1 Newly Developed Experimental Facilities

1-1 Overview

High-performance insertion devices and beamlines are currently highly demanded for increasingly sophisticated scientific research. Since the number of straight sections at the Photon Factory was initially limited, some beamlines were constructed as hybrid beamlines, where a single insertion device was used as an undulator for the soft X-ray region and as a multi-pole wiggler(MPW) for the hard X-ray region. Various new research activities emerged with hybrid use since many different experiments could be carried out, which was a good style in the early days. However, with the growth of synchrotron sciences, it has become essential to prepare application-specific beamlines and end-stations. We thus modified the lattice of the 2.5 GeV PF-ring and adopted the strategy of assigning five long and medium-length straight sections to experiments requiring soft X-rays, and the remaining two medium-length sections and four short ones for those requiring hard X-rays. This was realized in conjunction with improvements of the PF-AR, since some of the X-ray activities could be moved there. The straight-sections upgrade project of the 2.5 GeV PF-ring was successfully completed in FY2005. Some insertion-device beamlines have been reconstructed: BL-28A in 2004, BL-17A in 2005, BL-3A and 28B in 2006, and BL-16A in 2007. Following this trend, two new beamlines, BL-1A and 13A, were constructed using undulator sources and some related upgrades were carried out in FY2009. On the other hand, the beamlines in the PF-AR North East (NE) experimental hall were refurbished in FY2008; beamlines NE1 and NE3 have been reconstructed and NE5A has been decommissioned.

A new structural biology beamline BL-1A was constructed, with funding by the “Target protein” national project. Structural analyses of small crystals less than 10 µm can be carried out at BL-1A by means of 4 keV soft X-rays, enabling SAD experiments using anomalous signals of sulfur. The combination of a short-gap undulator and a carefully designed beamline achieved the designed experimental conditions. BL-1A is planned to be opened to project team users from May 2010. BL-1 had three branch beamlines using dipole radiation before the new beamline was constructed. Two of these (BL-1B and 1C) were shifted or decommissioned in FY2008, as mentioned in the previous volume. A remaining branch beamline for condensed matter physics using X-ray diffraction, BL-1A, was shifted to BL-8A in March 2009. The commissioning of the new BL-8A progressed well, and it has been open to public users since June 2009.

BL-13 was a typical hybrid beamline, which contained two branches that use MPW radiation for hard X-ray experiments and a branch that uses undulator radiation for soft X-ray experiments. It was decided to reconstruct BL-13 as a dedicated beamline for the study of functional organic materials using the existing undulator. Old beamlines were decommissioned during the spring shutdown, construction of the new BL-13A was completed in September 2009, and it was opened to public users from late January 2010. The new BL-13A provides synchrotron radiation between 30 and 1200 eV. Functional organic materials are being intensively developed since the usable resources of noble metals that are now considered important in the chemical and electrical industries are not sufficient to meet future demands such as those for electric vehicles. BL-13A is expected to play an important role in the study and analysis of these materials.

The second APPLE-II type variable polarization undulator is being constructed in order to realize fast polarization switching at BL-16A; the construction is progressing with the financial support from the quantum beam program of MEXT. Electron orbit switching is also being tested using five kicker magnets. These techniques are combined in order to conduct research on faint polarization dependent signals using a lock-in amplification technique. The second undulator will be installed during the summer shutdown of 2010.

BL-14 possesses a superconducting wiggler that emits vertically polarized hard X-rays. Because of this polarization property, BL-14 is suitable for phase-contrast imaging experiments that require a precise X-ray interferometer. In order to station the sensitive interferometer and dedicate BL-14C to phase-contrast imaging experiments, a large high-pressure anvil, MAX-III, stationed at BL-14C2, was moved to the newly constructed AR-NE7A during the 2009 summer shutdown. With this rearrangement, users can use beam time more efficiently since they are freed from the laborious setup of the sensitive interferometer and the time required for its stabilization. The beam time of AR-NE7A is shared with X-ray imaging such as microangiography that was transferred from the old AR-NE5A.

The commissioning of a new structural biology beamline AR-NE3A and its end station funded by Astellas Pharma Inc. was successfully completed, and NE3A has been opened to public users since April 2009. The exposure time to obtain the same or higher quality data is one third of that at NW12A. Thus, a higher throughput can be realized. The automation of crystal change and subsequent data acquisition has progressed, and remote control of the system has now been tested.

