

# BL-1

## Beamline for Extreme Ultraviolet Spectroscopy

Beamline 1 (BL1) was renewed as a beamline for extreme ultraviolet (EUV) spectroscopy from beamline for X-ray diffraction and scattering experiments. The beamline of the University of Tokyo in KEK-PF (BL-7B) was closed and donated to our center in 2009. Front-end part, including a toroidal pre-focusing mirror was designed and constructed and the whole system was installed on a base plate of 1m heights. This beamline is designed to accept the direct beam of  $10 \text{ mrad}^{\text{H}} \times 3 \text{ mrad}^{\text{V}}$  and to deflect it vertically into the 1 m Seya-Namioka type monochromator. An apparatus for the photoelectron spectroscopy system Phoibos 100 (SPECS ltd.) was installed. Also EUV irradiation equipment will be installed.

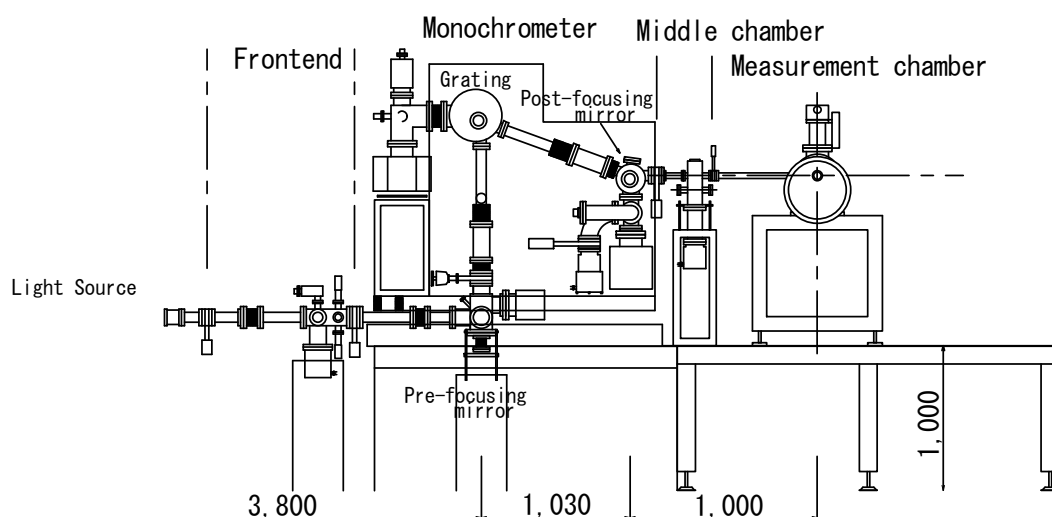


Figure 1 Outline of beamline 1

### Specification

Energy range: 5~50 eV

Energy Resolution:  $E/\Delta E > 1,000$  with 50  $\mu\text{m}$  slit width

Photon flux:  $1 \times 10^{11} \sim 1 \times 10^{12}$  photon/s / 200mA

Beam size:  $4 \text{ mm}^{\text{H}} \times 2 \text{ mm}^{\text{V}}$  at sample point

Mirror: Pre-focusing mirror, Au/Si, Post-focusing mirror, Pt/Cu

Monochromator: 1m grating Seya-Namioka monochromator

Three in-situ exchangeable grating

( $\lambda_b = 38, 96, 160 \text{ nm}$ ), 1200 line / mm

## BL-2

### Ultra Soft X-ray Absorption Spectroscopy

The ultra soft-XAS beamline at BL-2 was transferred from NTT Atsugi Laboratory in 2008, which consists of a conventional Monk-Gillieson-type monochromator, almost similar to that of BL-11[1]. A schematic view of the beamline is shown below.

4 varied-line-spacing plane gratings now provide strong ultra soft X-rays ( $10^{10-9}$  photons) from 35 eV to 1000 eV [2]. The beam size at the sample position is about  $0.6 \text{ mm}^{\text{H}} \times 1.5 \text{ mm}^{\text{V}}$ . An encoder system to read the rotation angle of each grating was developed for the precise photon energy calibration. Two analytical chambers are now in use: the general-purpose XAFS chamber which is open to all users and the XAFS-PES (photoelectron spectroscopy) chamber for NEDO-RISING project researching on lithium ion batteries. A high sensitive SDD with a large detection area ( $80 \text{ mm}^2$ ) was installed for the fluorescence X-ray yield XAFS measurements.

The transfer vessel system [3], compatible with BL-10 can be used for samples which do not want to be exposed to air.

[1] M. Koike et al., *Rev. Sci. Instrum.* **73**, 1541 (2002).

[2] H. Ishii et al., *Memories of SR Center* 13, XXX (2011).

