 16 shows some results of these experiments, XANES mapping analysis of the reduction of sintered ores at the Fe K-edge (station A1), the diffraction pattern from a dried chicken tendon and the GISAXS image from diblock PS/PMMA copolymers in a vacuum environment (station A2). The results were promising. After the commissioning experiments, BL-15A was opened to users in the autumn of 2014.

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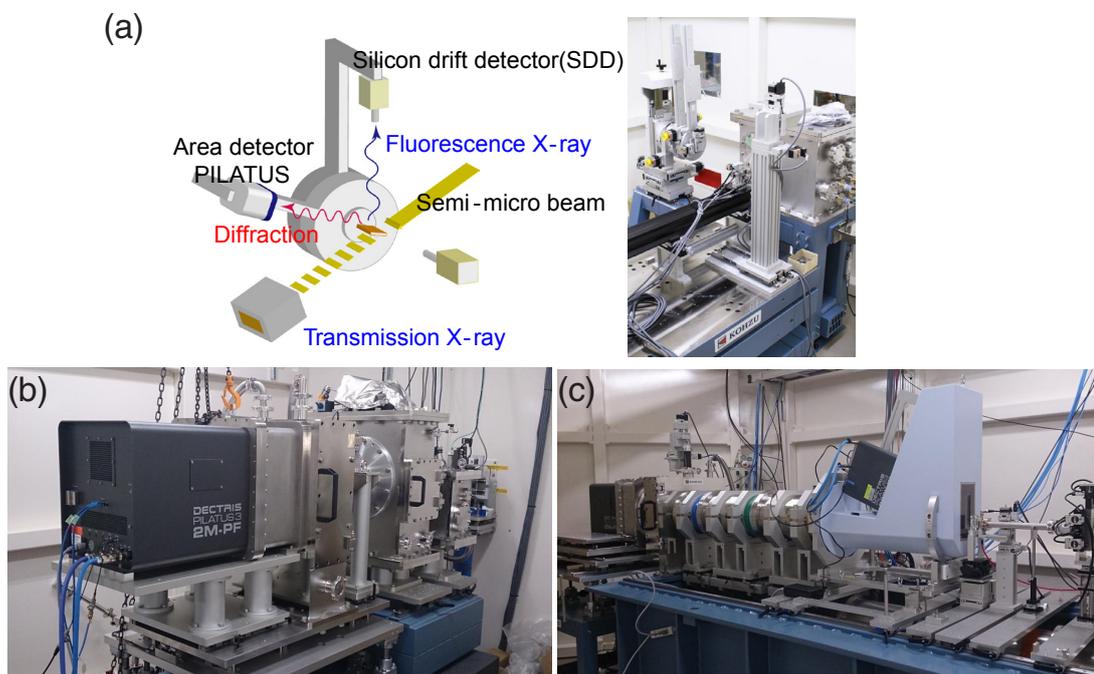


Figure 14: (a) Measurement geometry and simultaneous XAFS/XRF/XRD measurement system in station A1. (b) Low-energy GISAXS stage in the upstream of station A2. (c) Conventional SAXS/WAXS stage in the downstream of station A2.