A station for X-ray diffraction under high-pressure and high-temperature environments, combined with nuclear resonance capabilities, has been constructed at NE1A, and the activities formerly carried out at BL-13A were transferred here. Following commissioning of the beamline and its end station, the new NE1A has been opened for public use.

1-2 BL-1A: A Newly Constructed Macromolecular Crystallography Beamline

Recent advances in SAD (single anomalous dispersion) phasing techniques have facilitated solving macromolecular crystal structures using weak anomalous signals from sulfur atoms or light atoms which are naturally included in macromolecules. The method called S-SAD (Sulfur-SAD) would be particularly useful in solving membrane proteins or macromolecular complexes, for which heavy atom or selenomethionine derivative crystals are difficult to prepare. The Structural Biology Research Center at the Photon Factory has been developing a new beamline dedicated to S-SAD experiments under the national project 'Targeted Proteins Research Program'. The beamline should be designed to take full advantage of lower energy X-ray beams at around 4 keV to enhance anomalous signals from light atoms. In addition, the beam is highly demagnified to achieve a small beam size at the sample position, so experiments using micron-size crystals would be possible.

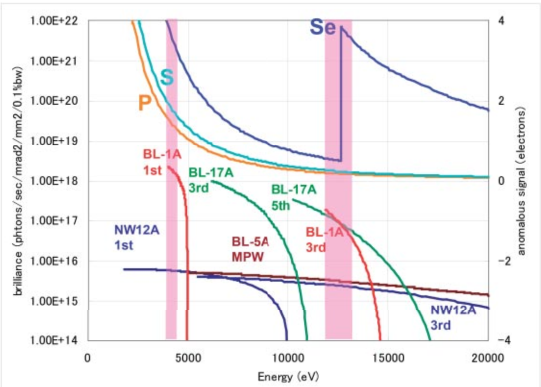

Figure 1
Energy spectrum of the light source of BL-1A, together with the ones of the other Macromolecular Crystallography (MX) beamlines. The energy dependence of the anomalous signals from selenium, phosphorus and sulfur is also shown.

The BL-1A has been constructed at the BL-1 site where three bending magnet branch beamlines were operated previously. The light source is an in-vacuum short gap undulator (SGU#01) newly installed at the short straight section between B#28 and B#01, optimized at around 4 keV with its fundamental harmonics (Fig. 1). The vacuum section of the beamline has only one beryllium window with the thickness of 0.2 mm, followed by a diffractometer equipped with a helium cryostream and a specially designed helium chamber to minimize the loss of the lower energy beam. Simple optics (a cryo-cooled channel-cut monochromator and bimorph Kirkpatrick-Baez (KB) focusing mirrors) are adapted to deliver a well-focused small-sized beam with good stability (Fig. 2). It should be noted that the energy range of 12–13 keV is also covered with the 3rd harmonics. Thanks to the good energy resolution of the cryo-cooled monochromator, conventional MAD (Multiple wavelength Anomalous Dispersion) experiments using micron-sized derivative crystals with Se, Hg, Au, or Pt atoms could be executed.

The construction of BL-1A started in April 2009, and was almost completed by January 2010. After commissioning, it will be opened to project users from May 2010 and to general users from October 2010. Currently, the focused beam size (FWHM) at the sample position is 70 µm (H) × 10 µm (V), which is slightly larger in the horizontal direction than expected. The measured and calculated beam intensities at 12 keV are in good agreement, in the order of 10^{10} photons/sec on an area of 10 µm square. We will proceed to optimize the beam focusing and equipment for S-SAD experiments to solve difficult macromolecular structures.

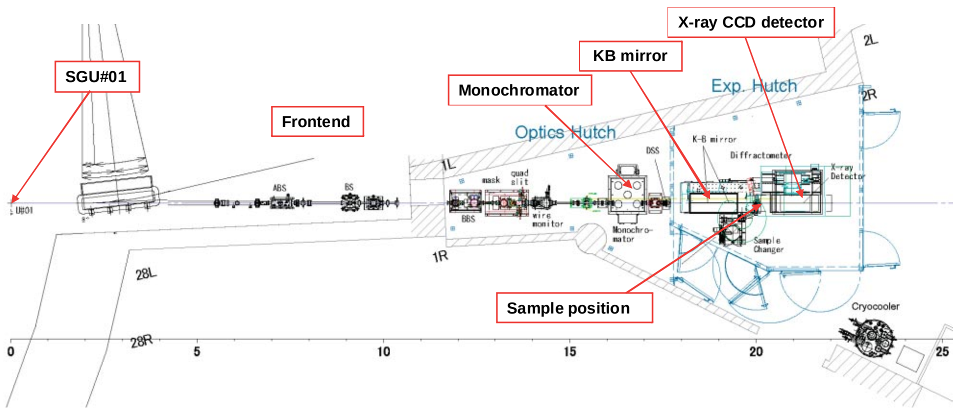

Figure 2
Schematic drawing of the BL-1A.

