2. Reconstruction and Upgrading of the Beamlines

2-1. BL-7A, New Soft X-Ray Beamline for Surface Chemistry

The construction and commissioning of a new beamline, BL-7A [1], has been completed by Research Center for Spectrochemistry (Univ. of Tokyo) in collaboration with the Photon Factory. Figure 1 gives photon flux distribution curves for 150-, 300- and 650-l/mm varied-line-spacing plane gratings. Soft X-rays in the energy range 50-1500 eV are available by using the three gratings. Note here that the resolving power alters as a function of the photon energy because of the fixed slit openings. In order to estimate the ultimate resolving power, the total ion yield spectra of gaseous N_2 and Ar were recorded, which are shown in Fig. 2.

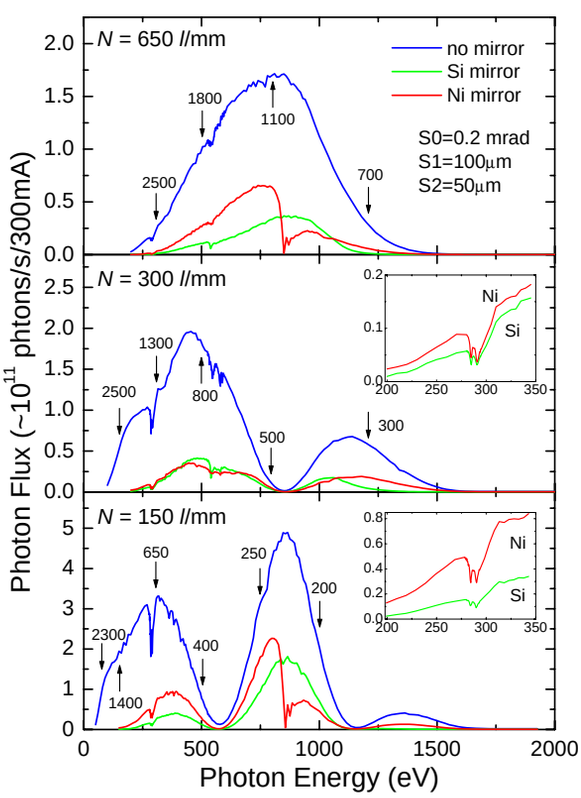


Figure 1.

Photon flux distribution curves for three gratings obtained without and with Si and Ni mirrors for harmonic rejection. The resolving power (E/ΔE) is indicated by arrows at several photon energies.

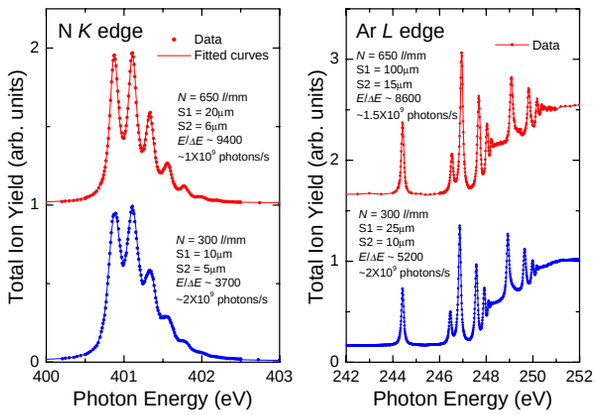


Figure 2.

Total ion yield spectra of N_2 and Ar taken with 300- and 650-l/mm gratings. The resolving power was estimated by fitting with voigt functions assuming Lorentzian peak widths of 117 and 113 meV for N_2 and Ar, respectively [3].

One of the main objectives of this beamline is to perform C K-edge XAFS experiments for surface adsorbates. Thanks to a moderate decrease in the photon flux around the C K edge, monolayer adsorbates can be investigated, as shown in Fig. 3. In addition to the conventional XAFS measurements, a new technique, called 'energy dispersive NEXAFS', has been developed. The principle of the measurements is described elsewhere [2]. In short, dis-

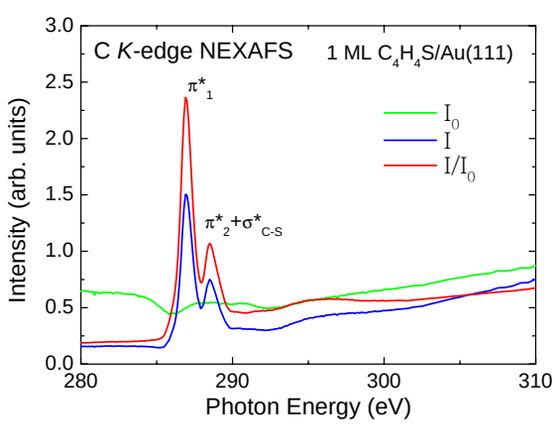


Figure 3.

