BL37XU Trace Element Analysis

BL37XU is a hard X-ray undulator beamline that is mainly used for studies of X-ray micro-spectrochemical analysis such as XRF imaging, XAFS, TXRF and XRF holography. This beamline has two branches of the standard undulator-beamline optics branch (branch A) and newly designed high-energy branch (branch B). For standard branch (branch A), both experimental hutches (EH) 1 and 2 can be used.

Area of research

X-ray microbeam spectrochemical analysis
Ultra trace element analysis
High energy X-ray fluorescence analysis

Keywords

Scientific field

Material science, Biology, Archaeology, Forensic science, Environmental science, Geochemistry

Equipment

X-ray microfocusing elements (Kirpatrick and Baez mirror, sputtered-sliced Fresnel zone plate), High spatial resolution X-ray microprobe, Multipurpose X-ray diffractometer, General X-ray fluorescence analyzer, High-energy X-ray fluorescence spectrometer, Grazing incidence spectro-reflectometer, Low-vacuum SEM

Source and optics

The light source of BL37XU is an in-vacuum type undulator, whose period length is 32 mm and the number of period is 140. The energy range of 4.5 ~ 18.8 keV is covered by the fundamental radiation from the light source, tuning its gap from 8 to 50 mm.

Fig.1 shows a schematic view of the beamline. A front-end in the storage ring tunnel and a transport channel in the optics hutch are composed of the standard components. A feature of this beamline is to consist of two branches: one is a SPring-8 standard undulator-beamline optics branch (Branch A) and the other is a high-energy branch (Branch B). Details of these branches are shown in the following.

Branch A : standard undulator-beamline optics branch

White undulator radiation is further monochromatized using a SPring-8 standard double-crystal monochromator located at 43 m from the source. X-ray energy range is tunable from 4.5 to 37.7 keV by using Si 111 reflection. The rotated-inclined geometry is used to manage high heat-load from the undulator radiation. The pin-post crystal is used as the first crystal and cooled by water directly, while the second crystal is cooled indirectly. The flux density of the monochromatic beam measured at 52 m from the source is more than 10^{13} photons/s for 8 ~ 30 keV. Two horizontal deflecting mirrors are placed downstream of the monochromator in order to eliminate higher harmonics and to obtain focused X-ray beam. A standardized mirror support at SPring-8 is used . Since each mirror is coated with two stripes of Rh and Pt, measurements are carried out with a suitable coating-material avoiding the absorption edges of mirrors.

Branch B : high-energy branch

A single-bounce monochromator which deflects the beam horizontally is located upstream of the double crystal monochromator, 37 m from the source. Currently, a Si (111) crystal is mounted on a water-cooled crystal holder with In sheets in order to achieve good thermal contact. The X-ray energy in the B branch is 75.5 keV, because the Bragg angle is fixed to 1.5˚. The mechanical system of the monochromator is similar to that of the standard mirror support of SPring-8. The flux density with various undulator gap measured at 75.5 keV is shown in Fig. 2. It was measured with an ionization chamber and normalized for ring current of 100 mA with the front-end slit aperture of $1 \times 1\ \mathrm{mm}^2$. The flux density was estimated to be 2×10^{10} photons/s at 5th harmonic, 4×10^{11} photons/s at 8th harmonic, 7×10^{11} photons/s at 11th harmonic and 1×10^{12} photons/s at 15th harmonic radiation of the undulator, respectively.

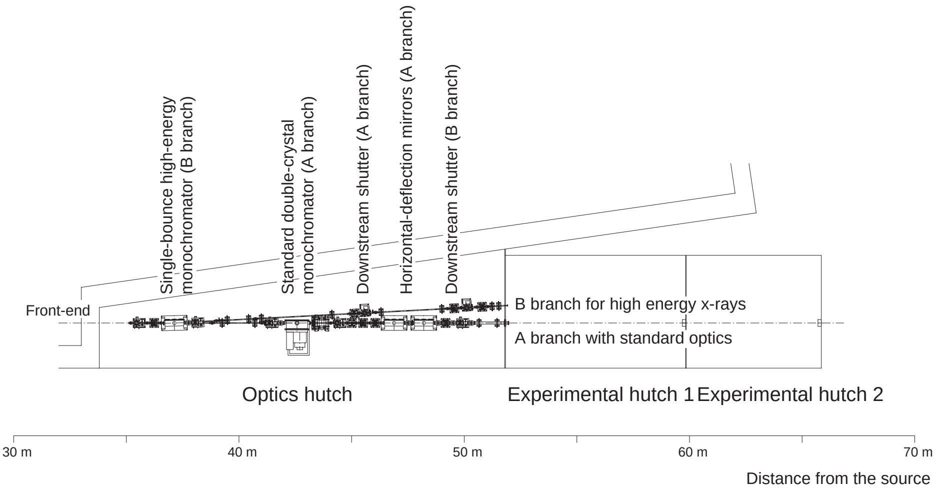

Fig.1. The schematic view of the beamline

X-rays beam parameters

Branch A

Energy range	5 ~ 37 keV
Resolution ΔE/E	2×10^{-4}
Flux at sample	10^{12} ~ 10^{13} photons/s
Beam size at sample	0.7 (V) × 2 (H) mm^2
Higher harmonic content	$< 1 \times 10^{-4}$