1-3 BL-8A: A New Beamline for Structural Materials Science

BL-8A dedicated for structural materials science was constructed at the Photon Factory, to which the activities carried out at BL-1A were moved. BL-8A was designed to perform X-ray single-crystal structure analysis and powder-diffraction experiments similar to BL-8B previously reconstructed.

The construction of BL-8 began in FY2007. BL-1A was closed in December 2008 after generic user operation commenced at BL-8B to prevent a gap without either being available. BL-8A was commissioned during January–May 2009 and user runs successfully began in June 2009.

Figure 3 shows the schematic layout of BL-8, including stations A and B. Synchrotron radiation emitted from a bending-magnet is split into BL-8A and 8B by the fixed masks of the front end. On the newly-constructed BL-8A, the incident beam is monochromatized by the Si(111) double-flat crystal monochromator located at 13.972 m from the light source. The monochromatic beam is focused at the sample position located at 29.0 m from the source by a newly designed Rh-coated bent cylindrical quartz mirror located at 17.5 m. The mirror is used to focus the beam in the vertical direction. The glancing angle of the mirror is 3.2 mrad, thus the beamline optics of the new BL-8A cover a photon energy range of 5 to 19 keV. The focused beam size and the typical photon flux at the sample position are 0.82 mm (horizontal) × 0.52 mm (vertical) and 3.2×10^{11} photons/sec at 12.4 keV (Table 1).

The Weissenberg camera type imaging-plate (IP) diffractometer (Rigaku Co.) with a camera having a radius of curvature of 191.3 mm was installed. We obtain X-ray scattering data over the 2θ range of −60 to 145˚ with a step width of 0.03˚. A helium/nitrogen gas open-flow-type cryostat (20–373 K/ 100–373 K) is used as the permanent cooling system. In addition, the following equipment is available: a closed cycle helium cryostat with a beryllium window producing no background scattering (10–300 K), a diamond anvil cell connected to a refrigerator (10–300 K, up to 50 GPa), and a furnace (300–1000 K). All instruments are controlled by a standard RIGAKU program running on a Windows machine.

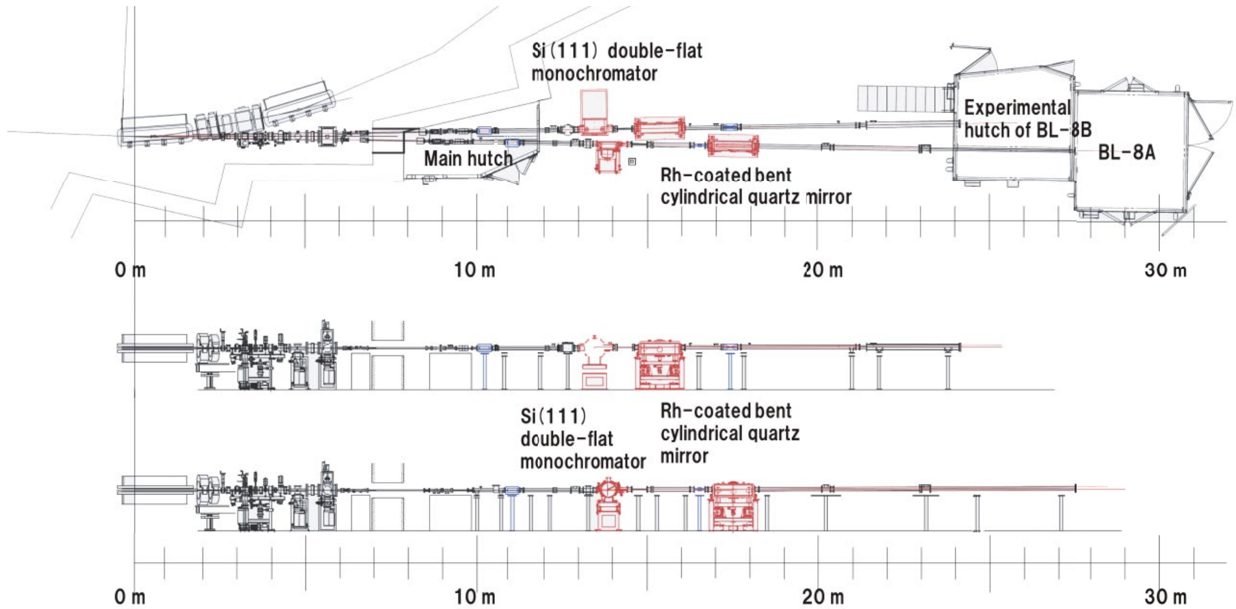

Figure 3
Schematic layout ot BL-8.

