1 Newly Developed Experimental Facilities

1-1 A New Multi-Purpose Imaging-Plate Diffractometer at BL-1B

Outline

BL-1B is used for diffraction experiments using an imaging-plate (IP), Weissenberg-camera-type diffractometer (Mac Science Co.). Because the diffractometer had become out-of-date, it was upgraded in FY2005 (Fig. 1). The new diffractometer has efficiency similar to that of the old one, but has the advantages of a shorter elapsed time for IP reading/erasing (ca. 3 min./ frame) and a higher angular resolution.

It can be used in conjunction with a refrigerator, pressure cells, or a furnace. It is possible to carry out experiments (1) at temperatures in the region of 10 K - 1000 K, and (2) under hydrostatic pressure up to 50 GPa over a temperature range of 10 K - 300 K. The camera was designed to be used for single-crystal structure analyses as well as for powder-diffraction measurements.

The main important investigations made at this station include (1) pressure and/or temperature dependence of the structure of strongly correlated materials, (2) accurate charge-density studies of endohedral fullerenes, and (3) structural studies of nano-materials (i.e., nano-tubes, nano-particles).

Beamline

This station extracts synchrotron radiation emitted from a bending-magnet section B1. The incident X-rays are monochromatized by a Si(111) double-flat monochromator and focused at the sample position by a Rh-coated bent Si mirror.

Equipment

An IP diffractometer (Rigaku Co.), equipped with a camera with a radius of curvature of 191.3 mm, is permanently installed. X-ray scattering over a 2θ angular range of –60 to 145 degrees is available. The equipments listed below are also available at the station:

- N_2 gas-flow-type temperature controller (100 K-373 K)
- custom-made refrigerator with a Be window producing no back-ground scattering (10 K - 300 K)
- diamond anvil cells connected to a refrigerator (10 K-300 K, up to 50 GPa)
- furnace (300 K-1000 K)

All instruments are controlled by a standard RIGAKU program running on a Windows XP machine. The file size for a single IP photograph is about 17 MB. We recommend users to bring a hard disk drive with USB connection for data transfer, although CD-R and DVD-R drives are available at the station.

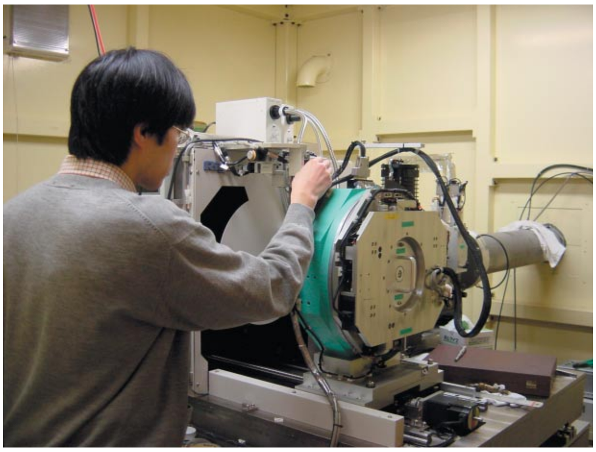


Figure 1
New imaging-plate Weissenberg-camera-type diffractometer (Mac Science Co.).

1-2 A New Short-Gap Undulator Beamline BL-17A for Structural Biology

Introduction

Four new short-straight sections were produced as the result of the "Straight-Section Upgrade Project" of the Photon Factory during a six-month shutdown in the first half of FY2005 [1].

A new short-gap undulator, SGU#17, was designed and constructed for one of the short straight sections [2]. With the expected high-brilliance beam emitted from SGU#17, we have proposed two frontier research areas in structural biology — micro-crystal structure analysis and structure determination using soft X-rays. The construction of BL-17A commenced just after the end of PF-ring operation in FY2004 (February 28, 2005) and completed in September, 2005. The first beam was successfully delivered to the experimental station on October 7, 2005, and test experiments immediately followed the commissioning of the beamline components and the data-acquisition system.

Beamline optics

Figure 2 shows a current view of BL-17A. The location and the specifications of the main optical components are summarized in Table 1. A detailed description was given in the preceding volume of the Activity Report [3].

Performance

Preliminary characteristics of the beam at the sample position are summarized in Table 2. The energy resolution of the monochromatized beam was estimated based on the FWHM of a rocking-curve measurement with Si(111) crystals. The beam size was measured by scanning a silt with 20 µm and 10 µm windows in the horizontal and vertical directions. The photon-beam intensity at the sample position was determined by using an ionization chamber through which nitrogen gas was flowed. The values expected from a simulation using ray-tracing software [4] are also shown in the table. The beam size is 40% larger than predicted, and the photon flux 3 times smaller. It is anticipated that the performance can be improved by optimizing the undulator and beamline optics.

Figure 2
Current view of BL-17A.

