4.3 Beamlines Improvement

4-3-1 The Soft X-Ray Capability of the XAFS Station, BL-9A

BL-9A was constructed as a beamline for XAFS experiments using a bending magnet source. A pair of bent conical mirrors is used for collimating and focusing the X-rays and a Si(111) double crystal monochromator is placed between them. Owing to this special optics BL9A provides high flux (6×10^{11} ph/s at 7 keV, 450 mA) in a 1 mm × 1 mm area without sacrificing energy resolution [1-4]. The maximum usable photon energy is designed to be 15 keV, limited by the critical angle of the mirrors.

In conventional X-ray beamlines, three 0.2 mm thick beryllium windows are placed between the storage ring and the experimental setup, and XAFS experiments are carried out under atmosphere. However the transmission of 2.1 keV X-rays (corresponding to the P K-edge) is less than 0.1% due to the presence of the Be windows. Thus most experiments using soft X-rays below 4 keV are carried out under a vacuum environment, limiting the kind of samples and environments available for study and lengthening experiment turn-around times.

Besides the features described above, BL-9A is designed to provide X-rays with energies down to 2.1 keV. In order to realize such performance, the number and thickness of the Be windows is limited; only two 0.1 mm thick Be windows are present in the beamline. Furthermore, a pair of flat mirrors is installed in order to reduce higher order reflections. By using this equipment and a fluorescent ion chamber [5], a XANES spectrum of Ar in air was easily obtained (Fig. 1). However it was not easy to measure XAFS spectra of sulfur or phosphorus with the usual setup owing to the absorption of the X-rays by air. Thus we have constructed a new, simple setup as shown in Fig. 2. The environment between the exit window of the beamline and the sample, including the entrance slit and I_0 monitor, is replaced by helium. Since the sample environment is a He atmosphere it is easy to use the conversion electron yield (CEY) detection method as well as fluorescent yield (FY) measurements. 700 V is applied to a thin aluminized Mylar film placed before the entrance window of the fluorescence detector and the CEY signal is obtained by directly measuring the sample current.

Powdery samples are usually rubbed on adhesive tapes, but most commercially available adhesive tapes contain sulfur compounds; an example is shown in Fig. 3. 3M's "Clear Tape" and Nichiban's "Nice tac" showed very little sulfur K-edge and so were used to support powdery samples in the FY detection mode. On the other hand Scotch Graphite tape AL-25DC was used despite the fact that it contains sulfur for the CEY detection mode in order to realize electrical conductivity.

References

[1] *Photon Factory Activity Report* **1996 #14** (1997) E-5.
[2] *Photon Factory Activity Report* **1999 #17** (2000) A90.
[3] M. Nomura and A. Koyama, *J. Synchrotron Rad*. **6** (1999) 182.
[4] M. Nomura and A. Koyama, *Nucl. Instrum. and Methods Phys. Res. A* **467-468** (2001) 733.
[5] F.W. Lytle, R.B. Greegor, D.R. Sandstrom, E.C. Marques, J. Wong, C.L. Spiro, G.P. Huffman and F.E. Huggins, *Nucl Instrum. Methods Phys. Res*. **226** (1984) 542.

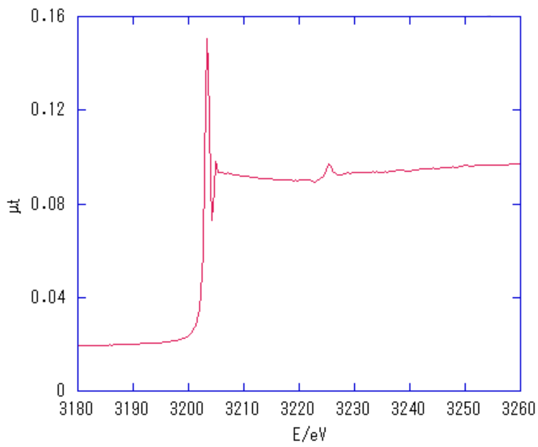

Figure 1
XANES spectrum of Ar in air.

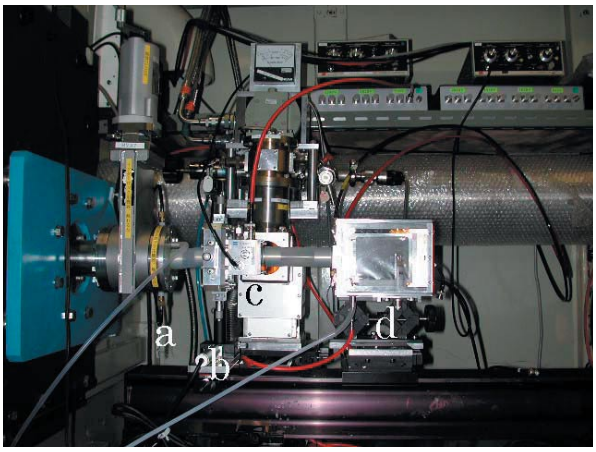

Figure 2
Photograph of the setup to soft X-ray XAFS experiments, a; beamline exit window, b; entrance slit, c; 10 monitor, d; sample chamber.

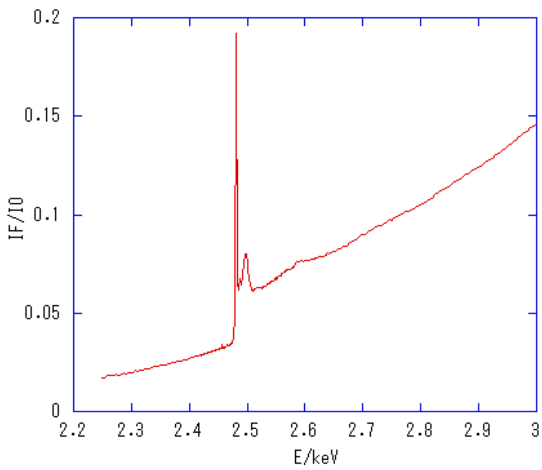

Figure 3
Sulfur K-XAFS spectrum of Scotch tape in FY mode.

