# 1 Newly Developed Experimental Facilities

## 1-1 Overview

The Straight-Sections upgrade project of the 2.5-GeV PF-ring that aimed at creating more space for insertion devices (ID) was successfully completed in FY2005, as reported in the previous issue. In order to fully utilize the upgrade, some insertion device beamlines have been reconstructed; BL-28A in 2004, BL-17A in 2005 and BL-3A and 28B in 2006. Among these, BL-17A and 3A use in-vacuum short-gap undulators to provide hard X-rays from the 2.5-GeV storage ring. BL-3A was constructed in order to solve the hybrid problem at BL-16, where a single insertion device is used as an undulator for the soft X-ray region and as a multi-pole wiggler for the hard X-ray region. Although it is possible to meet diverse requirements with hybrid use, beam time and working space are restricted, and the experimental systems had to be changed often. With the growth of synchrotron science in general, it is becoming more and more essential to provide application-specific beamlines. Accordingly, the activities carried out at BL-16A using the ID as a multi-pole wiggler source were moved to BL-3A, making BL-16 a dedicated beamline for soft X-ray experiments. The insertion device of BL-16 is to be replaced by an APPLE-II type variable polarization undulator, and the beamline is to be reconstructed in FY2007. Fast polarization switching of soft X-rays will be realized with the future fabrication of a second variable polarization undulator along with a kicker system. At the same time, a second branch was constructed at BL-28. This allows the existing BL-28A to be dedicated to high resolution angle-resolved photoelectron spectroscopy, while the new branch-line BL-28B will be used for other experiments using various techniques.

In order to construct new beamlines with ID sources, some of the dipole beamlines had to be moved or decommissioned. The macromolecular crystallography beamlines BL-6B and 6C completed their roles in March 2006. The X-ray diffraction and scattering beamline BL-3A was moved to the site thus vacated, making room for a new BL-3A. At the same time, the three experimental stations BL-3C1, 3C2 and 3C3 were merged to form a new BL-3C, and the VUV and SX spectroscopy beamline BL-3B was slightly modified.

The 6.5-GeV PF-AR is a good source of hard X-rays when an undulator is used. A new protein crystallography beamline for pharmaceutical research has been designed, with investment support from Astellas Pharma Inc. The beamline is to be constructed at NE3A, where the technique of nuclear resonant scattering using quantum beats measured in the time-domain was developed. The nuclear resonant scattering experiments will be further developed by combination with X-ray diffraction under high pressure at NE1 in the near future. This new facility will become a powerful tool for studying the structure and electronic environment of iron compounds under high pressure.

As well as its high energy capabilities, the PF-AR offers great possibilities for time-resolved studies, since it is dedicated to single-bunch operation, providing a bunch interval of 1.26 µs. As described in the previous volume of this report, a beamline dedicated to time-resolved X-ray diffraction and X-ray spectroscopy was constructed by Prof. Koshihara of the Tokyo Institute of Technology at NW14A in 2005 using an undulator with period length ($\lambda_u$) of 36 mm (U36). This U36 undulator is used with a double-crystal monochromator to provide high intensity monochromatic X-rays with energies between 5 and 25 keV. Another undulator U20, with a $\lambda_u$ of 20 mm was installed in 2006, and provides a ‘narrow-bandwidth white beam’ or a ‘wide-bandwidth monochromatic beam’ with $\Delta E/E \sim 10^{-1}$-$10^{-2}$ at energies between 12 and 18 keV and a photon flux of $\sim 10^{15}$ ph/s.

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

## 1-2 Reconstruction of BL-3A, 3B, 3C, 6B, 6C and 16A

Following the straight-sections upgrade project of the Photon Factory, reconstruction of the beamlines has begun. Following the recommendation of the March 2006 PF External Review Committee to "take the lead in further developing this important research field (VUV/soft X-ray) in Japan", it was decided to bring an end to the time-shared use of VUV and hard X-ray beamlines at the same long straight section. BL-16A was one of these tandem beamlines, although it provided the most intense X-rays of any beamline at the PF, and was a very active station for studies in structural materials science. To cancel the time-shear use and to develop the research, BL-16A has been closed, and its activities have been transferred to a new short-period, short-gap in-vacuum undulator (SGU) beamline, BL-3A. This required the decommissioning of BL-3A, 3B, 3C2, 3C3, 6B and 6C, construction of the new BL-3A, and the reconstruction of BL-3B, 3C, and 6C. Activities previously undertaken at BL-3A have been transferred to a reconstructed BL-6C.