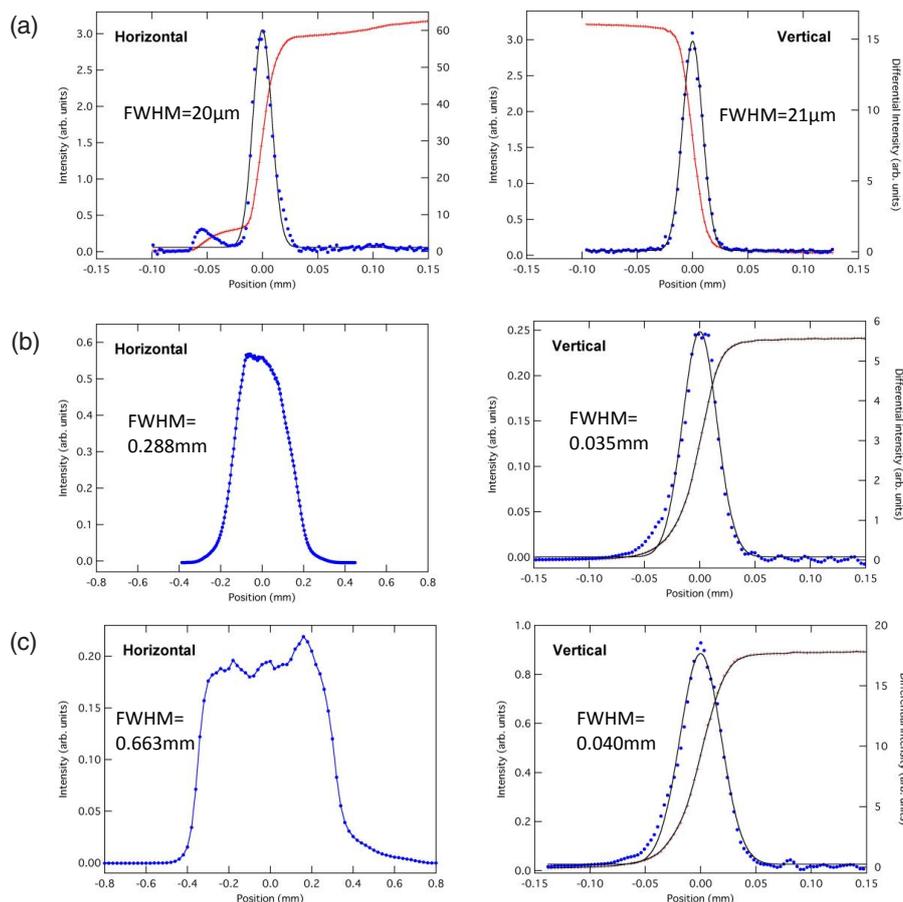


Figure 15: Beam profiles at three focal positions (10.4 keV, 0.1 mm width of the secondary light source): (a) Station A1 at 32.8 m, (b) GISAXS stage at 36.75 m and (c) SAXS/WAXS stage at 42.75 m.

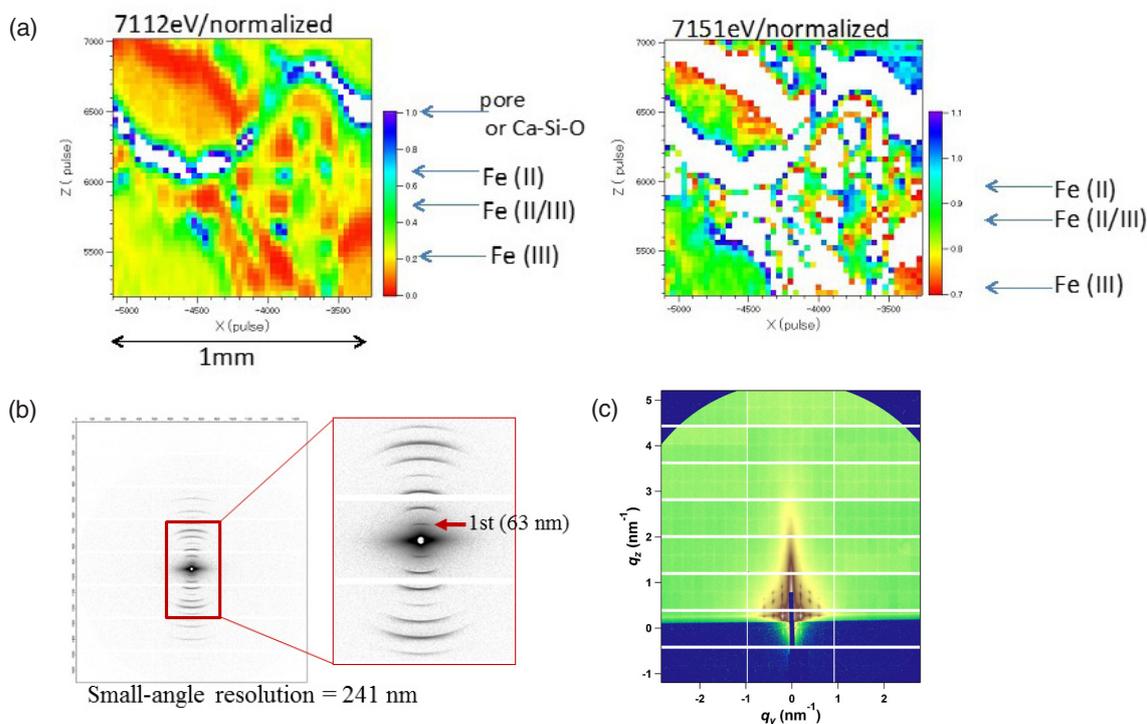


Figure 16: Commissioning experiments. (a) Station A1: XANES mapping analysis of reduction of sintered ore at the Fe K-edge in transmission mode (courtesy of Dr. Reiko Muraio, Nippon Steel & Sumitomo Metal Corporation). The beam size is 20  $\mu\text{m}$  square and the step size is 40  $\mu\text{m}$ . (b) Diffraction pattern from a dried chicken tendon. The X-ray energy is 10.4 keV and the sample–detector distance is 3657 mm. (c) GISAXS image from diblock PS/PMMA copolymers. The X-ray energy is 3.6 keV and the sample–detector distance is 825 mm.