4-3-2 BL-9C, A Versatile X-ray Beamline

BL-9C was kindly donated to the Photon Factory by the NEC Corporation in September 2000. The beamline had been owned by NEC since 1985, and was reconstructed in 1997. The beamline was designed for X-ray diffraction, XAFS, high-precision X-ray diffraction and topography experiments, and consists of a double-crystal monochromator and a bent cylindrical focusing mirror, as shown in Fig. 4. The mirror is placed at 16.1 m and the focus is expected at 30 m from the source point, thus satisfying a nearly 1:1 focusing condition. The maximum usable energy is 23 keV, limited by the critical energy of the mirror, but higher energy X-rays can be used by removing the mirror. The lower energy is limited by window absorption acceptable higher order contents for experiments. White X-rays can also be used by removing the first crystal of the monochromator.

Evaluation and commissioning of the beam-line began in October 2000. At this time, the high demand for small angle X-ray scattering (SAXS) became problematic and it was decided to provide some beamtime for SAXS studies at BL-9C. However, the beamline terminates with a 0.2 mm thick Be window followed by a Kapton window. Scattering from the Be window is not desirable for SAXS experiments, so the Be window at 28.1 m was moved to 20.2 m, and a slit system was installed at 24 m.

It was expected that several research areas would move to this beamline. One of these areas is time-resolved XAFS using dispersive optics, which requires white X-rays. Other areas included X-ray diffraction experiments using a six-circle diffractometer, and small- and medium-angle X-ray scattering experiments. Both of these activities require focused monochromatic X-rays. Among these fields however time-resolved XAFS has moved to AR-NW2A, where standard XAFS experiments are now carried out.

4-3-3 AR-NE1A2

We have been developing a two-dimensional imaging system for intravenous coronary angiography using synchrotron radiation monochromatic X-rays at AR-NE1A2 (see Section 5.9). After improvement of the imaging system and rearranging one of the conventional experimental hutches, AR-NE1A2, to become a dedicated hutch for clinical examinations, system II has been employed for clinical examinations since 2000 under collaboration between the University of Tsukuba and the Institute of Materials Structure Science. The following issues were improved to facilitate the more practical application of diagnostic imaging.

Beam size of monochromatic X-ray

The horizontal beam size had previously been limited to 73 mm at the patient's position due to the horizontal aperture of AR-NE1A, which was originally designed for Compton-scattering experiments. The horizontal size of the synchrotron radiation beam was enlarged from 73 mm to 93 mm by rearranging the beamline to more easily find the optimal patient position and to diagnose the movement of the heart in greater detail in 2003. Figure 5(a) and (b) show monochromatic X-ray images obtained before and after the rearrangement. The right coronary artery and the left ventricle are clearly seen in both images, however, a larger part of the aorta and left ventricle can be distinguished in Fig. 5(b).

Two-dimensional detector

We have been using an image intensifier–TV (II-TV) system as a two-dimensional detector. However, its dynamic range is limited (10^2) and this causes degradation of the vascular diacrisis in the mediastinal region where the coronary arteries overlap other organs, such as the aorta and the left ventricle. We introduced a flat-panel detector (FPD) in 2005 which has a larger dynamic range (10^3) than the II-TV system.

These improvements, along with the high current and the long lifetime of the electron beam obtained by efforts of the accelerator group have made more practical examinations possible.

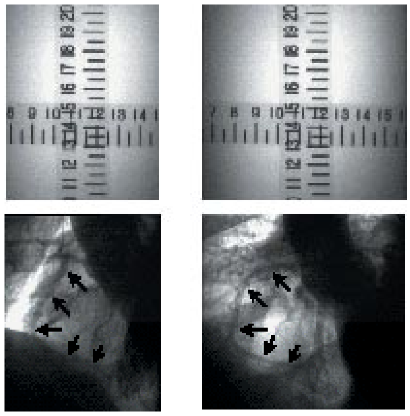

Figure 5
Images of the right coronary artery (arrows). (a) and (b) correspond to before and after expanding the horizontal size of synchrotron radiation beam, respectively.

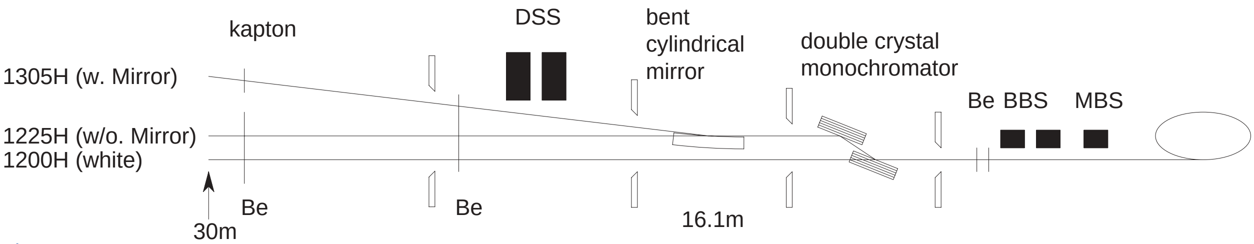

Figure 4
Schematic illustration of BL-9C. Three modes of the X-ray beam can be exchanged by inserting a monochromator and a mirror; i.e., white X-rays, focused monochromatic X-rays (< 23 keV) and high-energy monochromatic X-rays.