First experimental result

The first test X-ray crystallography experiment was carried out at the end of beamline operation in March 2006. We show here the experimental results reported by Drs. N. Tanaka and M. Tsunoda of Showa University, who carried out a small-crystal experiment using a crystal of a methyltransferase. The space group and the cell dimension of the crystal are P212121 and a = 48.32 Å, b = 51.62 Å, c = 62.66 Å, respectively. The crystal was 20 µm cubic in size (Fig. 3). There were two small crystals in the loop, but it is possible to selectively expose only one crystal to X-rays. A high-quality, single-crystalline diffraction image was successfully obtained (Fig. 3).

Although BL-17A is still under improvement, very promising results have already been obtained. Optimization of the optics, stabilization of the beam position with a feedback system, and tuning the diffractometer will provide users with highly brilliant, highly stable, highly energy-resolved, and sufficiently collimated x-ray beams. Micron-size structure analysis and structure determination using soft X-rays will be available.

Table 1 Locations and specifications of the optical components

Insertion device	Type: short-gap undulator Period length : 16 mm Number of periods : 29 Magnetic field: max 0.7 Tesla Energy range: 6-9 keV (3^{rd} harmonic) and 11-13 keV (5th harmonic)
Double crystal monochromator (17.5 m from the source)	Crystals: Si(111) Fixed exit: numerical link Cooling system: liquid nitrogen circulation Energy range: 4 – 20 keV
Vertical focusing mirror (24.0 m)	Type: flat-bent Material: Rh-coated Si single crystal Size (mm): 1000(L) × 100(W) × 50(T) Glancing angle: 4.0 mrad Radius of curvature: 4000.0 m Slope error: max. 0.75 µrad Roughness: max. 1.7 Å
Horizontal focusing mirror (30.8 m)	Type: flat-asymmetric-bent Material: Rh-coated Si single crystal Size (mm): 1000(L) × 100(W) × 50(T) Glancing angle: 4.0 mrad Radius of curvature: 2224.5 m Slope error: max. 0.95 µrad Roughness: max. 2.0 Å
Focal point (36.0 m)	Sample position

Table 2 Preliminary characteristics of the beam.

		Measured	Ray-tracing result
ΔE/E at 12.4 keV		2.5×10^{-4}	2.2×10^{-4}
Beam size at sample position (µm)		234 (H)	180 (H)
		32.9 (V)	22.0 (V)
Flux at the sample position (phs/sec/400mA)			
	100 µm × 100 µm	7.7×10^{10}	3.6×10^{11}
12.4 keV	40 µm × 40 µm	2.2×10^{10}	8.8×10^{10}
	20 µm × 20 µm	6.6×10^{9}	2.3×10^{10}
6.5 keV	20 µm × 20 µm	1.3×10^{10}	6.0×10^{10}

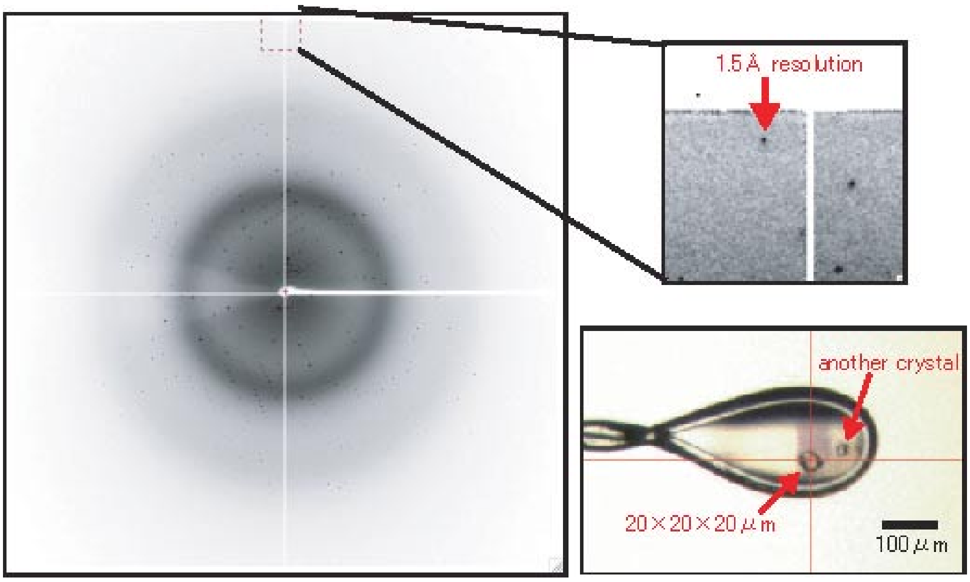


Figure 3
Test X-ray diffraction experiment of small crystals. The photograph of the crystals (lower-right panel) shows two small crystals and a trace of the X-ray beam in the cryo-loop.

References

[1] S. Asaoka, K. Haga, K. Harada, T. Honda, Y. Hori, M. Izawa, T. Kasuga, M. Kobayashi, Y. Kobayashi, H. Maezawa, Y. Minagawa, A. Mishina, T. Mitsuhashi, T. Miyajima, H. Miyauchi, S. Nagahashi, T. Nogami, T. Obina, C. O. Pak, S. Sakanaka, Y. Sato, T. Shioya, M. Tadano, T. Takahashi, Y. Tanimoto, K. Tsuchiya, T. Uchiyama, A. Ueda, K. Umemori, and S. Yamamoto, *AIP Conf. Proc.*, **705** (2004) 161.