[3] K. Nakanishi, S. Yagi and T. Ohta, *IEEJ Trans. EIS* **130**, 1762 (2010) (in Japanese).

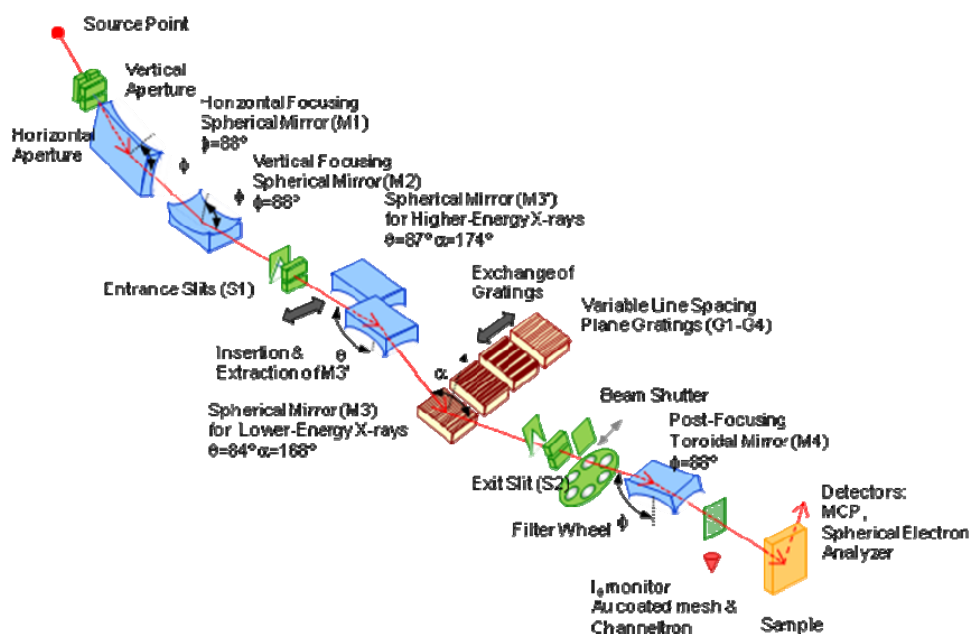


Figure Schematic view of the ultra soft X-ray absorption beamline BL-2.

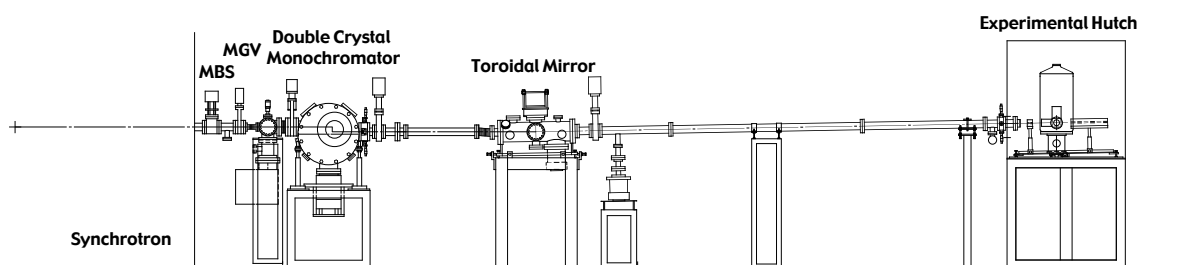
## BL-3

### X-Ray Absorption Fine Structure (XAFS) Spectroscopy

BL-3 is a beamline for XAFS measurements with the focused hard X-ray beam. It consists of a double crystal monochromator and a toroidal focusing mirror. The outline of BL-3 is depicted in Figure 1. White X-rays from the synchrotron are monochromatized and then are focused by the Pt coated toroidal mirror at the position of 4.5 m from the light source point. At the focus point in the experimental hatch, the X-ray beam size (FWHM) is 1 mm (V)  $\times$  2 mm (H). The monochromator is Golovchenko type which is the same as that of BL-4. The crystal size is 30 mm  $\times$  30 mm and is larger than that of BL-4. Character of the monochromator is described in the section of BL-4. The wider crystal and the focusing mirror supply the brighter X-ray beam than that of BL-4. The range of the X-ray energy is 3.4–9.1 keV with Si(220) and 2.1–7.6 keV with Si(111). The higher energy limit is due to the cut-off energy of the focusing mirror.

The operating system for the XAFS measurement was renewed in 2010. The step-scanning XAFS measurement is performed by the system modified using in the XAFS beamlines of Photon Factory of KEK. The user interface of the program and the output file format are common. Quick XAFS system is also installed to BL-3 and BL-4 in 2010 for the transmission mode. The monochromator is continuously moved during the measurement with QXAFS mode. The time needed to measure with QXAFS mode is shorter than that with the step-scanning mode.