Table 1 Preliminary characteristics of the beam of BL-8A.

	BL-8A
Photon energy (keV)	5-19
Horizontal acceptance (mrad)	2.22
Vertical acceptance (mrad)	0.406(12.4 keV) 0.589(18 keV)
Energy resolution (ΔE / E)$\times 10^{-4}$	3.67(12.4 keV) 3.65(18 keV)
Photon flux at the sample posi tion (ph /s) 400mA	3.2×10^{11}(12.4 keV) 8.8×10^{10}(18 keV)
Beam size (H (mm) × V (mm))	0.82 × 0.52

1-4 BL-13A: Vacuum Ultraviolet / Soft X-Ray Undulator Beamline for the Study of Organic Thin Films and Biomolecules Adsorbed on Surfaces

Recently, organic thin films and biomolecules adsorbed on surfaces have attracted the interest of many researchers. An understanding of the structure and electronic properties of their surfaces and interfaces is important not only in fundamental science but also in industry, because applications of organic electronics such as electroluminescent devices and biosensors are rapidly growing. To contribute to this field we have constructed a new vacuum ultraviolet / soft X-ray undulator beamline, BL-13A, covering the photon energy region of 30–1,000 eV [1]. An ultrahigh vacuum chamber equipped with an SES 200 analyzer (Gamma Data / Scienta) is used as the main end station. The main scientific targets are investigations of organic thin films and biomolecules adsorbed on well-defined surfaces using angle-resolved photoelectron spectroscopy (ARPES), high resolution X-ray photoelectron spectroscopy (HR-XPS) and X-ray absorption spectroscopy (XAS).

The light source of BL-13A is a planar undulator [2]. We adopted a Monk-Gillieson-type monochromator with two varied-line-spacing plane gratings (plane VLSGs) and a variable-included-angle mechanism [3]. The layout of BL-13A is schematically illustrated in Fig. 4. It consists of a focusing pre-mirror (M1), a plane mirror (M2), plane VLSGs, an exit slit (S), and a focusing post-mirror (M3). The 300- and 1,000-l/mm VLSGs cover the photon energy region of 30–300 eV and 100–1,000 eV, respectively.

Figure 5 shows the energy resolution (E/ΔE) in the simultaneous scan of M2 and VLSGs, estimated with analytical calculations assuming the acceptance angles of 0.4 mrad × 0.2 mrad (vertical × horizontal) for the light source. Figure 6 shows the calculated photon flux when the width of the exit slit is adjusted to achieve E/ΔE of 5,000 and 10,000. Figure 7 shows an example of the spot size estimated with ray-tracing simulations at the sample position, when the width of the exit slit is set at 40 µm.

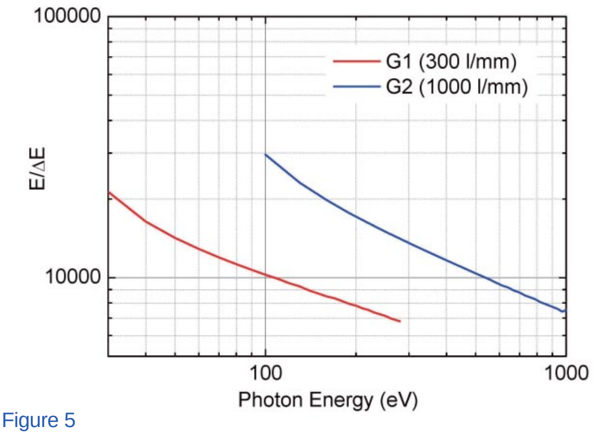

Figure 5
Energy resolution (E/ΔE) in the simultaneous scan of M2 and VLSG, estimated with analytical calculations [1].