C K-edge NEXAFS spectra of a thiophene (C_4H_4S) monolayer adsorbed on Au(111) taken in a partial electron yield mode.

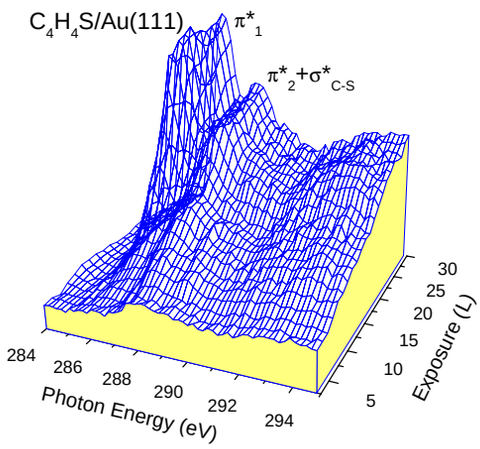
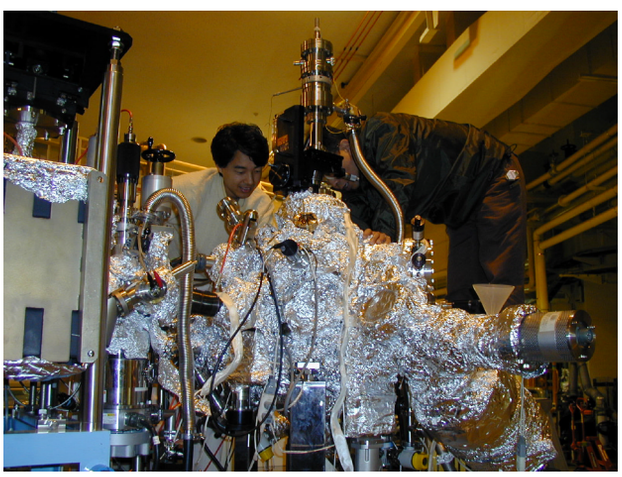


Figure 4.
In situ C K-edge energy-dispersive NEXAFS spectra recorded during thiophene adsorption. The accumulation period for each spectrum was 30 s. An exposure of ~10 L corresponds to the completion of a monolayer.

persed X-rays illuminate the sample surface, and Auger electrons emitted from each position of the surface are collected by a position-sensitive electron analyzer (GAMMADATA-SCIENTA, SES-2002). Thus, the Auger electron yield NEXAFS spectra are obtained by one shot. A typical example of the dispersive NEXAFS measurements is given in Fig. 4.

References
[1] Photon Factory Activity Report #17 (2000), A-89.
[2] K. Amemiya et al., Jpn. J. Appl. Phys. Exp. Lett., 40 (2001) L718.
[3] M. Watanabe et al., Proc. SPIE 3150 (1997) 58.



2-2. BL-11D, VUV-SX Beamline for Photoemission Experimentalists

BL-11D was designed for the photoelectron spectroscopy of solids and solid surfaces in the vacuum ultraviolet and soft X-ray region [1]. For this research field, (1) a small spot size and (2) a minimal intensity of higher-order light, are required. A negative incident-length varied deviation angle spherical grating monochromator is adopted for this purpose. Although the beamline construction started in 1996, it was opened for users in May 2000, taking a longer time than scheduled. This is because we experienced more mechanical troubles due to large rotation angles of the plane mirror. The optics of BL-11D is shown in Fig. 5. Two gratings (G1 and G3) cover the photon energy range from 60 eV to 245 eV (G3) and from 200 eV to 900 eV (G1). The energy resolution (E/ΔE) is about 2000 at the relatively wide slit size of 200 μm × 100 μm; this number should be better when reduced slit sizes are used. The photon flux is about 10^{11} photons/s at 200 μm × 100 μm, hν~100 eV (G3), and the ring current of 400 mA. The beam size is 1 mm(H) × 0.1 mm(V) for both G1 and G3. Typical Au Fermi-edge spectra taken at 200 μm × 100 μm at 78 K are shown in Fig. 6.