In addition to the ionization chambers for the transmission mode, the three-elements Ge solid state detector (SSD) is available for the fluorescence mode in BL-3. The fluorescence mode is effective for dilute solutions or thin film samples. Although the concentration limit depends on the sample condition, the XAFS spectra for the solution of 100 ppm 3d metal species can be measured. The X-ray fluorescence analysis is also available with the 3-elements SSD.



**Figure 1** Layout of BL-3.

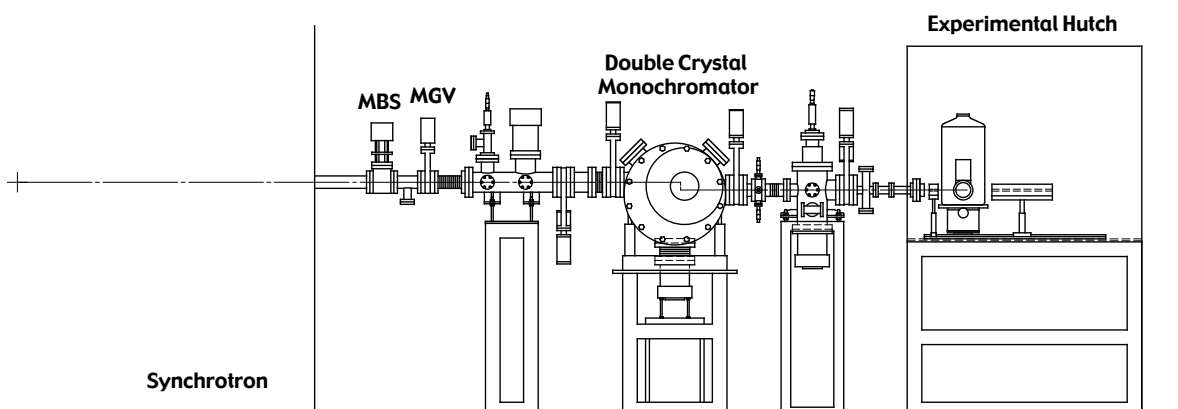
## BL-4

### X-Ray Absorption Fine Structure (XAFS) Spectroscopy

BL-4 is a beamline for XAFS measurements with the unfocused hard X-ray beam. The outline of BL-4 is depicted in Figure 1. White X-rays from the synchrotron are monochromatized by a double crystal monochromator of Golovchenko type. In this type of monochromator the both crystals move along the mechanical guide when a main axis is rotated, and the position of exit beam is independent from the monochromatized X-ray energy. The accessible angle range of the monochromator is from  $15^\circ$  to  $75^\circ$ . The available X-ray energy range is 1.8–3.5 keV with InSb(111), 3.5–11 keV with Ge(220), and 3.3–11 keV with Si(220). The covered energy range contains K-edge of the 3d transition metals and L-edge of the lanthanides. The dimension of the crystal is 20 mm  $\times$  20 mm, and the X-ray beam size is normally 12 mm(H)  $\times$  1 mm(V) in the experimental hutch and is variable.

The 4.5 cm and 31 cm ionization chambers are available as the detectors for the measurement of the incident and the transmitted X-ray intensity. For the effective detection, He(75%) + N<sub>2</sub>(25%), N<sub>2</sub>(100%), N<sub>2</sub>(85%) + Ar(15%), N<sub>2</sub>(50%) + Ar(50%), and Ar(100%) gases can be selected to flow the detectors. The measurement of the fluorescence mode with a Ge solid state detector or a scintillation counter and the measurement of the total electron yield mode in the vacuum chamber can be performed in BL-4.

The operating system for the XAFS measurement was renewed in 2010. The step-scanning XAFS measurement is performed by the system modified using in the XAFS beamlines of Photon Factory of KEK. The user interface of the program and the output file format are common. Quick XAFS system is also installed to BL-3 and BL-4 in 2010 for the transmission mode. The monochromator is continuously moved during the measurement with QXAFS mode. The time needed to measure with QXAFS mode is shorter than that with the step-scanning mode.



**Figure 1** Layout of BL-4.

## BL-6

### Exposure Beamline for LIGA Process

This Beamline BL-6 is set up for exposure of deep X-ray lithography in the LIGA (German acronym for Lithographir, Galvanoformung, and Abformung) process.

Fig.1 shows a drawing of the beamline BL-6. The whole length of the beamline is 1.43m. A 200  $\mu\text{m}$ -thick beryllium (Be) window which has a square of 5mm  $\times$  30mm is used to separate the ultra-high vacuum part and to obtain hard X-rays which have the photon energy of more than 1.7 keV. A 4-inch sample substrate can be set onto a chuck, while an X-ray mask is set into a mask chuck. The mask and sample substrate are mounted onto the X-Y stage in the exposure chamber. The chamber is then purged by flowing helium gas. The X-Y stage can move within a range of  $\pm 50\text{mm}$  in both directions for a large area exposure. Wafer bond alignment system is available for the multi-layer LIGA process.