Branch B

Energy range	Si (111) : 75.5 keV
Resolution ΔE/E	2×10^{-4}
Flux at sample	10^{10} ~ 10^{12} photons/s
Beam size at sample	0.5 (V) × 3 (H) mm^2
Higher harmonic content	$< 1 \times 10^{-4}$

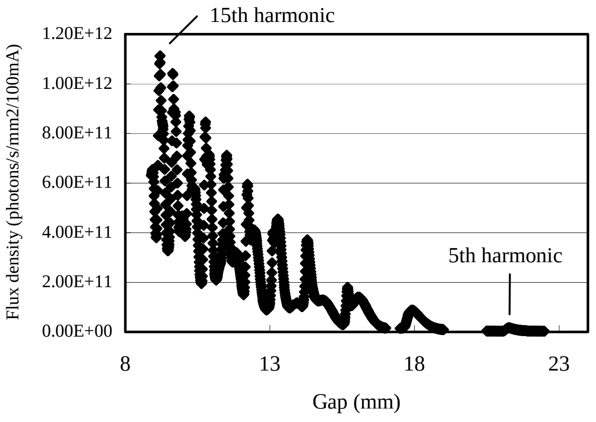

Fig.2. The estimated flux density with various undulator gaps at 75.5 keV

Experimental stations

The beamline has two tandem experimental hutches, which are located at 55 m and 62 m from the source. Experimental hutch 1 (EH1) has a size of 8 (L) × 5 (W) × 3.3 (H) m^3. Experimental hutch 2 (EH2) is connected to EH1 and has a size of 6 (L) × 4 (W) × 3.3 (H) m^3. A high spatial resolution X-ray microprobe [1] (Fig.3), a multipurpose X-ray diffractometer (Fig.4 left), a general X-ray fluorescence analyzer (Fig.4 right), and a high-energy X-ray fluorescence spectrometer (Fig.5) are installed in EH1. A grazing incidence spectro-reflectometer [2] (Fig.6) and a low-vacuum SEM (Fig.7) are equipped in the EH2. The outline of the X-ray microprobe and the high-energy XRF spectrometer are described as follows.

To realize energy tunable X-ray microbeam, Kirpatrick and Baez (K-B) mirror optics is adopted in X-ray microprobe system [1]. The beam size was 2 (H) × 4 (V) μm^2, and the flux was estimated to be more than 10^{10} photons/s at 10 keV. The focal length of the downstream mirror is 40 mm. A vacuum chamber to accommodate the K-B mirror and the scanning sample stage, are enables in-vacuum measurements. Recent experimental results show that the estimated minimum detection limits for Ni was 0.3 fg with the 9 keV X-ray microbeam. Detailed specification of the X-ray microprobe can be found in Ref. [3]. Trace characterization of individual atmospheric aerosol particles was carried out by the X-ray microprobe, and the data of elemental composition were utilized for investigating their origin and the physical and chemical processes during the transportation [4].

A high-energy XRF analysis system in the branch B composed of an XY stage, a pure-Ge solid-state detector, a spectroscopy amplifier, and a multi-channel analyzer. The X-ray beam size was adjusted by a combination of horizontal and vertical slits and was 0.2 × 0.2 mm^2. In order to examine the effectiveness of the high energy XRF in the analysis of rare-earth elements, we measured the XRF spectrum of a standard SRM612 glass sample. The nominal trace element concentration of this sample is 50 mg/kg (= 50 ppm) for each of the 61 elements that have been added to the glass support matrix. The XRF spectrum of the sample is shown in Fig.8. More than 20 heavy elements are clearly detectable, and the peak of each rare-earth element is clearly separated in the spectrum.

