1 Newly Developed Experimental Facilities

1-1 Overview

High-performance insertion devices (ID) 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 for the hard X-ray region. Various new research activities have been born 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 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 X-ray activities could be moved there.

The straight-sections upgrade project of the 2.5-GeV PF-ring that aimed at creating more space for insertion devices was successfully completed in FY2005. Following the strategy described above, some of the ID beamlines have been reconstructed; BL-28A in 2004, BL-17A in 2005, BL-3A and 28B in 2006, and BL-16A in 2007. Among the reconstructed beamlines, BL-17A and 3A use in-vacuum short-gap undulators (SGU) to provide hard X-rays from the 2.5-GeV storage ring.

New BL-16A aims to measure faint polarization-dependent signals by using the lock-in amplification technique. Two polarization-tunable undulators and a fast bump system consisting of five kicker magnets realize polarization switching at a frequency of ~10 Hz. The first APPLE-II undulator and kicker system set were installed during the spring shutdown in 2008. Circularly, elliptically and linearly polarized soft X-rays with photon energies between 200 and 1500 eV can be provided at this beamline with a fairly high energy resolution, $E/\Delta E \geq$ 5000. The commissioning of the new BL-16A started in May 2008, the first MCD spectrum was recorded in June, and the beamline has been opened for public users since October. The second APPLE-II undulator is to be installed in 2010 with the help of the MEXT (Ministry of Education, Culture, Sports, Science and Technology) quantum beam technology program. Special care has been taken in the design of the beamline optics to allow for lock-in amplification measurements.

A new structural biology beamline is under construction at BL-1A, which is based on the “Target Protein” national project. Structure analyses of small crystals less than 10 µm in size are planned by utilizing the anomalous signal from sulfur. A short-gap undulator has been constructed, and special care is taken in the design of the beamline in order to fully utilize softer X-rays. The construction of the beamline started in March 2009 and will be completed by September 2009. After commissioning the beamline and the end station, BL-1A will be opened for users. However, there were three branch beamlines using dipole radiation at BL-1. Among these, the photoelectron spectroscopy activity can be moved to BL-28 and 13 which are undulator based beamlines. Thus BL-1C was decommissioned in March 2008. A branch beamline for condensed matter physics by using X-ray diffraction, BL-1B, was moved to BL-8B until September 2008. The commissioning of new BL-8B progressed well and it has been opened for public users since November 2008. The other X-ray diffraction branch beamline BL-1A was moved to BL-8A. The commissioning is to start from April 2009 and the new BL-8A will be opened for public users in near future.

BL-13 was a typical hybrid beamline, having two branches for hard X-ray and one branch for soft X-ray experiments. Following the strategy described above, it was decided to reconstruct BL-13A as a dedicated beamline for the study of functional organic materials using the existing undulator. The new BL-13A is to be constructed by September 2009, and will be opened for public use in 2010 following beamline and end-station commissioning. The reconstruction made it necessary to move the activities using hard X-rays to other sites. The laser-heating high-pressure and high-temperature X-ray diffraction activities that were carried out at BL-13A will move to new AR-NE1A. XAFS activities at BL-13B will be moved to the other existing XAFS stations. Decommissioning of the old BL-13 started in March 2009.

There is a super-conducting wiggler that emits vertically-polarized hard X-rays at BL-14. Because of this polarization property, BL-14 is suited for phase-contrast imaging experiments, and such experiments using an X-ray interferometer are currently carried out at BL-14C. However, the interferometer must be installed and removed every beam time, since there are two end stations, BL-14C1 and 14C2. Imaging experiments are carried out at BL-14C1, and high pressure experiments using the large “MAX-III” press are carried out at BL-14C2. These two stations share the beam time. In order to permanently mount the sensitive interferometer and dedicate BL-14C to phase-contrast imaging experiments, the reconstruction of BL-14C is planned. High-pressure activities will be moved to AR-NE7.

The beamlines in the NE experimental hall of the 6.5-GeV PF-AR were constructed in 1989 and 1990. The

PF-AR beamlines were refurbished between March and September, 2008. Old NE1A1, NE1A2, NE1B, NE3A, NE5A and NE5B were decommissioned, and new beamlines were constructed at the NE1 and NE3 sites.

A new structural biology beamline funded by Astellas Pharma Inc., was constructed at NE3A. This beamline aims at high-throughput protein crystallography experiments using a robotics system, not only for the company but also for public use. The commissioning of the beamline and the end station progressed well, and the new NE3A is to be opened for public users from April, 2009.