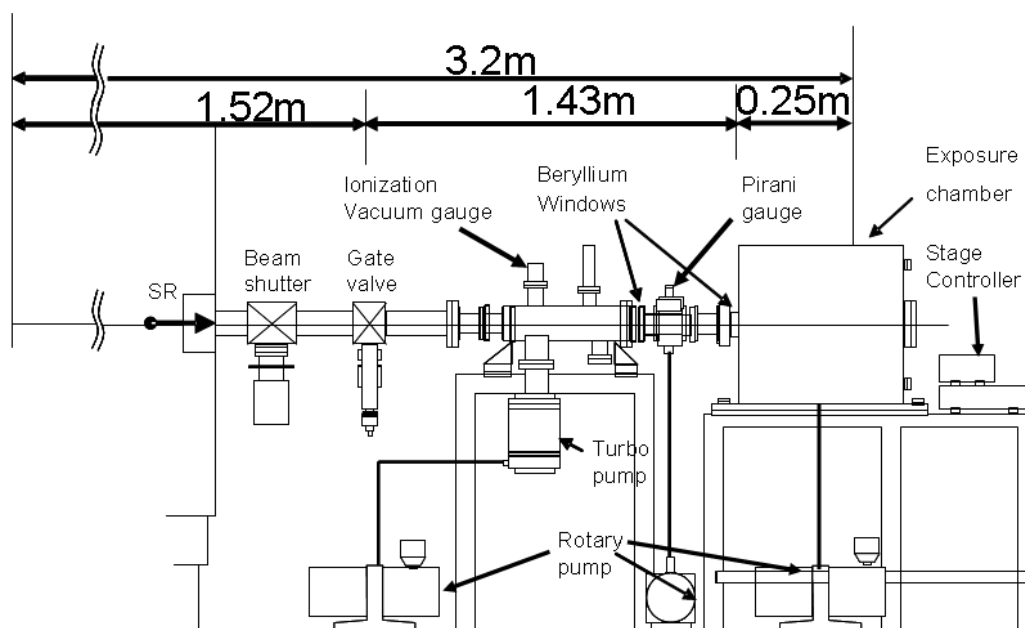


Fig.1 Schematic drawing of the BL-6

#### Specification

Energy range: 2-10 keV

Beam size: 5(H)  $\times$  30(V)  $\text{mm}^2$

Max sample size: 100 $\times$ 100  $\text{mm}^2$

Mask Holder: 126 $\times$ 126  $\text{mm}^2$

Mask-sample gap: 100-2000 $\mu\text{m}$  (adjustable)

Chamber pressure: 1 atm (He/Air $>$ 0.99)

## BL-7

### Linearly-Polarized Two-Dimensional Photoelectron Spectroscopy

VUV, soft X-ray beamline BL-7 is dedicated for solid and surface science. The range of photon energy covered by a monochromator with four spherical gratings is 10-160 eV. Two-dimensional display-type analyzer (DIANA) is installed at the end station. A schematic diagram of the beamline is shown in Fig. 1. The uniqueness of this beamline is a three-dimensional band measurement in a wide reciprocal space ( $\pm 2.3 \text{ \AA}^{-1}$  at kinetic energy of 36 eV), which contains new information on the motion of valence electrons (velocity, direction and mass). A linearly-polarized light enables the analysis of the atomic-orbital composition of each band.

A schematic cross section of DIANA used for the measurement of two-dimensional photoemission pattern is shown in Fig. 2. A linearly polarized synchrotron radiation (SR) is introduced through a hole in the obstacle rings. A spherical electric field applied between the obstacle rings and the main grid makes photoelectrons with a selected kinetic energy focus to the aperture. Photoelectrons are multiplied by micro-channel plates (mcp) and projected to a fluorescent screen with the emission angle preserved. Thus the momentum of the photoelectrons parallel to the surface can be deduced directly from the position of the photoemission pattern on the screen. The energy resolution of this analyzer  $\Delta E/E$  is 0.2 ~ 1% and the angular resolution is  $\pm 0.5^\circ$ .