A high energy-resolution photoemission spectrometer with a Scienta SES-200 photoelectron analyzer is always connected to the beamline so that users can immediately start their experiments only with loading samples. Figure 7 shows a schematic drawing of the photoemission measurement system

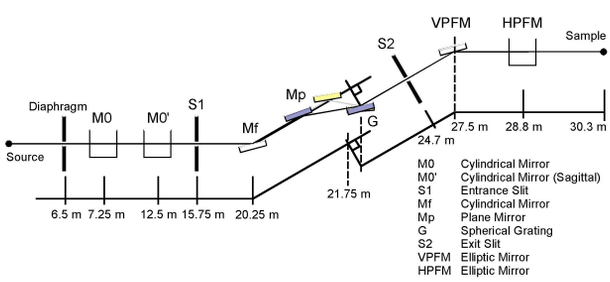


Figure 5.
Schematic drawing of the optical design of BL-11D.

at BL-11D. Solid samples can be transferred onto the temperature-controlled manipulator in the preparation chamber without breaking the vacuum using a sample-transfer system. The achieved total energy resolution of the analyzer is 19.6 meV. The temperature range is from ~15 K to 300 K using a He-flow type cryostat. For sample surface treatments, a diamond file and a cleaver for scraping or cleaving/fracturing samples can be used. Auxiliary UV and X-ray sources are also available.

BL-11D has been open for photoemission users from May 2000. In the year 2000, 11 experimental runs from 8 proposals were performed. Most of the measured samples were strongly correlated electron systems of bulk 4f and 3d transition-metal compounds. Since the beamline is suitable for the energy range of 90-130 eV, 4d-4f resonant photoemission experiments of Ce, Pr, Eu, Yb, etc. compounds have been performed. BL-11D is also suitable for photoemission measurements of 3d transition-metal oxides using the energy range of 150 to 200 eV where the photoionization cross section of oxygen becomes substantially smaller than that of 3d transition metals. Using this energy range, photoemission studies of perovskite-type iron or manganese oxides have been performed. We are calling for proposals on bulk samples of such 4f, 3d, and 4d electron compounds from anyone who is interested in quick, convenient, and accurate photoemission experiments.

In the future, the performances of both G1 and G3 will be improved by further fine optimizations. As

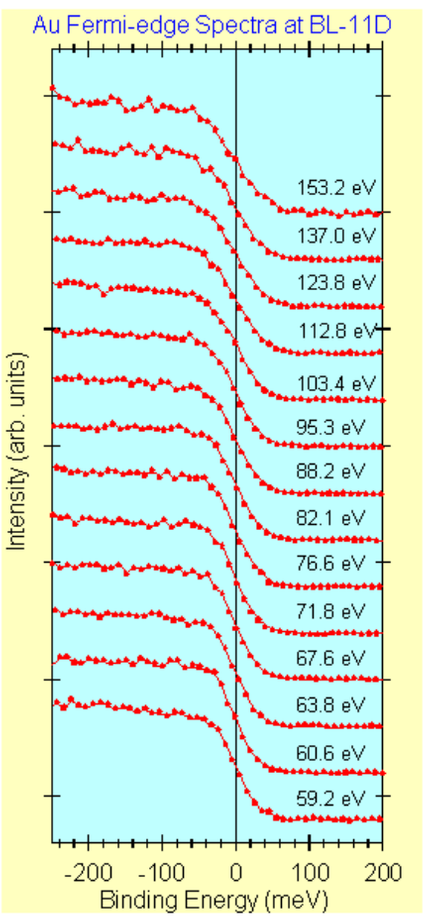


Figure 6.
Au Fermi-edge spectra in the photon energy range of 60 to 150 eV. The entrance and exit slits were 200 μm × 100 μm and the analyzer (SES-200) slit was the curved 0.8 mm. The temperature was 78 K.