[2] S. Yamamoto, K. Tsuchiya and T. Shioya, *AIP Conf. Proc.*, to be published.

[3] Photon Factory Activity Report, **22A** (2005) 65.

[4] K. Takeshita, *Rev. Sci. Instrum.*, **66** (1995) 2238.

1-3 Present Status of the ARPES Undulator Beamline BL-28A

The undulator beamline BL-28A (Fig. 4), dedicated to high-resolution angle-resolved photoemission (ARPES) experiments, was successfully commissioned in FY2005. The light source of the beamline is a helical undulator with a period length of 16 cm and a pole number of 23 + 2 (half poles), covering a photon energy from 30 to 300 eV. This energy range is suitable for detailed studies of the electronic structure of condensed matter. A high-resolution ARPES beamline at the Photon Factory had been strongly demanded by many users for a long time. The main purpose of the new beamline is ARPES studies of highly correlated electron systems, such as high transition-temperature (high-T_c) superconductors and colossal magnetoresistive oxides, and spintronic nanomaterials. Both high energy resolution and a high photon flux were required to enable high-resolution ARPES experiments for high-T_c superconductors and nanomaterials. An entrance-slit-less, varied-including-angle, varied-line-spacing, plane-grating monochromator (VLS-PGM) was designed for BL-28A to provide 10^{12} photons/sec at E/ΔE>10,000. The energy resolution of the monochromator was evaluated using rare gas photoionization. Figure 5 shows the $3s \rightarrow np$ photoionization spectrum of Ar recorded using the new monochromator. The energy width of the $3s \rightarrow 26p$ photoionization peak at 29.2 eV is less than 1 meV, corresponding to an energy resolving power (E/ΔE) of greater than 30,000. The photon flux has been measured using a calibrated photodiode, and the beam spot size has been estimated to be 150 µm(H) × 50 µm(V)

Figure 4
Photograph of the newly-built beamline BL-28A.

at the sample position using a photoelectron-emission microscope (PEEM). These results show BL-28A to exhibit high energy resolution, high photon flux, and a considerably small spot size.

The high-resolution ARPES end station at BL-28A consists of a VG Scienta SES-2002 analyzer and a multi-axis low-temperature sample manipulator (iGonio-LT). Commissioning of the ARPES end station was completed in FY2005, and is now open for users. Some recent ARPES results obtained at the new station are presented in the Electronic Structure section of this Activity Report.

In FY2006, a branch beamline BL-28B is planned to be commissioned. The new branch line will use the same monochromator as BL-28A, with the beam being horizontally deflected using a mirror placed just downstream of the monochromator. The two branch lines will operate in a time-sharing mode, with BL-28B dedicated to non-ARPES techniques such as PEEM, XMCD, and gas-phase experiments.

1-4 AR-NW10A: A Beamline for Hard X-Ray XAFS/AXS

Beamline NW10A, dedicated to hard X-ray XAFS/AXS experiments, was constructed in FY2005 and the first beam was delivered to the experimental station in January 2006. NW10A was designed to replace BL-10B, closed in December 2005. Because BL-10B had no focusing optics and used synchrotron radiation emitted from the 2.5-GeV storage ring whose critical energy is 4 keV, the photon flux above 20 keV was severely limited, restricting the quality of spectra recorded in this energy region. Nevertheless, more than 1030 papers resulting from the use of BL-10B have been published as of July 2006.

The main optical components of the new beamline are a Si(311) double-crystal monochromator and a Pt-coated bent cylindrical mirror. Both of the crystals and a copper plate are indirectly water-cooled to avoid thermal expansion of the crystals and mechanical components due to irradiation with the direct beam and Compton scattering from the first crystal. A focusing mirror is placed at 20.5 m from the source, focusing the beam at a position 32.8 m away from the source. Platinum was chosen as the coating material to realize a higher irradiation angle and higher reflectivity at 40 keV. The irradiation angle was chosen to be 1.9 mrad, since the critical energy at this angle was calculated to be 44 keV. The requirements to realize focusing conditions have led to a sagittal radius of curvature of 29.2 mm and a tangential radius of curvature of 8092 m.

The focal size has been determined to be 2.2 mm × 0.5 mm in FWHM, in agreement with ray-tracing results. This rather large focal size is due to the large electron beam size at the PF-AR, which has an emittance of 285 nmrad.