# 1-3 The New Short-Gap Undulator Beamline BL-3A for Structural Materials Science

### *Introduction*

Following the straight-sections upgrade, four short straight sections were created, allowing for the installation of additional short-gap undulators (SGUs). Beamline BL-3A, which is dedicated to diffraction measurements for structural materials science, is the second SGU beamline at the PF ring. It was constructed in the summer of 2006, and the first beam was delivered on October 5, 2006. Following commissioning of the beamline, user operation began in January 2007.

### *Optics and equipment*

A schematic view of BL-3A is given in Fig.1. The beamline optics consists of a short-gap undulator, a water-cooled flat double-Si(111) monochromator at 26 m from the light source, an in-vacuum phase retarder at 28 m, a bent cylindrical mirror at 30 m, a Huber 5020 6-circle diffractometer at 36 m, and a Cryo Industries 8-T superconducting magnet with a custom-made large two-circle diffractometer at 39 m. A polarization analyzer and a CCD detector are available for both diffractometers, and a 5.5-K refrigerator, a 10-K refrigerator with diamond anvil cell and a 1000-K furnace are available for the 6-circle diffractometer. Users can select between the two diffractometers by changing the focusing point.

### *Performance*

The energy range of the beamline is 4 keV to 14 keV, and the maximum photon flux available is 6 × $10^{12}$ photons/s at 6.56 keV with a spot size of 0.6 mm (horizontal) × 0.2 mm (vertical). The beam position is fixed within 0.2 mm in the photon energy range of 5 keV to 14 keV. The phase retarder can be used to produce a left- or right-handed circularly polarized beam, a vertically polarized beam, or a horizontally polarized beam with a photon flux of 3 × $10^{11}$ photons/s. The diffractometer, monochromator, phase retarder and undulator can be controlled simultaneously using the SPEC software and STARS. This allows measurement of an energy spectrum for a specific Q-vector with optimum undulator gap and controlled polarization.

### *Test measurements and future plans*

Standard in-vacuum phase retarders suffer from instabilities due to vacuum chamber distortion. To address this problem, we have installed an in-vacuum level block as a stage of the phase retarder system that has a multiple-crystal-holder that enables us to choose the best crystal for phase shifting at a particular X-ray energy. To test the new phase retarder, an MCD spectrum for an Fe film was recorded using magnetic field modulation method. The spectrum, shown in Fig. 2, was recorded in less than 30 minutes, and a clear MCD spectrum was observed. This result demonstrates the potential of the new beamline for polarized X-ray appli-

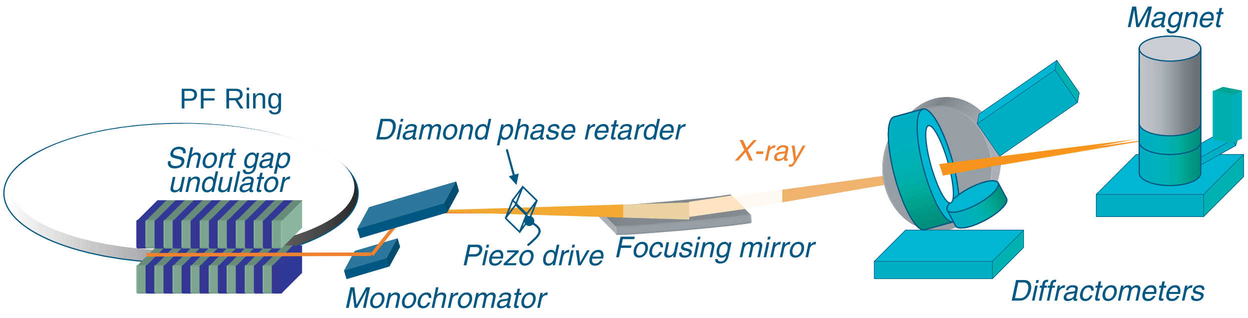

Figure 1
Schematic view of BL-3A.