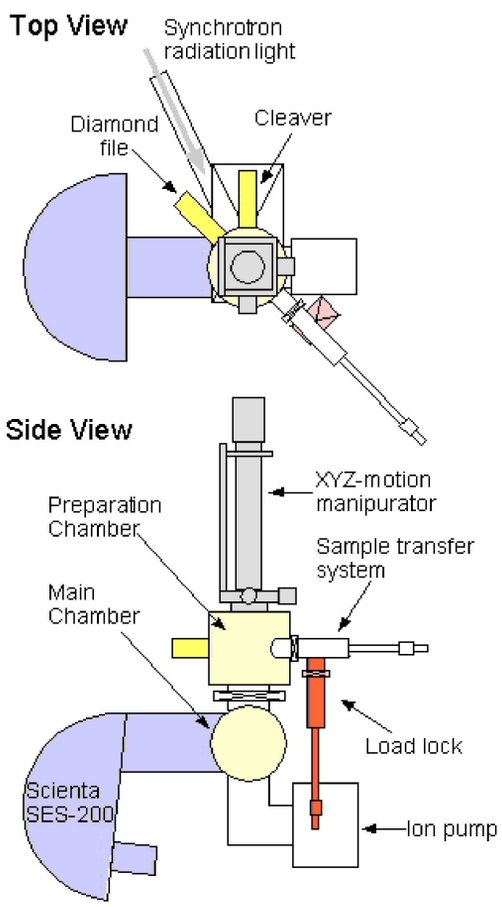


Figure 7.
Schematic side view of the photoemission spectroscopy system at BL-11D The synchrotron light horizontally comes into the main chamber and the light-to-analyzer angle is 65 degrees.

for the end-station, the best energy resolution of the SES-200 photoelectron analyzer will be improved by using a lower pass energy.

Reference

[1] S. Suzuki, Photon Factory Activity Report 1997 #15 (1998) A101.

2-3. BL-9C, Versatile X-Ray Beamline

BL-9C was kindly donated to the Photon Factory by NEC Corporation in September, 2000. They used this beamline since 1985, and the present one was constructed in 1997. This beamline consists of a double-crystal monochromator and a bent cylindrical focusing mirror, as shown in Fig. 8. The mirror is placed at 16.1 m and the focus is expected at 30 m from the source point, thus satisfying the nearly 1:1 focusing condition. The maximum usable energy is 23 keV, which is limited by the critical energy of the mirror, though higher energy X-rays can be used by removing the mirror. The lower energy is limited by the absorption with windows and acceptable higher order contents for experiments. Also, white X-rays can be used by removing the first crystal of the monochromator.

An evaluation and commissioning of the beamline started in October, 2000. The problems found in the monochromator were solved during the winter shutdown between late December and early January. Usually, beamlines terminate with a 0.2 mm thick Be window followed by a Kapton window. However scattering from the Be window is not desirable for small-angle X-ray scattering (SAXS) experiments. Therefore, the Be window at 28.1 m was moved to 20.2 m, and a slit system was installed at 24 m.

Some groups are expected to be users of this beamline. One is time-resolved XAFS using dispersive optics, which needs white X-rays. The second is X-ray diffraction experiments using a six-circle diffractometer. The third is small- and medium-angle X-ray scattering experiments; these two activities require focused monochromatic X-rays. However, BL-9C will not be dedicated for specific research or equipment, and will be opened for wide research fields, since there are only a few versatile X-ray experimental stations in the Photon Factory.

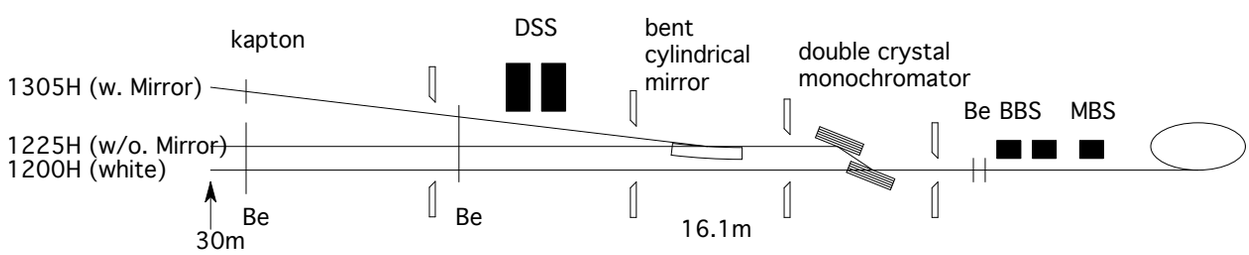


Figure 8.

Schematic illustration of BL-9C. Three modes of the X-ray beam can be exchanged by inserting a monochromator and a mirror; i.e., white X-rays, focused monochromatic X-rays (< 23 keV) and high-energy monochromatic X-rays.