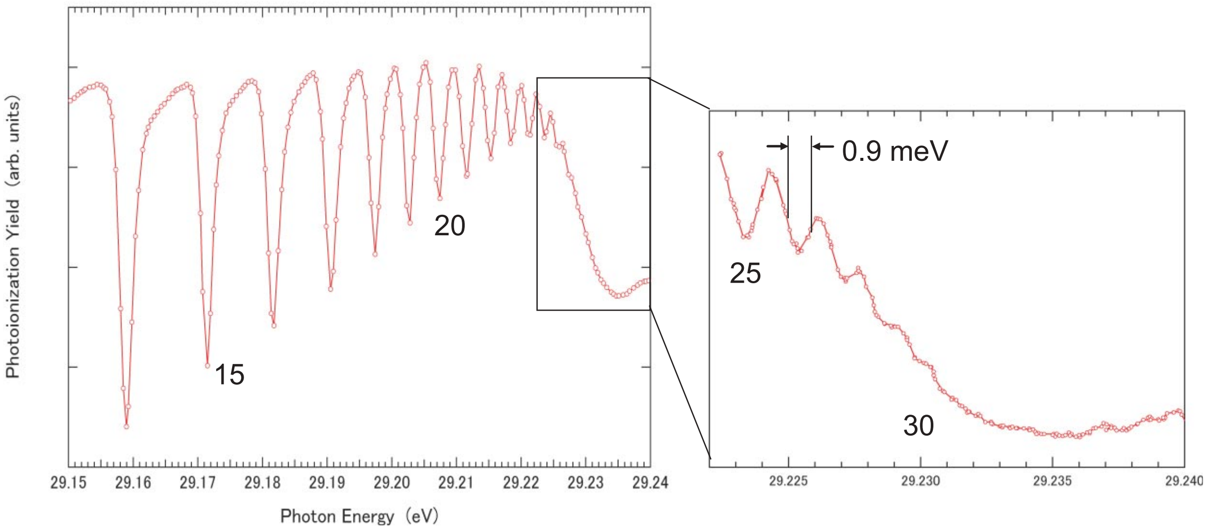


Figure 5
Photoionization yeild of Ar 3*s* → *np* around 30 eV.

The photon flux has been evaluated using an ionization chamber and compared with the flux recorded at BL-10B (Fig. 6). The entrance-slit size was chosen to be 1 mm^2 for the focused beam and 5 mm × 1 mm for the unfocused beam. The flux gain is most prominent above 20 keV, with the flux at the new beamline being 20 times greater than at BL-10B at 20 keV, 70 times greater at 25.5 keV and 200 times greater at 30 keV. When the mirror was aligned for a 1.9 mrad irradiation angle, the photon flux decreased with photon energy above 40 keV, indicating the critical energy for this mirror setting.

To increase the photon flux above 40 keV the irradiation angle was changed to 1.85 mrad. Figure 6 shows the photon flux recorded under this condition. The reason why the critical energy is rather lower than that expected is not clear at present, but a lower density of the coated platinum layer is suspected.

The ratio of the third to the first order is sufficiently low above 15 keV when the double crystals are properly detuned. However, it is higher than 10^{-4} below 12 keV. Thus it will be necessary to take special care when using this energy region, for example, selecting lighter gases for the ionization chambers or installing a mirror for higher-order light rejection.

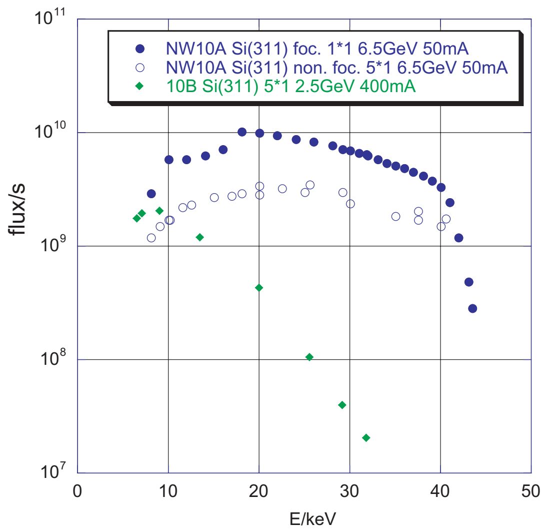


Figure 6
Photon flux obtained at NW10A. The closed and open circles denote the photon flux with and without focusing. The flux at BL-10B is shown as diamonds.