References

1. Hayakawa, S., Ikuta, N., Suzuki, M., Wakatsuki, M., Hirokawa, T., *J. Synchrotron Rad.* 8, 328-330 (2001).
2. Sakurai, K., Uehara, S., Goto, S., *J. Synchrotron Rad.* 5, 554-556 (1998).
3. Hayakawa, S., Tohno, S., Takagawa, K., Hamamoto, A., Nishida, Y., Suzuki, M., Sato, Y., Hirokawa, T., *Anal. Sci.* 17S, i115-i117 (2001).
4. Hayakawa, S., Tohno, S., Hamamoto, A., Suzuki, M., Hirokawa, T., *J. Phys. IV* 104, 309-312 (2003).

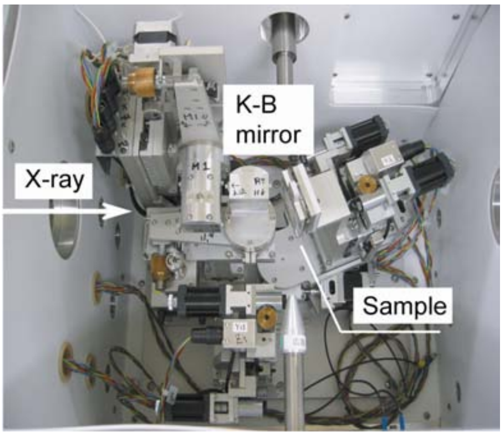

Fig.3. High spatial resolution X-ray microprobe, upper: outview, lower: inside of chamber

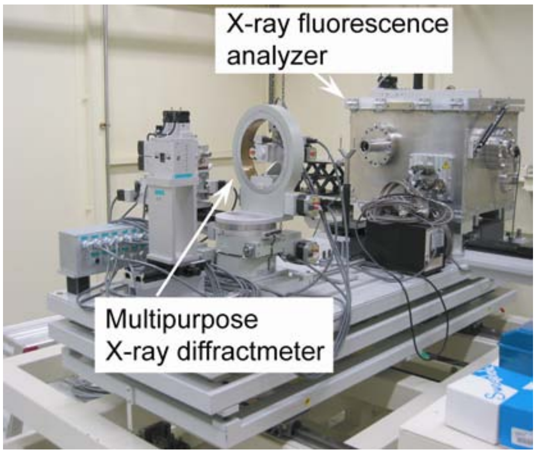

Fig.4. Multipurpose X-ray diffractometer (left) and general X-ray fluorescence analyzer (right)

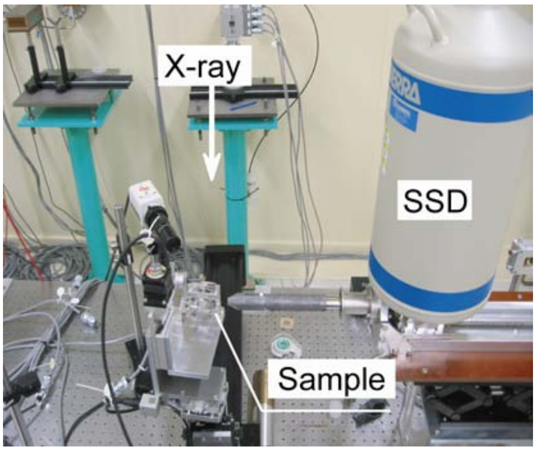

Fig.5. High-energy X-ray fluorescence spectrometer

Fig.6. Grazing incidence spectro-reflectometer

Fig.7. Low-vacuum SEM

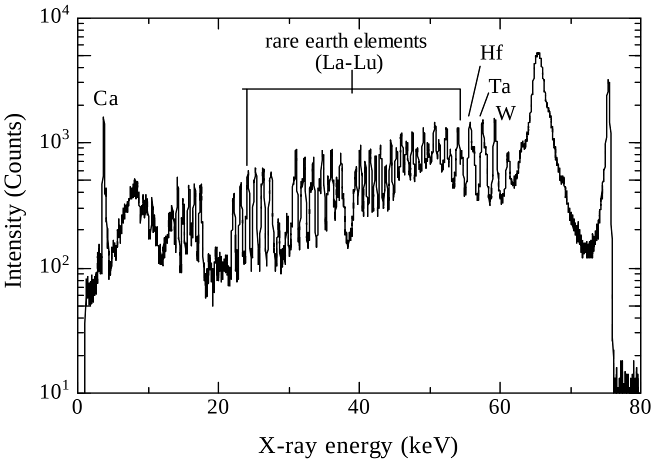

Fig.8. XRF spectrum of SRM 612 glass sample (counting time : 1000 s)

Contact information

Yasuko TERADA
SPring-8 / JASRI
1-1-1 Kouto, Mikazuki-cho, Sayo-gun, Hyogo 679-5198
Phone : +81-(0)791-58-0833
Fax : +81-(0)791-58-0830
e-mail : yterada@spring8.or.jp