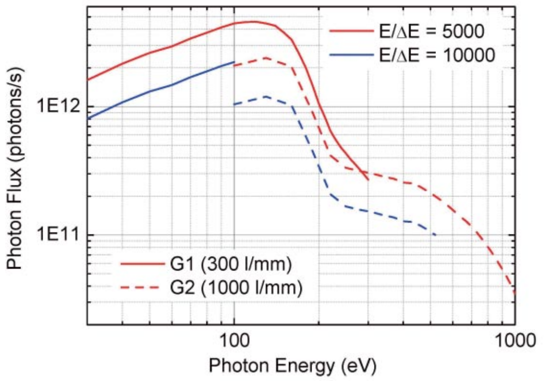

Figure 6
Photon flux estimated with analytical calculations when the exit slit is adjusted to achieve the energy resolution (E/ΔE) of 5,000 and 10,000 [1].

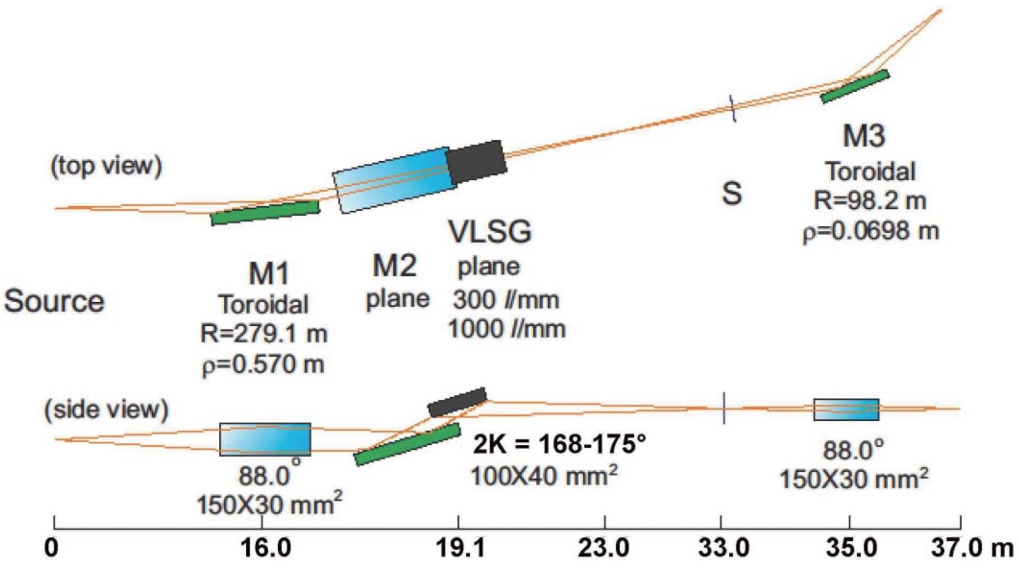

Figure 4
Schematic layout of BL-13A [1].

BL-13A was constructed from July 2009 to Sep. 2009 (Fig. 8). After careful baking for three weeks, BL-13A achieved a base pressure of ≤ 1 × 10^{-8} Pa. The commissioning of BL-13A started in October 2009, and the beamline has been open for users since January 29, 2010.

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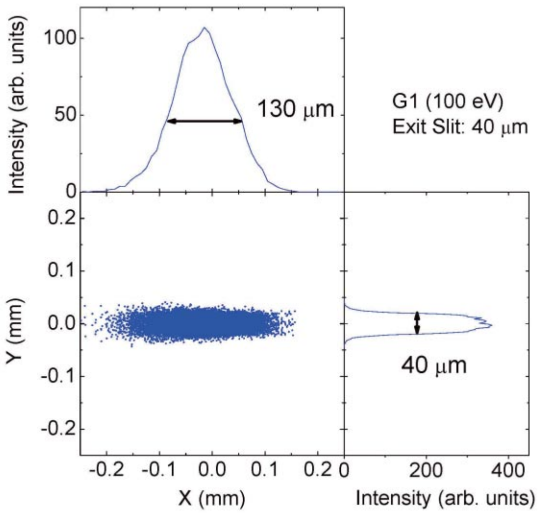

Figure 7
Spot size at the sample position estimated with ray-tracing simulations [1].

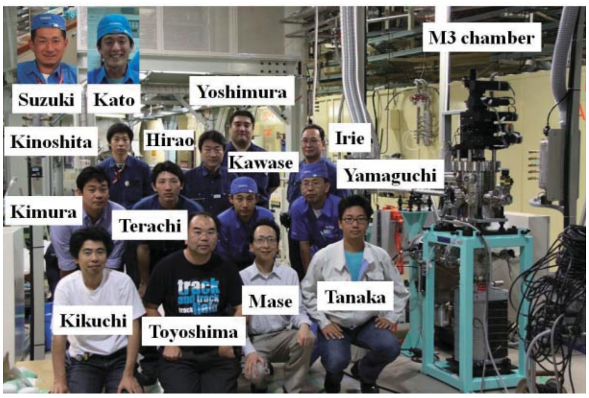

Figure 8
Members contributed for BL-13A construction.