A station for X-ray diffraction under high-pressure and high-temperature environments, combined with nuclear resonance capabilities, has been constructed at NE1A, to where the activities formerly carried out at BL-13A have been moved. Higher flux, more work space, and longer beam times are expected for the highly crowded high-pressure activities by this movement. After commissioning of the beamline and the end station, the new NE1A will be opened for public use in early FY2009.

A new beamline NE7 will also be constructed, to which the activities currently carried out at NE5A and BL-14C2 will be moved. Higher flux and more work space is expected to be realised by this movement. Also, the movement of the MAX-III press from BL-14C2 to NE7 will make it possible to permanently mount the sensitive interferometer at BL-14C, effectively providing more beam time. NE7 was initially named IT4, and was used for the calibration of detectors for high-energy experiments. The Institute of Particle and Nuclear Studies kindly permitted us to use the beamline due to the users' high demand.

More detailed information on the newly-constructed beamlines can be found in the following pages.

1-2 BL-16A: A Variable Polarization Beamline for Soft X-Ray Spectroscopy

BL-16A is a variable-polarization soft X-ray (200-1500 eV) undulator beamline, which provides right/left handed circular and horizontal/vertical linear polarizations [1]. Although fast polarization switching using two APPLE-II undulators in a tandem configuration [2] is planned, only the upstream undulator was installed first, in March 2008. User experiments using the circular polarization mode began in October 2008, and the horizontal and vertical linear polarization modes have been available from December 2008. The elliptical polarization mode, which provides 80-90% circular polarization up to ~1500 eV, will be available from April 2009.

The beamline has four focal points, F1 to F3 and Fm, in order to realize various experimental techniques, as illustrated in Fig. 1. For example, an XMCD apparatus equipped with a superconducting electromagnet has been fixed at F2 and opened for all users. A depth-resolved XMCD apparatus is also available, which can be easily connected to any experimental port (usually F3). Resonant X-ray scattering and wavelength-dispersive surface X-ray absorption fine structure techniques are also under commissioning. A typical X-ray magnetic circular dichrosim (XMCD) spectrum is shown in Fig. 2, which demonstrates that even a sub-monolayer specimen can be investigated in a practical data acquisition time.

The second undulator for fast polarization switching will be installed in 2010, with financial support from the quantum beam technology program of MEXT. The different polarizations from the two undulators are alternately led to the beamline optics by modulating the electron orbit through the undulators [2]. The feasibility

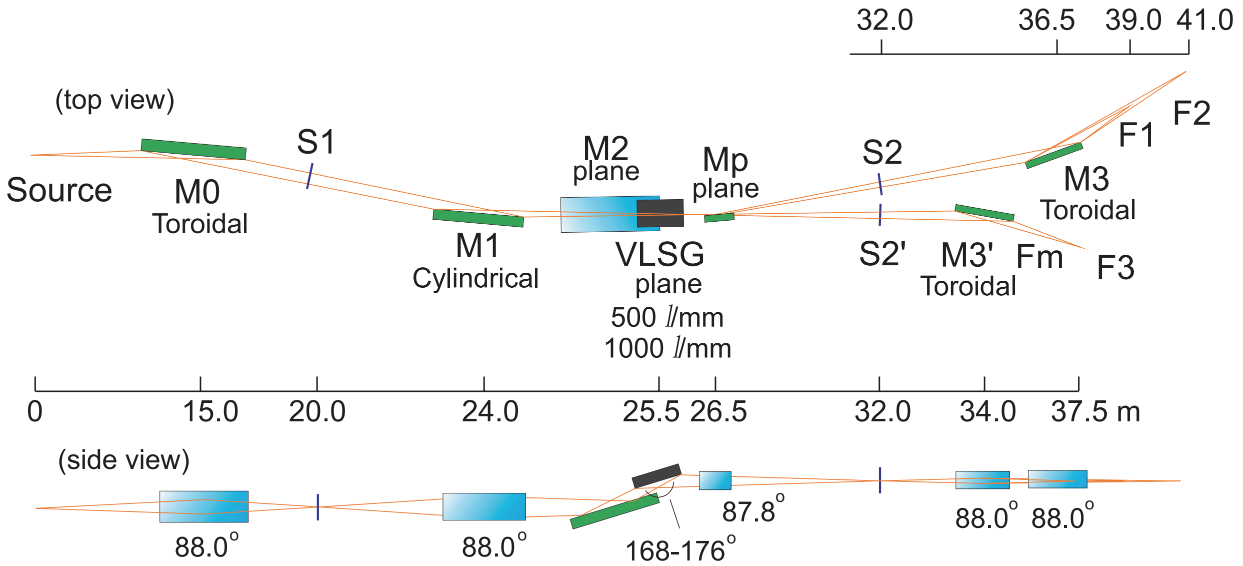

Figure 1
Schematic layout of BL-16A.

of this technique has been checked prior to the installation of the second undulator, by modulating the electron orbit at 10 Hz and monitoring the beam intensity. Figure 3 shows the modulation in the X-ray intensity, which agrees well with the expected modulation estimated from an X-ray tracing simulation.