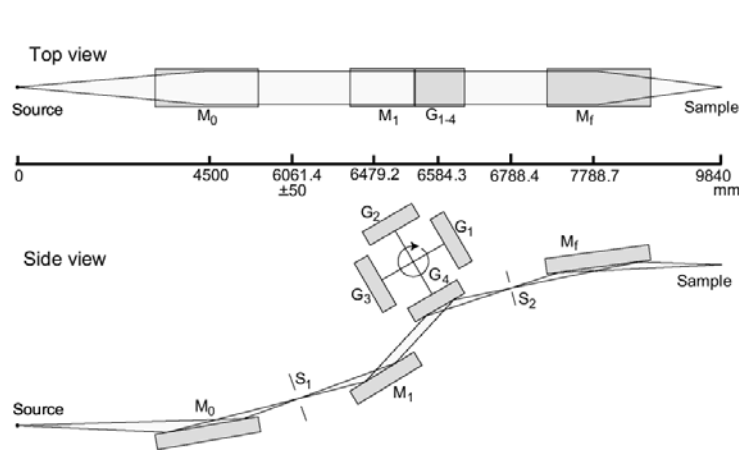


Fig.1. Optical arrangement of the new BL-7. M<sub>0</sub>, M<sub>1</sub>, M<sub>f</sub>: mirrors; S<sub>1</sub>, S<sub>2</sub>: entrance and exit slits; G<sub>1-4</sub>: spherical gratings.

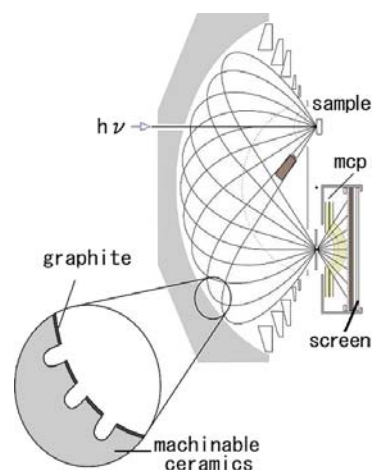


Fig. 2. Schematic diagram of DIANA with an electron gun. Incidence angle of electron beam is 45° off from the sample normal direction.

## BL-8 (SORIS-PES)

### Varied-Line-Spacing Plane Grating Monochromator for UV and SX Spectroscopy for Solid Surfaces

This beamline is constructed for studies of solid surfaces by photoelectron spectroscopy, x-ray absorption fine structure and photochemical reactions in the photon energy from 10 to 700 eV. At the end of the beamline, the apparatus of medium energy ion scattering (MEIS) is connected in situ. The electronic states and the atomic structures on surfaces and interfaces can be studied by this experimental system named as SORIS (Synchrotron Orbital Radiation and Ion Scattering). The figure shows the layout of the monochromator. The first cylindrical mirror M0 made of a Si single crystal focuses SR horizontally. A couple of plane mirrors (M0 and M1) and a varied-line-spacing plane grating (VSPG) G disperses and focuses SR on the exit slit. Either 400 l/mm VSPG or 1800 l/mm one can be chosen. Monochromatic light is focused on the sample position by a toroidal mirror M2. The beam size on the sample is about 3 and 1 mm in horizontal and vertical, respectively. The calculated photon energy resolution  $\Delta E/E$  is 5500 at 10 eV, and 1500 at 700 eV. The photon flux at 100 eV was estimated to be  $6 \times 10^{11}$  photons/s/300 mA. A UHV chamber equipped with a high energy resolution photoelectron spectrometer (Special version of PHI model 10-360 Omni Focus III) is installed at the experimental station.

#### Specification

Photon energy	: 5-700 eV
Resolution	: $\Delta E/E=5000-1500$
Photon flux	: $10^{10}-10^{11}$ photons/s/300 mA
Beam size	: SR = 10.5(H)-3(V) mrad <sup>2</sup> Target = 2(H)-1(V) mm <sup>2</sup>
Mirror	: M0 = cylindrical, Si, M1= plane, quartz M2 = toroidal, quartz
Grating	: Varied-line-spacing plane grating (Hitachi, mechanically ruled, 400 and 1800 l/mm)
Exit beam	: In horizontal plane at 1238 mm high from the floor
Special notation	: A photoelectron spectrometer and others are usually connected to this beamline

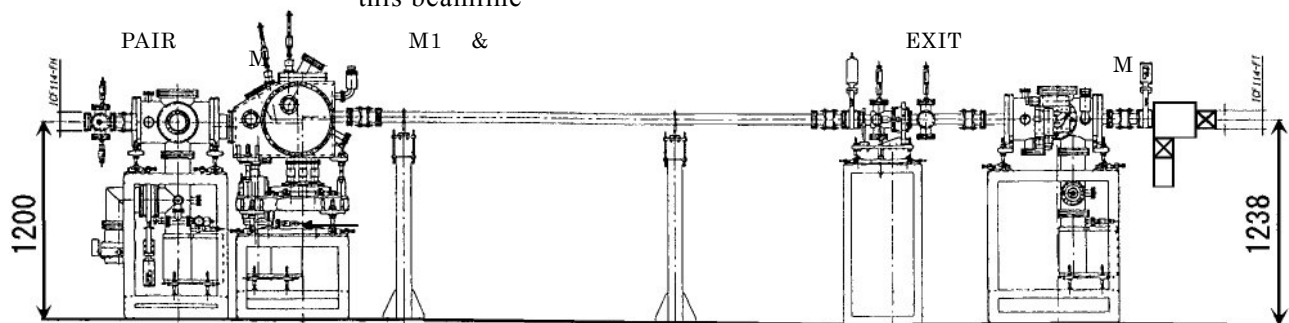


Fig. Outline of BL-8 (SR section only)