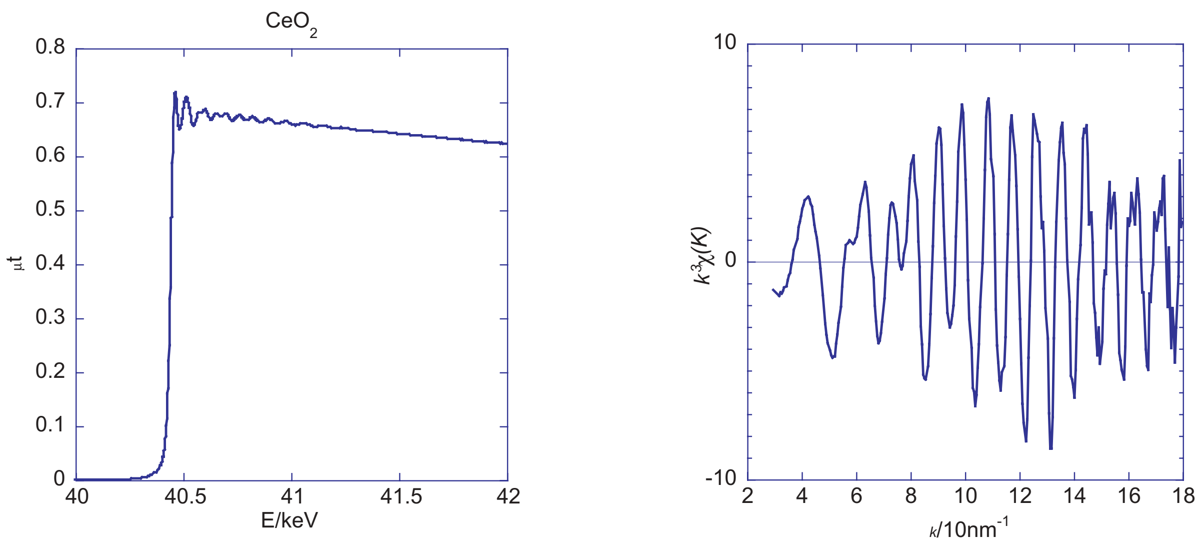
Figure 7 shows a raw XAFS spectrum and extracted EXAFS wiggles recorded for CeO_2. The EXAFS wiggles can be clearly observed up to k=180 nm^{-1}, indicating the high-energy capabilities of NW10A. Although it is not easy to record rhodium XAFS spectra at most beamlines due to the use of Rh-coated mirrors, Rh XAFS spectra can easily be recorded at NW10A. The Pt L edges can also easily be cancelled if the higher orders are properly suppressed.

1-5 Beamline AR-NW14A for Time-Resolved X-Ray Studies

Beamline AR-NW14A is a new insertion-device beamline at the PF-AR dedicated to time-resolved X-ray diffraction, scattering and absorption experiments. The primary scientific goal of the beamline is to observe ultrafast dynamics triggered by optical pulses in condensed matter such as organic and inorganic crystals, biological systems and liquids. Use of the high photon flux emitted from two undulators should make it possible to take snapshots of atoms and/or molecules in non-equilibrium states at 100-ps resolution.

The beamline is financially supported by the Non-equilibrium dynamics project under the ERATO program of Japan Science and Technology Agency (JST), and Professor Koshihara at the Department of Chemistry and Materials Science of Tokyo Institute of Technology, is the project director.

The beamline has two undulators with period lengths of 36 mm (U36) and 20 mm (U20). The U36 covers a photon energy range of 5-25 keV using the



(a) (b)

Figure 7
Raw XAFS spectrum (a) and extracted EXAFS wiggles (b) of CeO_2.

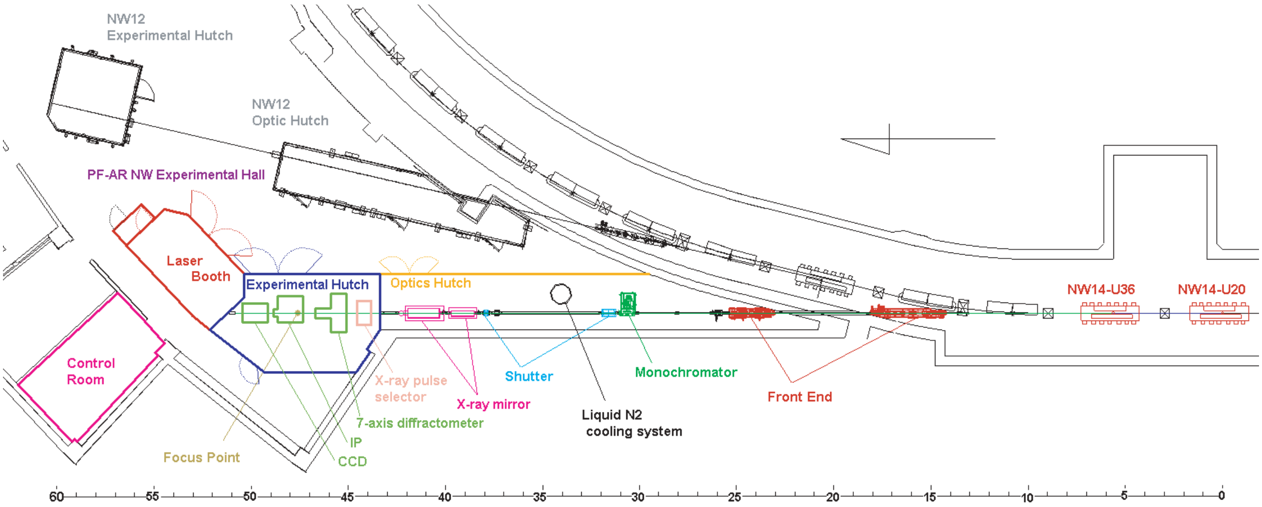


Figure 8
A plan view of the beamline AR-NW14A.