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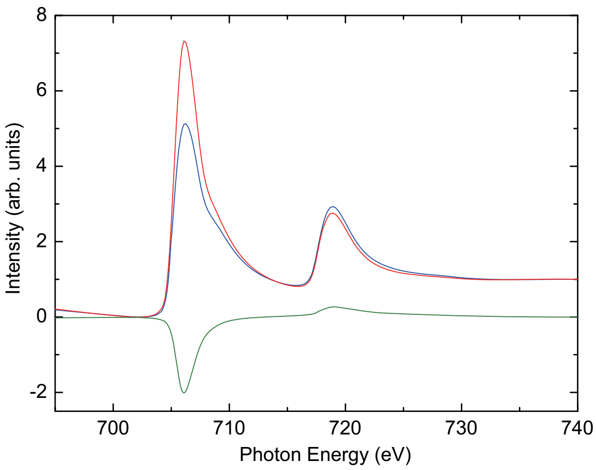

Figure 2
Fe L-edge XMCD spectrum recorded in the total electron yield mode with a data acquisition time of 3 s/point for an Fe(0.9 ML)/Ni(6 ML)/Cu(100) film.

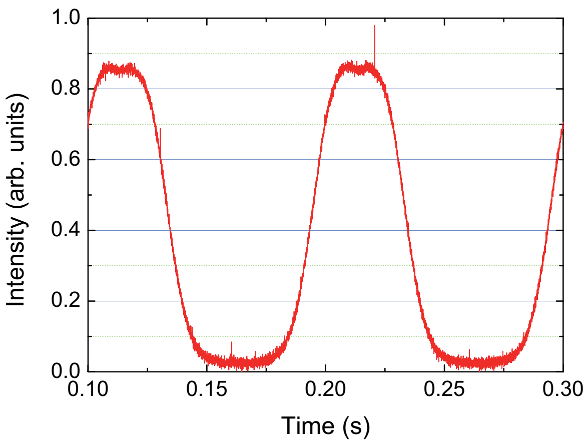

Figure 3
X-ray intensity monitored by the photocurrent from M3 during the electron orbit modulation by ~0.28 mrad at 10 Hz.

1-3 AR-NE1A: A New Beamline for High-Pressure Experiments Combining X-Ray Diffraction and Mössbauer Spectroscopy

At the end of March 2009, activities at BL-13A[1], dedicated to high pressure X-ray diffraction experiments using a laser heated DAC, were moved to the reconstructed AR-NE1A for further developments in the earth sciences, combined with nuclear resonance capabilities. The old AR-NE1A[2], where magnetic Compton scattering experiment were carried out for more than 18 years, was terminated in March 2008. During the spring and summer cycles the old beamline was removed, and in the autumn most of the new beamline components were installed. Commissioning of the new beamline optics began after safety inspections in the beginning of January 2009, and the new beamline will be opened for user experiments from the end of May.

The merits of moving activities from BL-13A to AR-NE1A are listed below.

(1) Increased photon flux in the high energy region (× 4@30 keV, × 20@50 keV)

(2) Increased beamtime due to the dedicated station (BL-13A was a tandem station)

(3) Variability of energy (fixed energy at BL-13A)

(4) Introduction of a new experimental method (Mössbauer spectroscopy) to high-pressure earth sciences, due to the world's only single-bunch dedicated ring, the PF-AR.

The beamline design was optimized considering these advantages.

The structure of the new AR-NE1A is shown schematically in Fig. 4. Each optical component is explained in the figure, and the main features are listed below.

(a) Double-crystal monochromator. Due to the high heat-load from the high-power MPW, a micro-channel water-cooled crystal was adopted. Output energy ranges from 7 keV to 50 keV, corresponding to the absorption edge of Fe and high-Q for structure refinement, respectively.

(b) High-resolution monochromator and graphite refraction lens. In order to achieve super monochromatized X-rays, with $\Delta E/E \sim 5\times10^{-7}$, two channel-cut crystals, Si(4,2,2) and (12,2,2) are arranged for Mössbauer spectoroscopy. To improve the throughput at the high-resolution monochromator, a parallelism conversion refractive lens made of graphite will be installed in FY2009.