2-4. New BL-13A for Laser-Heating High-Pressure and High-Temperature Studies

BL-13A was first built in FY1990 under a cooperative project between National Institute for Measurement and KEK. In FY1999 this beamline was reconstructed to take over the activities at BL-13B2, where high-pressure and high-temperature experiments had been carried out with diamond anvil cells (DAC) and laser-heating equipment. The new beamline provides more intense 30-keV X-rays than the previous one [1] with newly designed optics, even though the source (MPW#13) is the same. A double-crystal monochromator and a pair of focusing mirrors are installed in the beamline (Fig. 9). The monochromator, which is equipped with two flat crystals, has no function to sweep the energy over a wide range due to a geometrical limitation. The first crystal is a water-cooled Si(111) and the second is an asymmetrically-cut Ge(111) with an asymmetrical factor of b = 7.75. The monochromator is designed so that the throughput is multiplied and the beam width is reduced to the aperture of the horizontal mirror. The horizontal and vertical mirrors, which are arranged in the Kirkpatric-Baez type optics, focus the beam onto a point 31.1 m from the source. They are both made of Pt-coated fused quarts and a fixed-radius type cylindrical mirror with a critical energy of E_c = 30 keV. The horizontal mirror is located 29.2 m from the source and the radius of concavity is 1497.52 m. The vertical mirror is located 30.1 m inside the experimental hutch and its radius is 792.95 m. The optics has been used to focus high-flux photons onto a very small sample area in a DAC, where high-temperature (> 3000 K) and high-pressure (> 100 GPa) conditions are attainable by YAG-laser heating.

In the experimental hutch, the YAG-laser heating optical system and X-ray powder diffraction equipment including an imaging plate (IP) read-out system (R-AXIS-IV, RIGAKU Co.) were installed by Professor Yagi of ISSP, Univ. of Tokyo. The diffraction pattern on an IP (two-dimensional intensity data) can be transformed into one-dimensional intensity data in the 2θ range by the PIP program in the same way as in BL-18C with a network computer connected to the R-AXIS system. These systems are being progressively opened for general users.

<Performance of beamline>
Energy: 29.087 keV (30 keV in design)
Energy Resolution: ~2 ($\Delta E/E \times 10^{-4}$)
Beam size: ~70 μmV × ~80 μmH
(FWHM, measured with 50 μm square slit;
31.8 μmV × 45.4 μmH in design)
Photon Flux: ~5 × 10^{11}/s.mm^2

The observed intensity was more than 1-order higher than that at the previous beamline. Figure 10 shows a comparison of diffraction patterns obtained at BL-13A and 13B2. It proves the high performance of the new BL-13A.

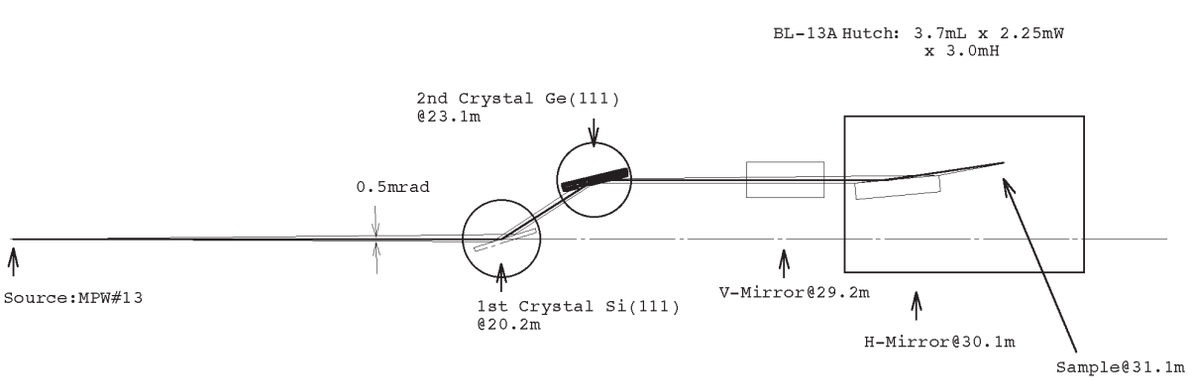
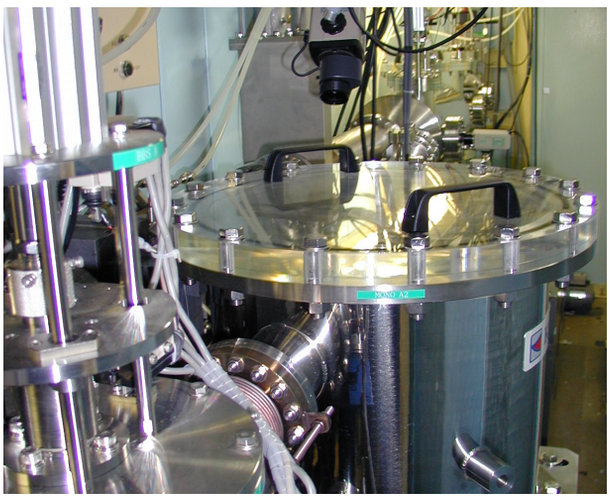