1st, 3rd, and 5th harmonics, providing a tunable and intense monochromatic X-ray source. The typical photon flux of the monochromatic beam is estimated to be ~10^{12} photons/s with an energy bandwidth of $\Delta E/E$ ~10^{-4}. The 1st harmonic energy of the U20 is 13-20 keV, with an energy bandwidth of $\Delta E/E$ ~10^{-1}. The U20 thus provides a 'narrow-bandwidth white beam' or 'wide-bandwidth monochromatic beam' with a photon flux of ~10^{15} photons/s. The X-ray pulses are delivered at a frequency of 794 kHz with a pulse duration of ~100 ps (FWHM).

A plan view of the beamline and beamline components in the optics hutch is given in Fig. 8. The front-end consists of a fixed mask, a beam-position monitor, an absorber, a beam shutter, one or more graphite heat absorbers, XY-slits for white X-rays and Be windows. The main optical components are a double-crystal monochromator and an X-ray mirror system, which are located 30.5 m and 39-42 m downstream of the center of the U20 insertion device. The double-crystal monochromator consists of flat Si(111) crystals which are cooled with liquid nitrogen to reduce deformation caused by heat load. The cooling system can handle a heat load of up to 450 W. The X-ray mirror system consists of three Rh-coated mirror assemblies: a bent cylindrical mirror for focusing X-rays, and a double-mirror system (cut-off mirrors) to reduce contamination from the higher harmonics.

The construction of beamline NW14A was completed in the summer of 2005. The U36 device was installed in the summer of 2005, and the U20 will be installed during summer 2006. NW14A was commissioned during October-December 2005, and user runs successfully began in January 2006.

Figure 9 shows the photon-flux spectra of the U36 undulator measured in the experimental hutch with an undulator gap of between 10 - 20 mm (K=1.1-2.7). X-rays in the low-energy region are absorbed by the Be windows and graphite filters placed in the front end, and the photon flux in the high-energy range is reduced by the energy cut-off of the Rh-coated mirrors. The Si(111)

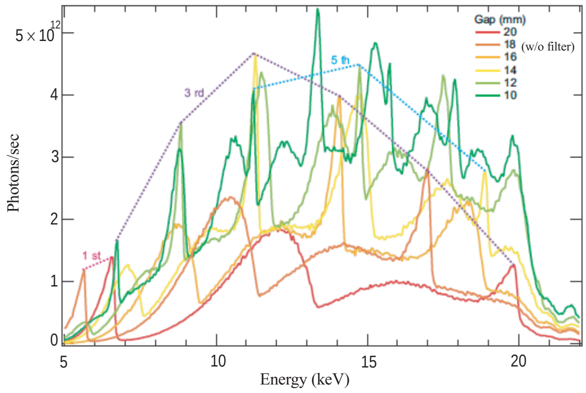


Figure 9
The photon flux spectra of U36.

double-crystal monochromator was used for this measurement with a source divergence of 0.32 mrad (H) × 0.05 mrad (V), and the energy bandwidth was $\Delta E/E$ ~10^{-4}. The focused beam size was 0.44 mm (H) × 0.24 mm (V) at the sample position with a focusing ratio of 4.53 : 1.

The experimental equipment installed at NW14A is shown in Fig. 10. Three different diffractometers (Huber 4-axis, RIGAKU Imaging Plate and marccd) were installed for various time-resolved diffraction and scattering experiments. A fs laser system (Spectra Physics Tsunami + Evolution + SpitFire) and a ns laser system (Continuum PowerLite8000) were installed in the laser booth. To isolate X-ray pulses from the storage ring, a mechanical chopper (X-ray pulse selector) was inserted in the forefront of the experimental hutch. The chopper rotates at a frequency of 1 kHz with a timing jitter of less than 2 ns, and isolates 1-kHz X-ray pulse trains from the 794-kHz X-ray pulse trains emitted from the storage ring.

A typical timing scheme for a pump-probe experiment at NW14A is shown in Fig. 11. The PF-AR uses 508.58 MHz RF signal for its RF cavities. The RF signal is delivered to the timing-control modules at NW14A, and processed for further synchronization. The signal is divided for laser and X-ray timing following RF amplification. For X-rays, both the RF signal (508 MHz) and the revolution signal (794 kHz) are used for synchronization of the mechanical chopper. For the lasers, the RF signal is divided by 6 for cavity mode-locking, or by 89600 for regenerative amplification. The laser-X-ray delay can be set with a delay generator (Stanford Instruments DG535) in the ns time domain, or with an RF phase shifter in the sub-ns time domain.

The beamline has been fully operational since January 2006, and various preliminary pump-probe experiments have been carried out. Scientific output from NW14A will be presented in the 2006 Activity Report.