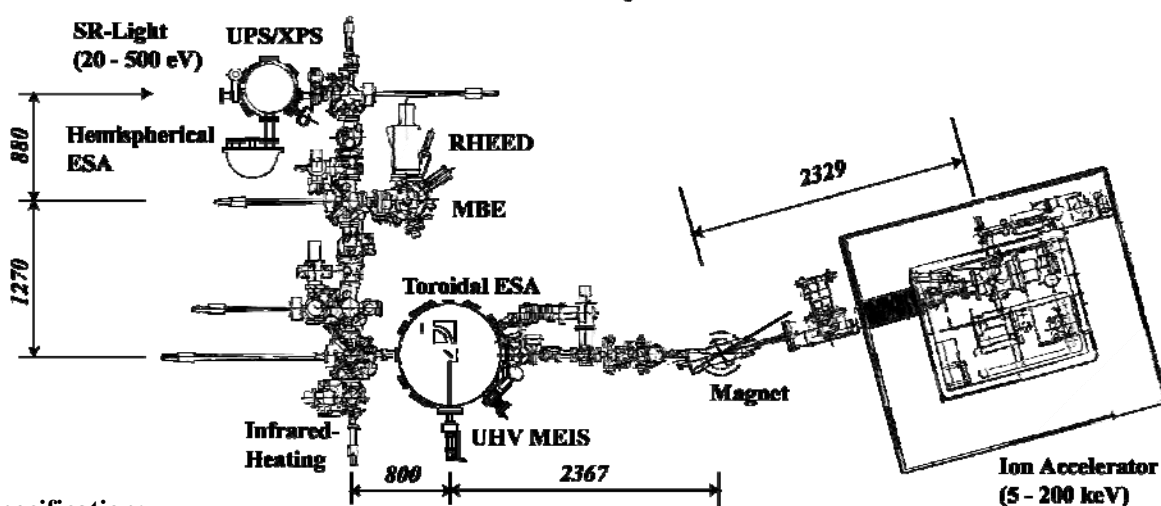
## BL-8 (SORIS-MEIS)

### Medium Energy Ion Scattering Spectroscopy with a High-resolution Toroidal Analyzer for Structural Analysis of Surfaces and Interfaces

The beamline 8 named SORIS consists of mainly two modules, (1) SR-photoelectron spectroscopy and (2) medium energy ion scattering spectrometry (MEIS). The former provides the information about the electronic states of surfaces and interfaces and the latter makes it possible to analyze the atomic configurations. In addition, it is equipped with sample preparation chambers for molecular beam epitaxy (MBE) and for sample heating from RT up to 1200°C under ultrahigh vacuum ( $2 \times 10^{-10}$  Torr) and various gas ambiance (O<sub>2</sub> etc). So, the analysis is basically performed *in situ*.

The MEIS system comprises an ion accelerator (5 – 200 keV), a switching magnet and an ion scattering chamber. A duoplasma ion source of a hollow cathode type generates dense plasma of H, He and Ne and provides the intense ion beams with good emittance. The accelerated ion beam is collimated finally to 0.18(horizontal)  $\times$  2.0(vertical) mm<sup>2</sup> and the angular spread is  $\pm 0.2^\circ$ (vertical). Scattered ion energies are analyzed by a new toroidal electrostatic analyzer with an excellent energy resolution of  $\Delta E/E = 9 \times 10^{-4}$ . It has a wide interelectrode distance of 16 mm and a radius of central curvature is 150 mm and thus accepts scattered ions in a wide energy range of 10 % of the pass energy at a fixed applied voltage, giving a good statistics in a short acquisition time.