Figure 9.
Schematic illustration of BL-13A.



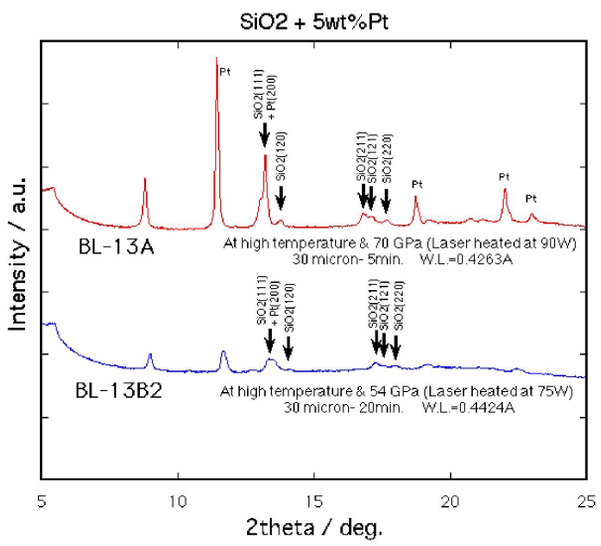


Figure 10.
Diffraction patterns of SiO_2 observed under nearly the same conditions, but for different measuring times. The intensities of the diffraction peaks at BL-13A are remarkably improved as compared with those at BL-13B2 for shorter times.

It is planned to replace the Imaging Plate by an X-ray CCD (C4880, HAMAMATU Co, Japan; an effective diameter ϕ160 mm) to shorten the recording time in the laser-heating high-pressure X-ray diffraction experiments, such as time-resolved observation in kinetic studies.

References

[1] Photon Factory Activity Report 1996 #14 (1997) E-7.

2-5. Diamond Crystal Monochromator for an X-Ray Undulator Beamline, AR-NE3A

Because diamond crystals have excellent anti-thermal features, they are widely used for high heat-load monochromator. There are, however, two problems with them: 1) the sizes of the available diamond crystals are small (around 10 mm in diameter); 2) there is no reliable way to mount a diamond crystal without introducing lattice strain. To overcome the second problem, we developed a low-stress crystal-mounting method [1]. We also improved the monochromator of beamline AR-NE3A by replacing silicon crystals with diamonds.

Figure 11 schematically shows the mounting method of diamond crystals. Two <111>-oriented diamonds of type Ib were used: one for X-ray diffraction (8 mm × 4 mm × 1 mm) and the other for reducing distortion (4 mm × 3 mm × 2 mm). We brazed the diamonds with Ag-Cu alloy and then soldered them. The diamonds holder is an oxygen-free

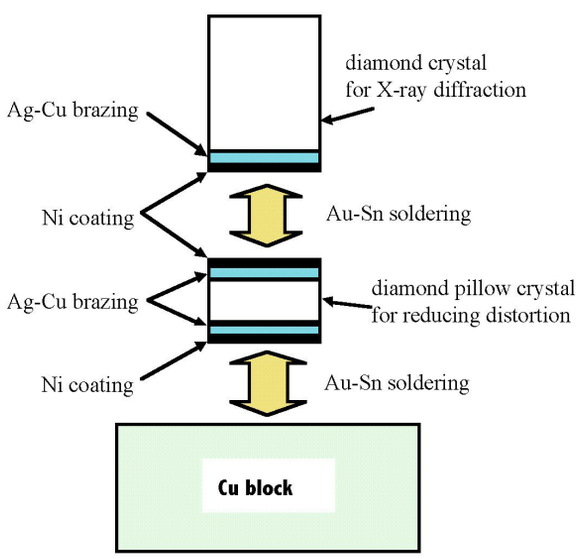


Figure 11.
Structure of diamond crystal mounting. The crystals were brazed with Ag-Cu alloy before soldering. Nickel was also coated to prevent any chemical reaction between the soldering flux and Ti included in the Ag-Cu alloy.

copper block with a water channel for cooling. In order to improve the heat-transfer coefficient on the inner wall of the channel, a wire coil was inserted in the channel [2].