1-5 AR-NE1A for X-Ray Diffraction and Mössbauer Spectroscopy Experiments at High Pressure

In early January 2009, commissioning of the X-ray optical system of the new beamline AR-NE1A started without installing the horizontal focusing mirror. During the commissioning period some high-pressure diffraction experiments were carried out as a trial. At the end of May, the whole beamline was completed and user operation started.

The beamline optics consist of two kinds of monochromator and two K-B type focusing mirrors. A double crystal monochromator was installed on the upstream side. It has a micro channel type water-cooled first crystal in order to withstand the high heat load from the high-power MPW. The other one is a high-resolution monochromator with two channel-cut crystals of Si(4,2,2) and Si(12,2,2) for Mössbauer spectroscopy. The main feature of this beamline is the system of changing over the focusing optics. Two multilayered mirrors, named a tri-chromatic mirror, were newly developed for three discrete energies of 14.4 keV, 30 keV and 50 keV at the same diffraction angle. The first energy is for nuclear resonance of ^{57}Fe and the latter two energies are for X-ray diffraction experiments with a wide-Q range. Three W/C multilayer strips are fabricated on an Si substrate. Each strip is made out of 10, 40 and 60 pairs of tungsten and carbon layers with periodic length of 10.2 nm, 4.2 nm and 2.4 nm, respectively. The details and concept of the beamline AR-NE1A are described in the previous report [4].

In order to utilize the high intensity and high energy single-bunch SR source of the PF-AR, X-ray diffraction or Mössbauer spectroscopy experiments under high pressure have been carried out in the first stage. Under conditions of low temperature and high pressure, time-domain ^{57}Fe nuclear resonant forward scattering (NFS) experiments have been carried out by using the 14.4-keV single-bunch SR. Figure 9 shows quantum beat signals from ^{57}Fe nuclei of $EuFe_2As_2$ sample in a minia-

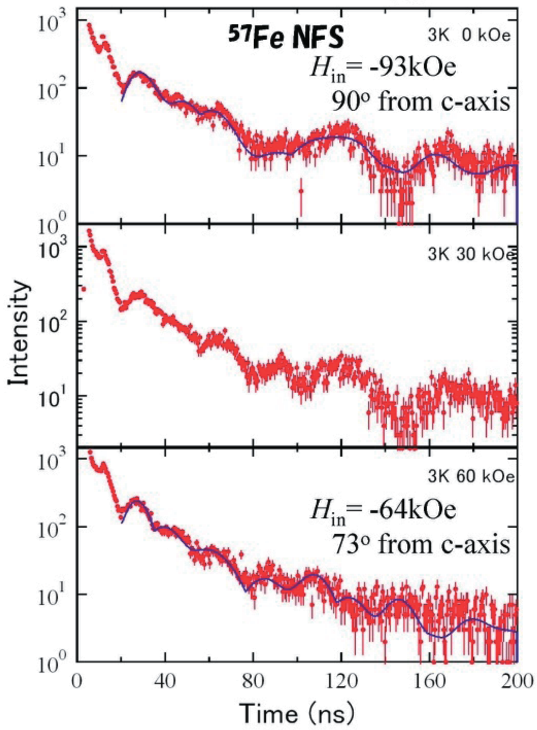

Figure 9
Time-domain ^{57}Fe NFS spectra of $EuFe_2As_2$ at low temperature(T=3K) and high pressure (P=1.6 GPa) with external magnetic field. The Quantum beats in the spectra showing a magnetic ordering in $EuFe_2As_2$.

ture DAC [5]. Figures 10 and 11 show powder-diffraction profiles recorded on IP of the same CeO_2 sample in a diamond anvil cell (DAC) obtained by focused 30-keV SR and higher energy 50-keV SR [6]. As the next stage, in FY2010 we are planning to combine these two methods of measurement for one sample under the same pressure and temperature conditions.