(c) Trichromatic focusing mirror. The multi-layered W/C mirror has been newly developed for three energies of 14.4 keV, 30 keV and 50 keV. The former is for Fe nuclear resonance experiments, and the latter two are for x-ray diffraction experiments.

(d) The sample in the DAC can be studied using both X-ray diffraction and Mössbauer spectroscopy under the same pressure and temperature condition, with

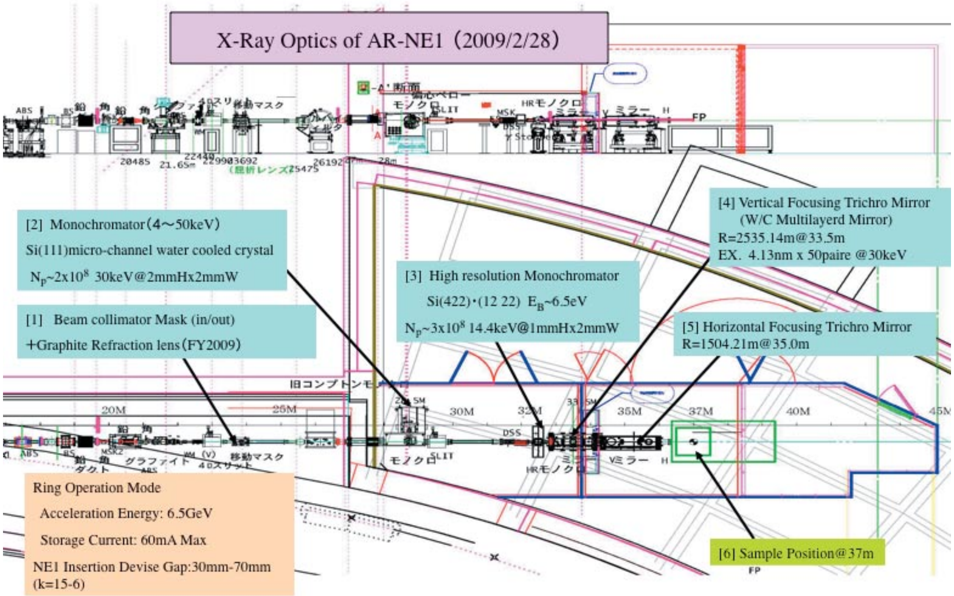

Figure 4
Plan view and side view of AR-NE1A.

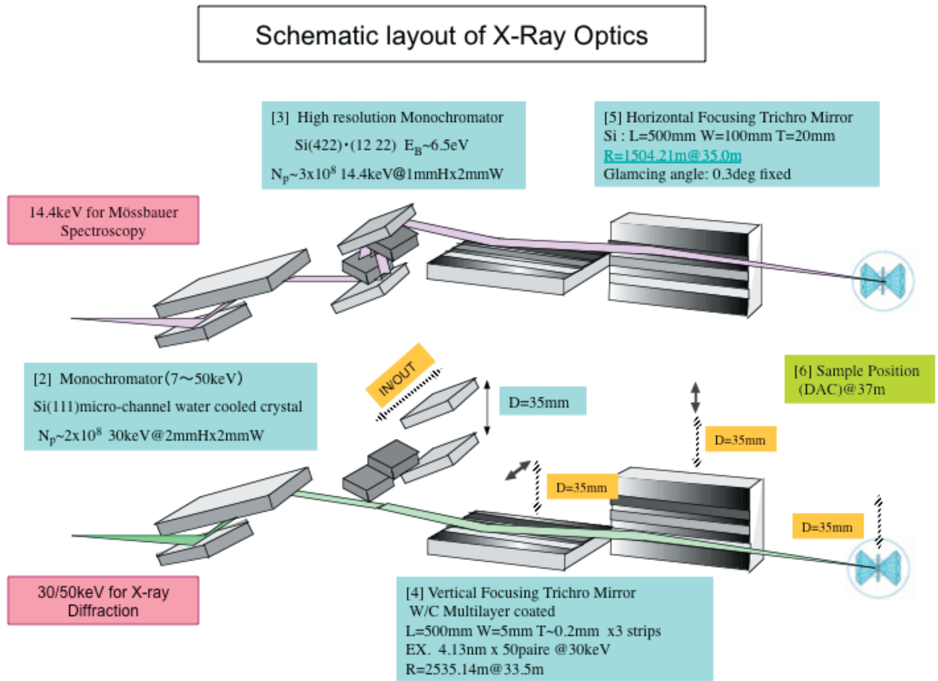

Figure 5
Configuration of the newly developed optical systems.

instantaneous changes between the two configurations possible. In Fig. 5 we illustrate the configuration of the newly developed optical systems which enable us to extend the high-pressure science field.