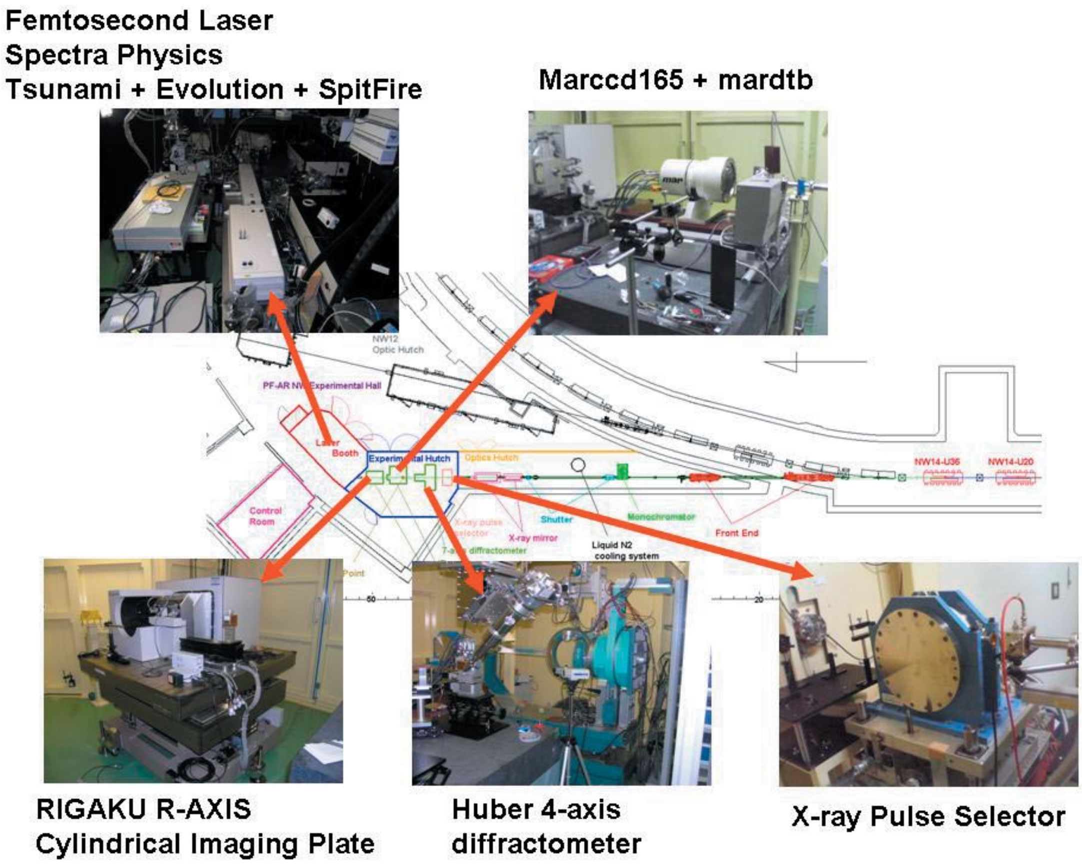


Figure 10
Experimental apparatus and laser booth at NW14A.

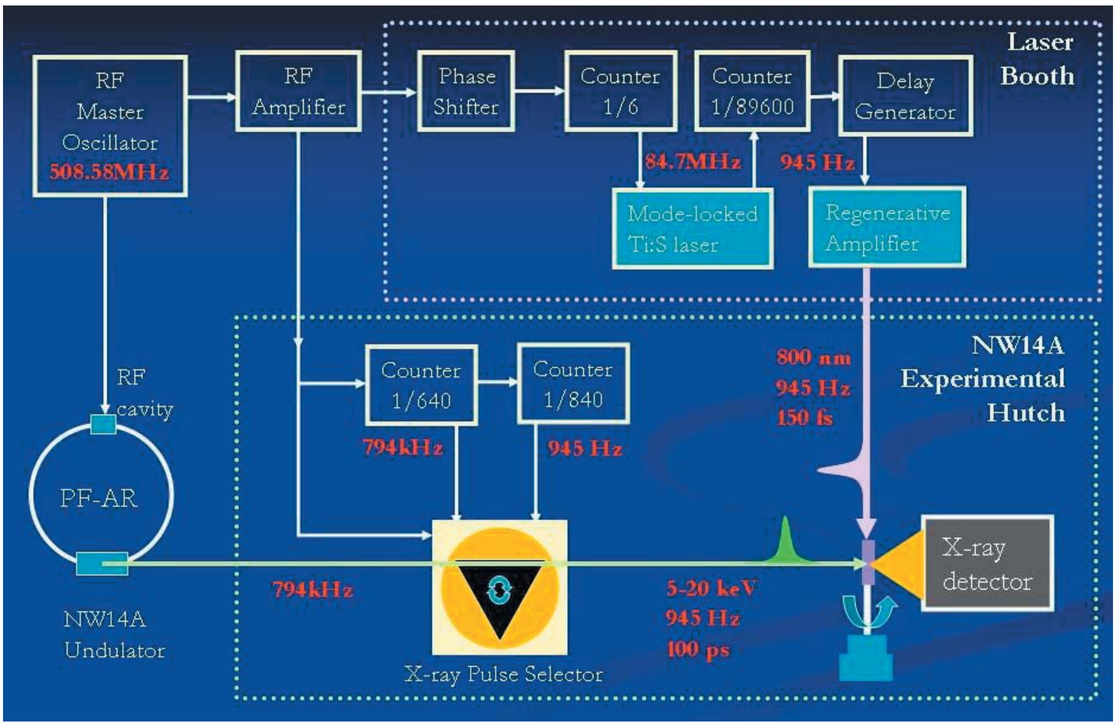


Figure 11
Typical timing scheme of a pump-probe experiment at NW14A.