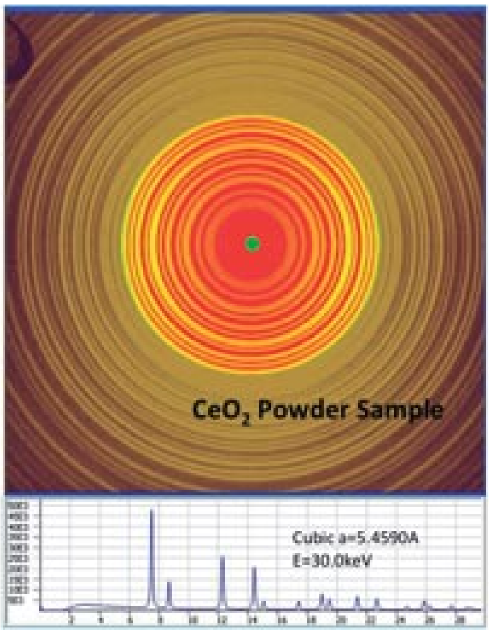

Figure 10
Diffraction profile of CeO_2 sample showing many diffraction peaks. It was obtained by using normal energy (30 keV) SR focused by multilayered mirrors. The incident beam was shaped by a pin-hall of 30 microns in diameter.

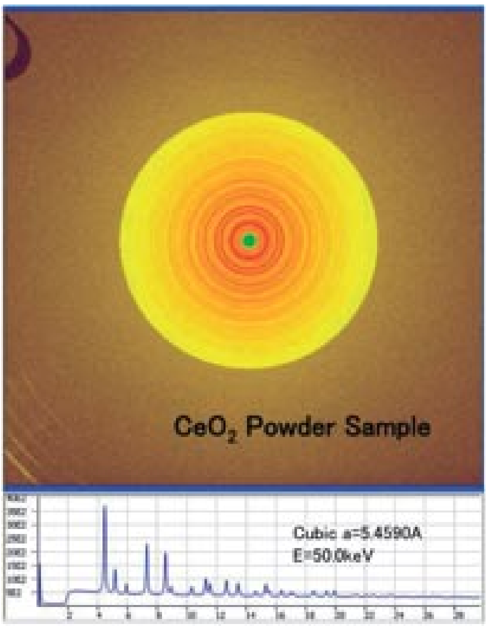

Figure 11
The diffraction profile of CeO_2 sample, the same as in Fig. 10, indicates many diffraction peaks in a wide Q-range (see lower column). It was obtained by using higher energy (50 keV) SR focused by multilayered mirrors. The incident beam was collimated by a pin-hall of 50µm square size.

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[6] Courtesy of Prof. N.Funamori, Dr. T.Sato and Mr. D.Wakabayashi (The University of Tokyo).

1-6 Construction of AR-NE7A and Rearrangement of BL-14C for X-Ray Imaging and X-Ray Diffraction Experiments under High Pressure and Temperature

Based on suggestions by the Photon Factory International Science Advisory Committee (PF-ISAC) and the Medical Applications ISAC Subcommittee, we have rearranged all beamlines and experimental stations for medical applications, including stations NE1A2 (MPW), which was dedicated to clinical applications, and NE5A (BM), which was dedicated to X-ray imaging, at the PF-AR NE experimental hall. NE1A2 was rearranged to perform other kinds of experiments in 2008 after the termination of the project on intravenous coronary angiography in a collaborative work between the University of Tsukuba and the Institute of Materials Structure Science. After disassembly of NE5A, which was one of the oldest stations at the PF-AR, we constructed a new bending magnet beamline, NE7A (BM), at the PF-AR NE experimental hall using the parts of beamline components of NE5A to begin experiments in 2009 on X-ray diffraction under high pressure and temperature, and X-ray absorption-contrast imaging, such as micro-angiography, monochromatic X-ray CT, and X-ray fluorescent CT. The large experimental equipment, MAX III, for the high-pressure and -temperature experiments, was transferred from BL-14C2 to NE7A to be installed at the back of the NE7A hutch, where the available photon flux of 50 keV is approximately twice as large as that available at BL-14C2. Figure 12 shows the plan view of NE7A at the PF-AR NE experimental hall. The main hutch for installing an X-ray double-crystal monochromator and a Downstream shutter (DSS), and an experimental hutch, where either a white or monochromatic beam can be delivered, were constructed at the experimental hall. This beamline covers the photon energy of 25 to 60 keV using the Si(111) double-crystal monochromator. In addition, we have rearranged BL-14C at the PF experimental hall, where a vertically polarized synchrotron radiation beam can be obtained, to investigate X-ray phase-contrast imaging, such as X-ray phase-contrast CT using an X-ray interferometer, X-ray diffraction-enhanced imaging (DEI), DEI-CT and X-ray dark field imaging (DFI). BL-14C1, which was dedicated to X-ray imaging, and BL-14C2, which was dedicated to X-ray diffraction experiments under high pressure and temperature, were rearranged to a large single experimental hutch, BL-14C, for X-ray imaging. A large X-ray interferometer for X-ray phase-contrast CT was installed at the back of the BL-14C hutch. Figure 13 shows the plan view of BL-14C at the PF experimental hall, and Table 2 summarizes the specifications of NE7A and BL-14C.