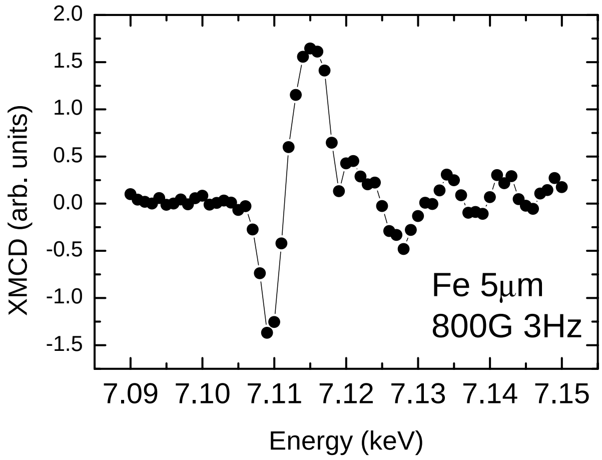

Figure 2
The first MCD spectrum recorded at BL-3A using the magnetic field modulation method.

Figure 3
Current view of BL-3A, along with members of the beamline construction team.

cations.

During the summer of 2007 a monochromator stabilization system and a piezo-drive for the phase retarder will be installed. These features will improve the beamline's capabilities for precision experiments and highly sensitive magnetic scattering measurements.

## 1-4 A Fast Polarization-Switching Beamline for Spectroscopic Studies in the Soft X-Ray Energy Region

The program to refurbish the 2.5-GeV PF-ring lattice completed during the 2005 summer shut down has resulted in the creation of four 1-m short-straight sections and the elongation of the existing straight sections. It is crucially important to make full use of all straight sections over the short and medium time span. Short-period small-gap undulators are to be installed in the newly created short straight sections to provide high intensity, well-collimated X-rays, while various types of undulator are to be installed in the elongated medium and long straight sections to promote activities in the vacuum ultraviolet and soft X-ray (VUV-SX) energy region. This strategy for the straight sections was strongly supported by the International PF Review Committee held in March 2006, which urged the PF to take the lead in developing the VUV-SX research field following the decision by the Univ. of Tokyo not to build a new 3rd generation VUV-SX facility.

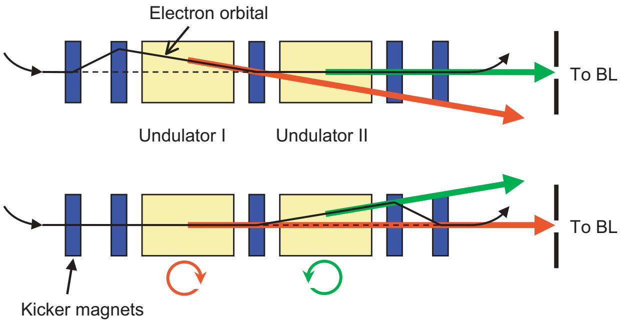

Figure 4
Twin APPLE-II undulators together with five kicker magnets produce right and left circular polarization selectable with a ≈10-Hz switching speed.

Over the last decade, we have studied the feasibility of a fast polarization-switching spectroscopic facility in the soft X-ray energy region using a ≈9-m long straight section. The main scientific targets are investigations of nano-scale magnets, strongly correlated electron systems and critical phenomena near phase-transition regions using soft X-ray magnetic circular/linear dichroism (XMCD/XMLD), as well as studies of chiral molecules and biomolecules using soft X-ray natural circular dichroism (XNCD). The construction of such a facility at BL-16, where a 9-m long straight section is available to be fully utilized for insertion devices, was approved by the PF Program Advisory Committee in July 2006. Fig. 4 shows a schematic of the expected realization of fast polarization switching using two APPLE-II type undulators and five kicker magnets. The upper panel of the figure shows the left circularly polarized beam from Undulator II being delivered to a newly constructed beamline, and the lower panel shows the right circularly polarized beam from Undulator I being delivered to the beamline. The polarization switching speed is expected to be ≈10 Hz. The twin APPLE-II undulators can also be used to supply linearly polarized light, offering the opportunity to study linear dichroism. Fig. 5 shows a schematic of the spectroscopic beamline. A varied-line-spacing grating (VLSG) monochromator is adopted, since there is a wealth of experience at the PF in VLSG monochromators following the installation of the first one at BL-11A. The combination of the twin APPLE-II undulators with the VLSG monochromator is expected to cover a photon energy region of 200-1500 eV with the fundamental and third harmonics of the undulator radiation. It has been strongly requested that the photon beams from the two undulators have equal intensities and the same size at the sample position, to allow CD/LD to be studied precisely using lock-in detection.