### SORIS System



#### Specification:

Vacuum:  $\leq 2 \times 10^{-10}$  Torr (Base)

Ion Beam: 5 – 200 keV He<sup>+</sup>, H<sup>+</sup> and Ne<sup>+</sup>, Beam Size: 0.18(horizontal)  $\times$  2.0(vertical) mm<sup>2</sup>

Toroidal Electrostatic Analyzer:  $\Delta E/E = 9 \times 10^{-4}$ , Sample preparation Chamber: 3 K-cells + RHEED

Sample Heating with Infrared Radiation under UHV from RT up to 1200°C



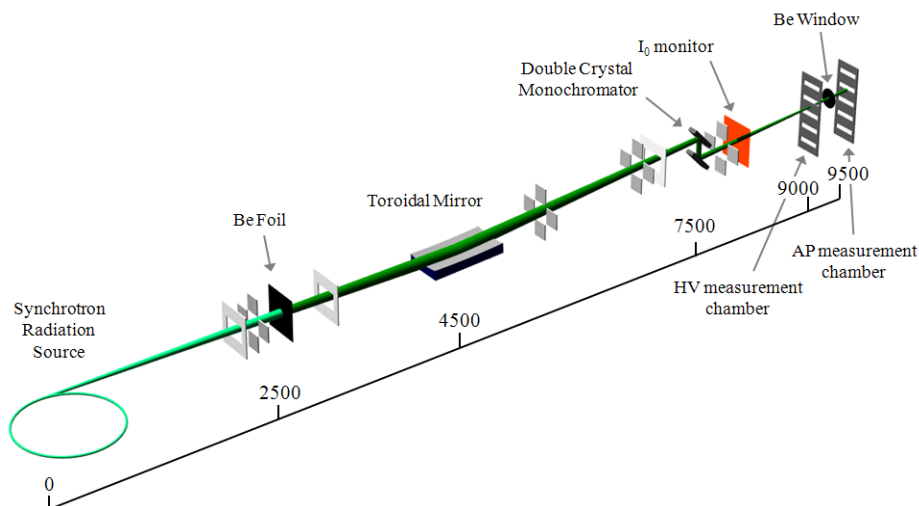
# BL-10

## Soft X-Ray XAFS Beamline

BL-10 is designed for study of X-ray absorption fine structure (XAFS) spectroscopy, using soft X-rays, the most brilliant energy range of the spectrum emitted from the light source, AURORA. It consists of a pre-focusing toroidal mirror, a Golovchenko-type double-crystal monochromator, an  $I_0$  monitor made of copper mesh, a high-vacuum (HV) sample chamber and atmospheric-pressure (AP) sample chamber. The radiation beam with 6 mrad (horizontal) and 2 mrad (vertical) is deflected upward by  $1.4^\circ$  and focused at the sample position about 9 m apart from the source point with the 1:1 geometry. The available photon energy covers from about 1000 to 4500 eV (K-edge of Na ~ Ca and L-edge of Zn ~ Sn) by exchanging several monochromatizing crystals. The detection sensitivity limit was improved dramatically by newly installing a silicon drift detector (SDD, TXD2300H50, Techno X Co.,Ltd.) with a wide detection area of  $50 \text{ mm}^2$  which is an order of magnitude larger than the previous one).

### Specification

Photon Energy	about 1000 ~4500 eV
Optics	Be foil (5.1 $\mu\text{m}$ thickness), Ni toroidal mirror (water cooled), Be window (15 $\mu\text{m}$ thickness) Double crystal monochromator Beryl(10-10), KTP(011), quartz(10-10), InSb(111), Ge(111), Si(111), Si(220).
Beam size	<HV> 5 mm (horizontal) $\times$ 2 mm (vertical) : focused <AP> 8 mm (horizontal) $\times$ 2.5 mm (vertical) : non-focused
Photon flux	about $10^8$ - $10^9$ photons/sec
Detecting mode	<HV> TEY mode (sample drain current), PEY mode (developed MCP detector) PFY mode (SDD). <AP> PFY mode (SDD).
Other	A transfer vessel system (common to BL-2) for anaerobic or easily-oxidizable samples.



## BL-12

### Soft X-Ray Microscopy

This beamline has been operating since 1995. Advantages of this X-ray microscope station are as follows: (1) a specimen is located under atmospheric pressure, (2) for object finding and pre-focusing, a light microscope can be used, (3) multi-wavelength imaging enables, and (4) an X-ray image can be recorded on a CCD camera system. In order to improve this X-ray microscope, a new project started at 2007. Full automatic and wide energy range observation system is under construction.

This beamline consists of a SiC plane mirror and an optical stage. The optical stage is composed of a condenser zone plate (CZP), a pinhole (15 $\mu\text{m}\phi$ ), a specimen stage, an objective zone plate (OZP), and a cooled CCD camera. Images are focused with the first-order diffraction. Groove efficiency and absolute efficiency of the CZP are 7.5% and 3.6% at  $\lambda=2.5\text{nm}$ . The resolution is estimated to be below 71nm (20–80%) from the intensity gradient of its knife edge profile at 2.4nm.