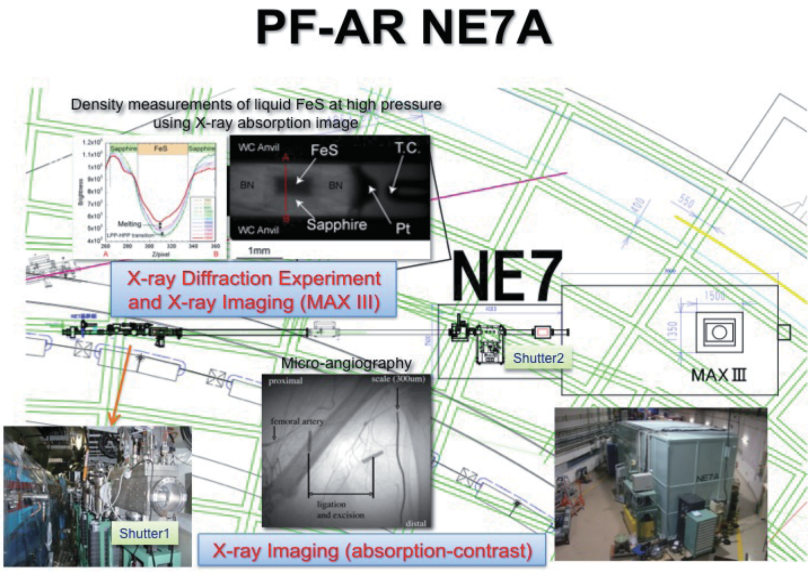

Figure 12
Plan view of NE7A.

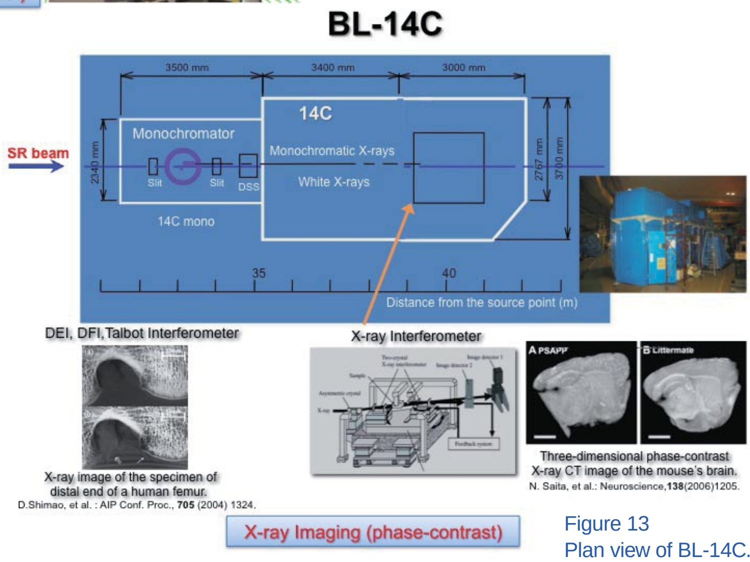

Figure 13
Plan view of BL-14C.

Table 2 Specifications of NE7A and BL-14C.

Experimental station	PF-AR NE7A	BL-14C
Source	Bending magnet	Vertical wiggler
X-ray beam size	3 mm (V) by 80 mm (H) @ 20 m	70 mm (V) by 6 mm (H) @ 38 m
X-ray optics	Si(220) double-crystal asymmetric crystals	Si(111) double-crystal asymmetric crystals X-ray interferometer Laue crystal
Photon flux at 30 keV	10^8 photns/mm^2/sec	10^8 photns/mm^2/sec
Spectral range	White X-rays Monochromatic X-rays: 25-60 keV	White X-rays Monochromatic X-rays: 8-80 keV
Experiments	X-ray diffraction experiments under high-pressure and high-temperature Micro-angiography Monochromatic X-ray CT Fluorescent X-ray CT	Diffraction enhanced imaging (DEI) Dark-field imaging (DFI) DEI-CT Imaging using an X-ray interferometer Talbot interferometer