Unfortunately, budgetary limitations constrain us to initially construct only a single APPLE-II undulator and the new beamline. The first undulator radiation is expected to be observed in May 2008, following which we plan to begin scientific activities, which do not nec-

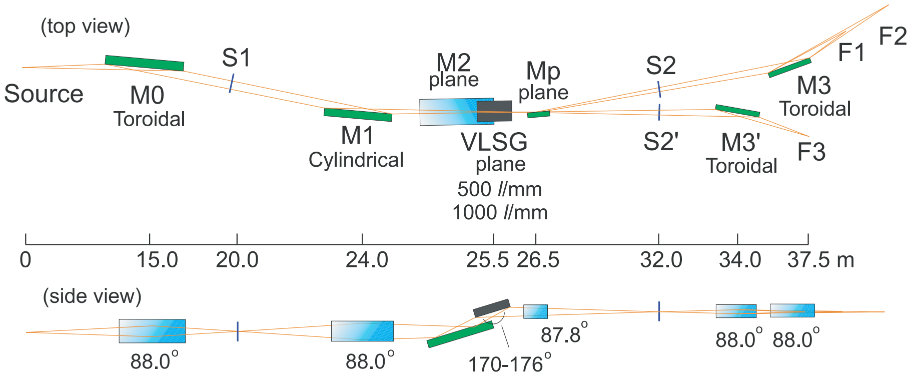

Figure 5
Schematic of the optical layout of the high-resolution VLSG monochromator beamline in the soft X-ray energy region adopted for the fast polarization-switching spectroscopic facility at BL-16.

essarily call for fast polarization switching. Good candidates include studies of 1) strongly correlated electron systems and nano-scale spintronic magnets which are expected to show large XMCD, 2) chemical reactions of surface adsorbed systems and dynamics of surface magnetism, 3) correlation between nano structures and electronic states with resonant SX magnetic scattering, and 4) magnetism imaging of mesoscopic magnets using a photoelectron emission microscope. It is expected that high-quality scientific achievements in these areas will strongly promote the realization of the second undulator installation.

## 1-5 Commissioning of a New Branch Beamline BL-28B

BL-28A is an undulator beamline that covers the photon energy range of 30 to 300 eV using an entrance-slitless varied-line-spacing plane-grating monochromator (VLSPGM). The beamline is mainly used for angle-resolved photoemission spectroscopy (ARPES) experiments, and offers relatively high energy resolution along with high photon flux. Many ARPES experiments (see the Electronic Structure section of the Highlights in this Report) have successfully been performed at the beamline over the past two years. The ARPES endstation (SES-2002) is fixed at the beamline, and a new branch beamline was needed in order to satisfy demands for various other experiments requiring high energy resolution and high-flux in the vacuum ultraviolet (VUV) region. This new branch beamline, BL-28B, was constructed during the summer shutdown period of 2006.

The new branch beamline shares the monochromator of BL-28A, and a transferrable plane mirror is used to direct monochromatized photons into the new branch. Commissioning was completed after solving problems due to vibrations from the vacuum pump, and BL-28B is now open for users. Various experiments were successfully carried out at BL-28B in FY2006 in fields including atomic and molecular physics, *in situ* photoemission with pulsed laser deposition, and photoemission electron microscopy (PEEM).

## 1-6 AR-NE3A, A Protein Crystallography Beamline for Pharmaceutical Research

Recent advancements in high-throughput techniques in macromolecular crystallography have heightened the importance of structure-based drug design, and demand for beamtime for such purposes has correspondingly increased. In order to meet this demand, the Photon Factory has decided to reconstruct AR-NE3A as a new beamline dedicated for a high-throughput protein crystallography, especially for a pharmaceutical research, in a partnership with Astellas Pharma Inc.