1-6 Construction of a New Short-Period, Short-Gap In-Vacuum Undulator Beamline BL-3A for Structural Material Science

Outline

The new BL-3A is a short-period, short-gap in-vacuum undulator (SGU) beamline optimized for resonant X-ray scattering and polarization-controlled X-ray diffraction studies. Such experimental techniques are becoming increasingly important in applications of structural material science. The high brightness of the X-ray beams from the SGU light source together with advances in X-ray optics will enable experimentalists to perform frontier research at a 2nd-generation synchrotron facility. The SGU has been developed specifically for this purpose, and will be installed in the short straight section of the PF ring. This will be the second SGU installed in the PF following SGU#17 which supplies X-rays for structural biology research. One of the beamlines previously used for structural material science, BL-16A, has been closed, and activities at the old BL-3A will be transferred to a reconstructed BL-6C. The construction of the new BL-3A and the reconstruction of BL-6C are currently in progress. The new BL-3A will deliver its first beam to the experimental station in October 2006.

Insertion device and front end

Four new short-straight sections have been produced as the result of "Straight-Section Upgrade Project" of the PF which has been under way since 2002. A new SGU will be installed at the short (1.4 m) straight section between bending magnets B2 and B3. The central 0.5-m-long part of the straight section will house the undulator magnets which comprise 26 periods with a period length of 18 mm. The magnet gap will be variable from 3.5 mm to 40 mm. The beam acceptance of the beamline is restricted to 1.00 mrad(H) and 0.30 mrad(V) by a water-cooled main mask positioned just after the main beam shutter located upstream of the shield wall. In contrast with the SGU beamline at BL-17, BL-3A coexists with bending-magnet beamlines BL-3B and BL-3C. Since BL-3B is a VUV beam line, there is neither beryllium window nor graphite filter in the front-end section.

Optics

The schematic beamline optics of the new BL-3A is shown in Fig. 12. A vertically-deflecting double-crystal monochromator (DCM) is located 26 m from the light source. BL-3A will use the DCM which was previously used at BL-16A, following a partial remodeling. The monochromator produces a fixed-exit beam over an energy range of 4.0 to 25.0 keV. The crystals for the DCM have a direct water-cooling system to reduce crystal deformation due to high heat load.

Following the DCM, a crystal phase retarder is used for polarization control. By using five type-IIa (001) diamond crystals, horizontally linearly, circularly, and vertically linearly polarized X-rays can be supplied to the experimental station over an energy range of 4.5 to 14.0 keV. A Rh-coated bent cylindrical mirror is located 30 m from the light source. For diffraction studies using a six-axis diffractometer located at 36 m from the light source the cut-off energy is approximately 16 keV with a glancing angle of 4.3 mrad. The cut-off energy is 21 keV for a grazing angle of 3.3 mrad for diffraction studies using a two-axis diffractometer with a super-conducting magnet. The expected beam intensity at the focal position is calculated to be almost 10^{11} photons/sec at 6 keV.

Construction schedule

With the aim of introducing the first beam to the experimental station in October 2006, construction began just after the end of PF-ring operation in FY2005. The necessary work includes the decommissioning of the old BL-3A, 3C, 6B, 6C, and 16A, the construction of the new BL-3A, and the reconstruction of BL-3C and BL-6C. By the end of FY2005, the closure of BL-6B and 6C was complete, and the decommissioning of BL-3A, 3C, and 16A is scheduled for the end of June 2006. The installation of the new optical elements will be completed by the end of August 2006. Commissioning of the new beamline will begin in October 2006, and general user operation will commence in the beginning of 2007.

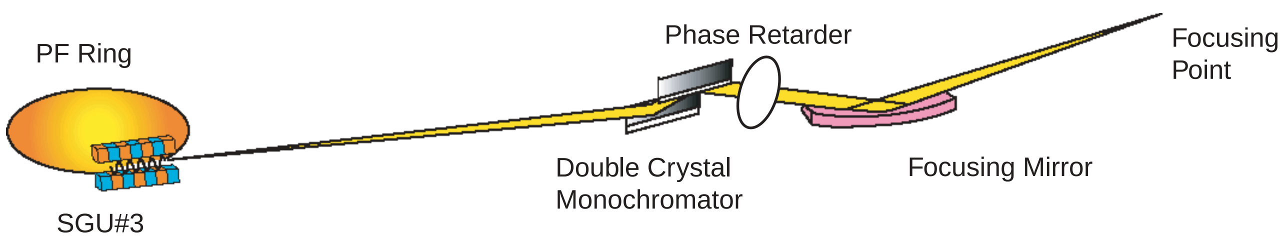


Figure 12
Schematic beamline optics of the new BL-3A.