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1-4 AR-NE3A: A Protein Crystallography Beamline for Pharmaceutical Applications

AR-NE3A is a new protein crystallography beamline dedicated to pharmaceutical research where a large number of protein/ligand complex crystals must be examined. The beamline is designed to deliver the most intense X-ray beam at the sample position among the protein crystallography beamlines at the PF, and also to allow the performing of high-throughput diffraction experiments. The light source is an in-vacuum undulator located in the NE3 section of the PF-AR 6.5 GeV ring. There are three main optic components, a water-cooled collimating mirror, a double-crystal monochromator with a liquid nitrogen cooling system, and a toroidal focusing mirror. For the high-throughput diffraction experiments, the beamline is equipped with a diffractometer with a high-precision spindle axis and shutter, an X-ray detector featuring a large active area, fast readout and high X-ray sensitivity, and a sample exchange system, PAM, which can store 288 samples in a Dewar placed in the end-station.

AR-NE3A was used as a beamline for nuclear resonant scattering using quantum beats until March 2008, and construction work for the new beamline began in March 2008 with the decommissioning of the previous beamline components and hutches. After this decommissioning, the hutches for the new beamline were constructed. The components of the front-end and the beamline were installed during the summer shutdown of PF-AR, and the first light was introduced in October 2008. After commissioning the beamline optics in the autumn, the experimental apparatus at the end-station was installed in January 2009. Following this, commissioning for protein crystallography experiments was successfully completed by March 2009. Table 1 shows a comparison of the beam intensities at the sample position measured at AR-NE3A and AR-NW12A, another high-throughput protein crystallography beamline. The results show that the beam intensity at AR-NE3A is about three times higher than that at AR-NW12A. During the commissioning, actual diffraction experiments were carried out by users from outside KEK (see table 2). Consistent with the increase of beam intensity, a data set of almost the same quality was able to be collected at AR-NE3A with a shorter exposure time. This suggests that more rapid data collection is possible at AR-NE3A than at AR-NW12A.

During the commissioning, a fully automated data collection and processing system we had been developing was also extensively tested. This system will be utilized in the pharmaceutical research carried out at this beamline from April 2009. Details of the system are described in the “Structural Biology Research Center” section of this volume.

User operations of AR-NE3A will begin in April 2009. Because the construction of the beamline is financially supported by Astellas Pharma Inc., they will have priority access to the beamline for their research. The remaining beam time will be assigned to general academic users and other industrial users.

Table 1 Comparison of beam intensity and beam size between AR-NE3A and AR-NW12A.

	Flux*(ph/s) at 1.0 Å	Beam size (mm)	
		Horizontal	Vertical
AR-NE3A	8.0×10^{11}	0.70	0.17
AR-NW12A	2.9×10^{11}	1.50	0.22

* The flux was estimated by measuring the intensities of the X-ray beam through ϕ 0.2 mm - pinholes at the sample positions

Table 2 Data collection conditions and statistics for a human metabolic enzyme. (With courtesy of Drs. N. Tanaka and Y. Kusakabe of Showa University)

	NE3A	NW12A
Crystal size (mm)	0.5 × 0.1 × 0.02 (thin plate)	0.5 × 0.1 × 0.02 (thin plate)
Space group	*P*1	*P*1
Cell dimensions		
a (Å)	86.137	86.152
b (Å)	85.152	85.326
c (Å)	86.147	86.117
α (deg.)	94.362	94.337
β (deg.)	93.885	93.887
γ (deg.)	119.113	119.11
No. of subunits / ASU	8 (2 tetramers)	8 (2 tetramers)
	2,080 a.a. (2 × 4 × 260 a.a.)	2,080 a.a. (2 × 4 × 260 a.a.)
Detector	ADSC Q4R	ADSC Q210r
Wavelength (Å)	1.000	1.000
Exposure time / image (sec.)	3	10
Total rotation range (deg.)	180 (0.5 x 360 images)	180 (0.5 x 360 images)
Resolution (outer shell) (Å)	1.6 (1.63-1.60)	1.65 (1.68-1.65)
No. of observed reflections	510,629	480,415
No. of unique reflections	267,586	243,952
Multiplicity	1.9 (1.9)	2.0 (1.9)
Mean $I / \sigma_{(I)}$	24.4 (2.6)	21.8 (2.1)
R_{sym} (%)	7.3 (40.5)	6.9 (45.5)
Completeness (%)	95.7 (94.4)	95.9 (94.7)