In pharmaceutical research, a large amount of samples are required to be examined in a short period. This is because researchers often need structural information of a target protein in complex with as many compounds as possible, and the new beamline has been designed for such experiments. A schematic layout of new AR-NE3A is shown in Fig. 6. The light source is an in-vacuum undulator, providing a high flux X-ray beam. The main optical components are similar to those of AR-NW12A and BL-5A [1, 2], the high-throughput protein crystallography beamlines which were installed at the Photon Factory over the past several years. A collimating mirror, double crystal monochromator with liquid nitrogen cooling system, and a toroidal focusing mirror are located in the optical hutch. Ray-tracing simulations suggest that the new AR-NE3A affords higher X-ray beam flux at the sample position than either AR-NW12A or BL-5A, although the beam divergence is larger (Table 1). In the experimental hutch, there are three main components, a diffractometer, an X-ray detector and a sample exchanger. The diffractometer has a high-precision sample rotation axis and a high speed shutter, both of which were developed in collaboration with KOHZU Precision Co., Ltd (Japan). The X-ray detector is a CCD-type detector with high sensitivity, a large active area, and high-speed readout. The sample exchanger, developed in collaboration with SSRL (USA), allows for automatic sample exchange, and can store 288 cryo-cooled samples in a Dewar [3].

The reconstruction of AR-NE3A will begin in March 2008. The first beam will arrive at the experimental sta-

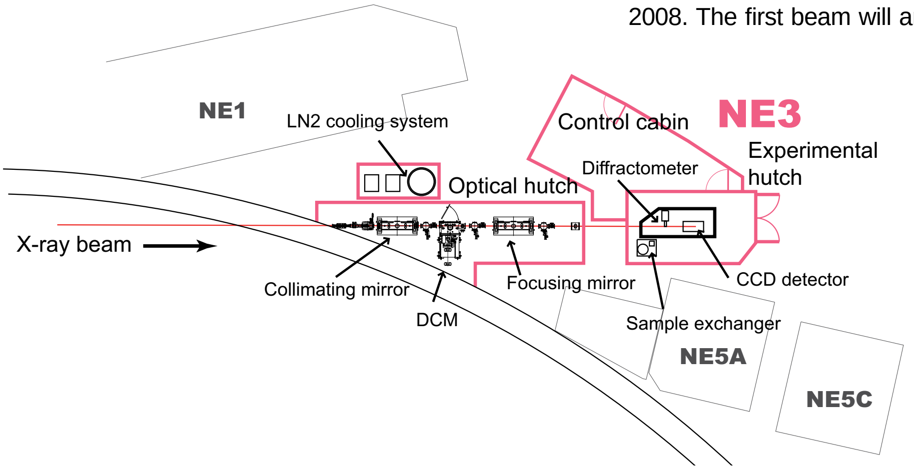

Figure 6
Schematic view of AR-NE3A.

Table 1 Comparison of ray-tracing simulation results for three beamlines.

| | Flux* (phs/s) | Size H × V (mm$^2$) | Div H × V(mrad$^2$) | $\Delta E/E$ |
|---|---|---|---|---|
| AR-NE3A | $1.8 \times 10^{12}$ | 0.736 × 0.151 | 0.957 × 0.284 | $1.6 \times 10^{-4}$ |
| AR-NW12A | $4.6 \times 10^{11}$ | 1.470 × 0.223 | 0.485 × 0.125 | $1.5 \times 10^{-4}$ |
| BL-5A | $6.9 \times 10^{11}$ | 0.973 × 0.195 | 0.673 × 0.211 | $1.4 \times 10^{-4}$ |

* These values represent the flux through a 0.2 × 0.2 mm$^2$ slit or a 0.2 × 0.2 mm$^2$ pinhole at the sample position. The actual flux measured at AR-NW12A and BL-5A is about half the flux given by the simulations.

tion in October 2008, and the beamline will be open to users from April 2009. The beamline is funded by Astellas Pharma Inc., who will be allocated a portion of the available beam time for research. The remaining beam time will be offered to general users, and this is expected to promote the further development and utilization of protein crystallography beamlines at the Photon Factory.

## References

[1] *Photon Factory Activity Report*, **20A** (2003) 64.

[2] *Photon Factory Activity Report*, **21A** (2004) 61.

[3] M. Hiraki, S. Watanabe, Y. Yamada, N. Matsugaki, N. Igarashi, Y. Gaponov and S. Wakatsuki, *AIP Conf. Proc.* , **879** (2006) 1924.

## 1-7 AR-NW14A: A Beamline for Time-Resolved X-Ray Research

AR-NW14A is an in-vacuum undulator beamline at the PF-AR aiming for 100 picosecond (ps) time-resolved X-ray diffraction, scattering and absorption experiments [1]. The dedicated single-bunch operation of the PF-AR enables us to explore a wide variety of time-domain sciences in physics, chemistry and biology at 100 ps resolution. The primary scientific goal of the beamline is to investigate the dynamics triggered by short laser pulses in condensed matter such as organic and inorganic crystals, protein crystals and liquids.