We estimated the crystalline property of diamonds by measuring the rocking curves of the 111 Bragg reflection. Figure 12(a) shows a rocking curve measured before soldering. The FWHM is 5.4 arc seconds, which is slightly larger than that for a perfect crystal (4.3 arc seconds at the wavelength of 1.396 Å). Figure 12(b) is a rocking curve measured after soldering. The diamond was soldered onto the cooling holder with Au-Sn alloy (the melting point is 280°C). The FWHM is 20% larger than that of Fig. 12(a) due to the soldering process.

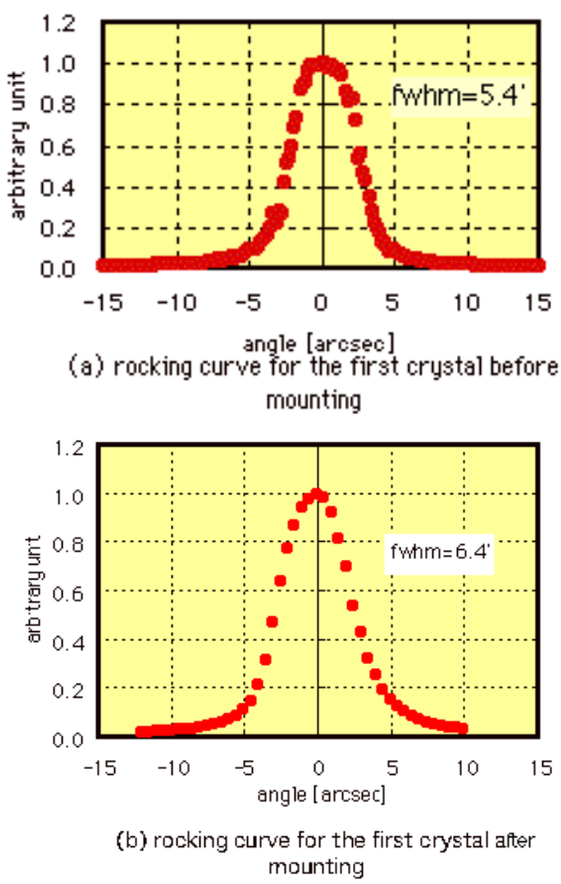


Figure 12.
Symmetric 111 Bragg reflection of a diamond crystal, (a) before and (b) after crystal mounting, respectively. The width of curve (b) is 20% wider than (a) due to the crystal mounting process.

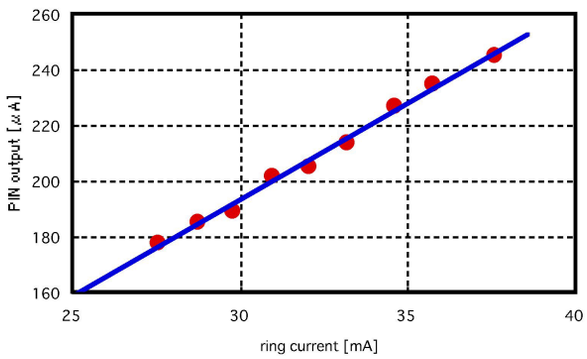


Figure 13.
Throughput of the diamond monochromator versus the ring current.

To improve the monochromator of beamline AR-NE3A, we replaced the silicon crystals with diamonds. For commissioning the monochromator, we measured rocking curves under several conditions of the SR beam power and power density. When the SR power was low, the FWHM remained constant. However, the FWHM began to gradually increase above a power density of 1.8 W/mm^2. Figure 13 shows the throughput of the monochromator at various ring currents. The intensity of 14.4-keV photons is proportional to a ring current of up to 40 mA. It is worth noting that throughput saturated at 30 mA due to thermal deformation when the water-cooled silicon crystals was used. This result shows the advantage of diamond crystals.

References

[1] T. Takiya, H. Sugiyama, X.W. Zhang, S. Shimada, K. Yamazato, A. Komura and M. Ando, Rev. Sci. Instrum., 70 (1999) 3523.

[2] T. Takiya, T. Mochizuki and H. Kitamura, SPring-8 Annual Report 1998, 164.