AR-NW14A has two undulators with period lengths of 36 mm (U36) and 20 mm (U20). The measured photon flux density from each undulator is shown in Fig. 7. The 1st harmonic of U20 provides photons in the energy range of 12-18 keV with a bandwidth $\Delta E/E \sim 10^{-1}$. Since most time-resolved experiments are "photon-demanding", U20 is useful as a wide-bandpass, high-flux X-ray source with a photon flux of $\sim 10^{15}$ photons/s at $\Delta E/E \sim 10^{-1}$. U36 covers the energy range of 5-25 keV with its 1st, 3rd, and 5th harmonics, and is used as an intense, tuneable, monochromatic X-ray source. The typical photon flux of the monochromatic beam is $\sim 10^{12}$ photons/s with an energy bandwidth of $\Delta E/E \sim 10^{-4}$. The X-ray pulses are delivered to the experimental hutch at the PF-AR revolution frequency of 794 kHz with a pulse duration of ~100 ps (FWHM).

The experimental equipment installed at AR-NW14A is shown in Fig. 8. Three different diffractometers (Huber 4-axis, RIGAKU Imaging Plate and MarCCD) have been installed for various time-resolved diffraction and scattering applications. A femtosecond (fs) laser system (Spectra Physics Millennia, Tsunami, Evolution and SpitFire) and a nanosecond laser system (Continuum PowerLite8000) are installed in the laser booth.

The synchronization scheme for the laser and X-ray pulses at AR-NW14A is shown in Fig. 9. The timing control is based on the RF master clock (508 MHz) that drives the single electron bunch in the ring. The X-ray chopper (pulse selector) is phase-locked at 945 Hz and isolates X-ray pulses at 945 Hz from the 794 kHz X-ray pulse train. The mode-locked Ti:sapphire laser is oper-

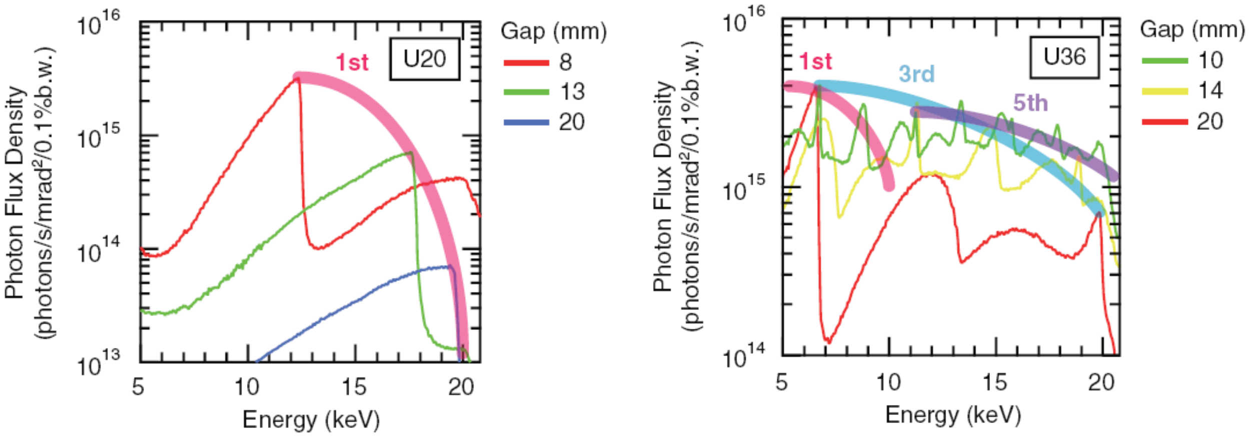

Figure 7
Photon flux density from U20 and U36.

ated as the seeding laser and is guided to the regenerative amplifier operated at 945 Hz. Thus, the 100 ps X-ray and the 150 fs laser pulses are synchronized at 945 Hz.

The beamline has been fully operational since 2006, and various pump-probe experimental projects are underway. Scientific output from AR-NW14A is reported elsewhere.

## Reference

[1] S. Nozawa S. Adachi, J. Takahashi, R. Tazaki, L. Guérin, M. Daimon, A. Tomita, T. Sato, M. Chollet, E. Collet, H. Cailleau, S. Yamamoto, K. Tsuchiya, T. Shioya, H. Sasaki, T. Mori, K. Ichiyanagi, H. Sawa, H. Kawata and S. Koshihara, *J. Synchrotron Rad.*, **14** (2007) 313.

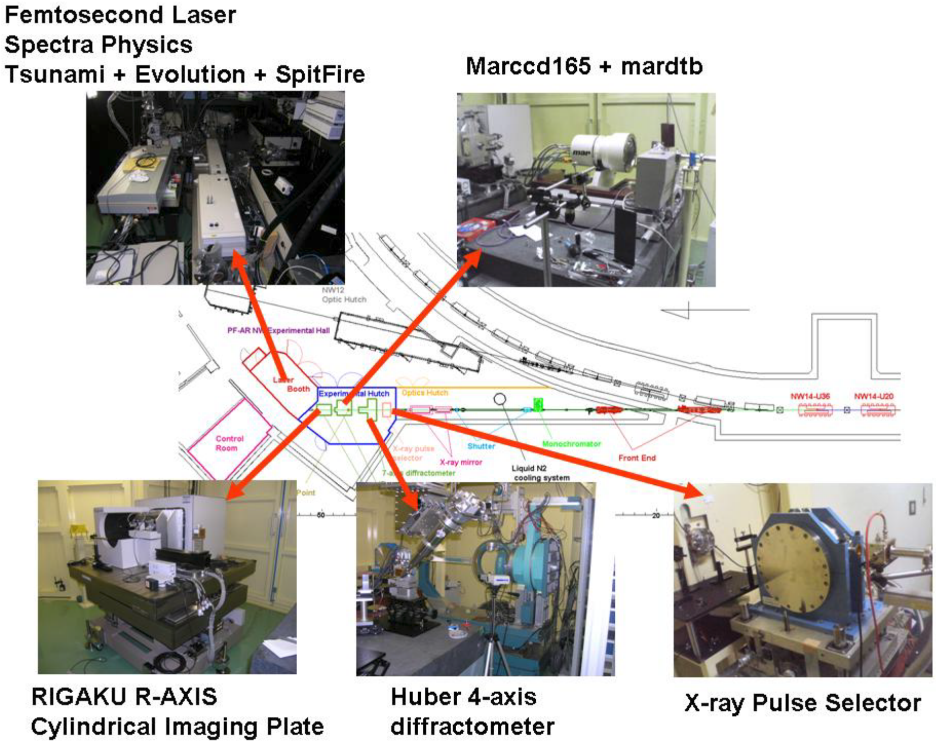

Figure 8
Experimental apparatus at AR-NW14A.

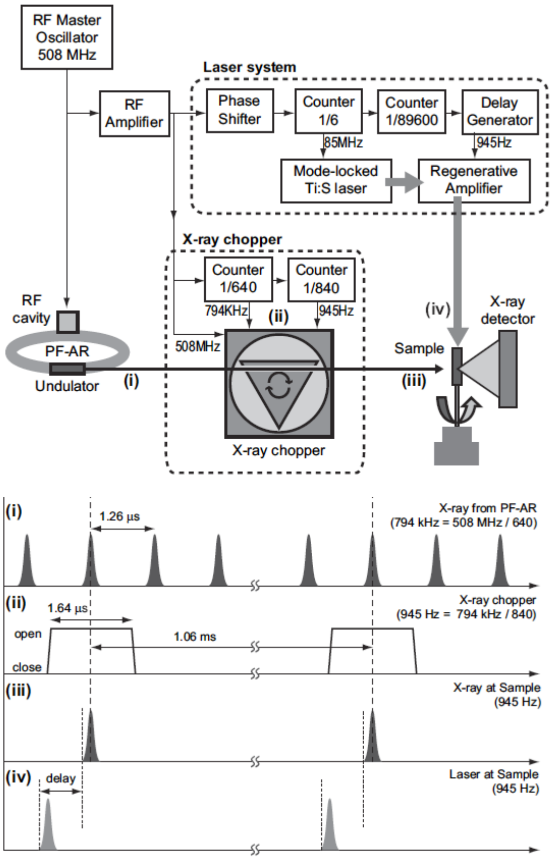

Figure 9
Typical timing scheme at AR-NW14A.