For dry samples, almost any kind of holder can be mounted in the sample stage, and micro grids with carbon substrate are typically used. For wet samples, a special wet cell is prepared. It consists of two thin polyimide films (below 300nm in thickness) supported by thick ones. Wet samples are placed between the two thin polyimide films and the wet cell is sealed with silicon grease. In this term, cryogenic sample observation starts. Imaging time is typically 30 - 180s.

#### Specification

Energy range (new system): 0.26–0.71 keV (Wavelength : 1.73–4.73nm )

Optical element : SiC Plane mirror, CZP, OZP

Detector : Cooled CCD camera  
(C4880-21-24WD: Hamamatsu Photonics)  
512 pixel  $\times$  512 pixel, 24 $\mu\text{m}$   $\times$  24 $\mu\text{m}$  each

Spatial resolution : 71nm (20–80%) at  $\lambda=2.4\text{nm}$

Scientific applications : Biology, Medical, Polymers, Material sciences,  
Environmental sciences, Powder.

Table1 Specification of the CZP and OZP.

	CZP	OZP 1	OZP 2
Diameter ( $\mu\text{m}$ )	9,000	50	56
Number of the zones	41,890	277	311
Outermost zone width (nm)	53.7	45	45
Zone material	Ge (t=0.3 $\mu\text{m}$ )	Ni (t=0.13 $\mu\text{m}$ )	W (t=0.12 $\mu\text{m}$ )
Support material	SiN(t=0.1 $\mu\text{m}$ )	SiN (t=0.1 $\mu\text{m}$ )	SiN (t=0.1 $\mu\text{m}$ )

## BL-13

### Exposure Beamline for LIGA Process with High Flexibility

This beamline has been built in 2002. This equipment can be used for LIGA process. We can move X-ray mask and sample (PMMA) independently, whose stage is controlled by computer. Further, we can make many different two-dimensional and three-dimensional microstructures. Fig.1 shows a drawing of BL-13. The whole length of the beamline is 1.58m. A 200  $\mu\text{m}$ -thick beryllium (Be) window ( $5\times 30\text{ mm}^2$ ) is used to separate the ultra high vacuum part from the low vacuum part and we get hard X-ray photons higher than 1.3keV. The sample can be moved by 70 mm both in X and Z directions and rotated freely by computer. Besides, we can tilt the sample in any direction ( $\delta$ -axis). On the other hand, we can also move X-ray mask in X-direction by computer and in Z-direction manually (Fig.2). Experiments can be done either in vacuum or in He gas. This beamline enable us to make many different two-dimensional and three-dimensional microstructures by changing the mask shape, sample moving speed and tilting direction.

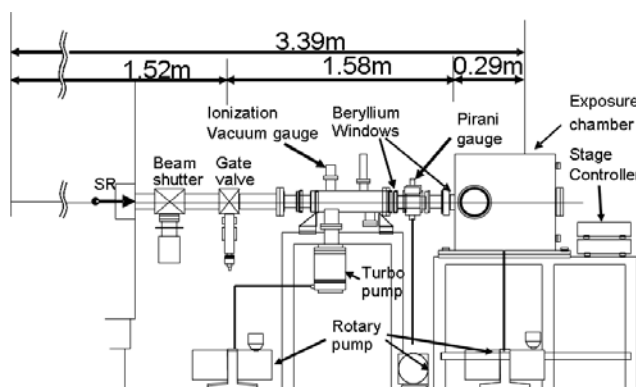


Fig.1 Schematic drawing of the BL-13

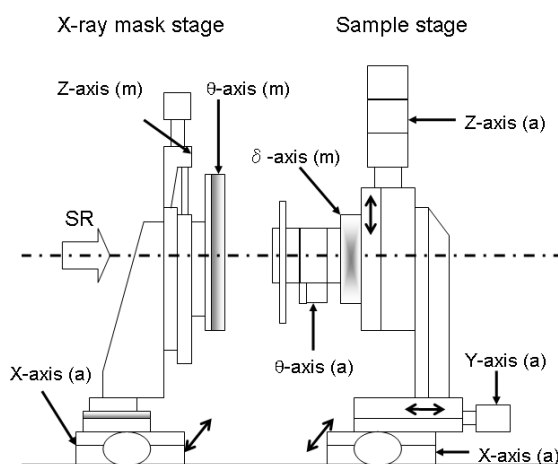


Fig.2 Sketch of the stage  
(a: automatic, m: manual)

### Specification

Energy range: 2-10 keV

Beam size: 5(H)  $\times$  38(V)  $\text{mm}^2$

Max sample size: 100 $\times$ 100  $\text{mm}^2$

Mask Holder: 126 $\times$ 126  $\text{mm}^2$

Mask-sample gap: 100-2000 $\mu\text{m}$  (adjustable)

Chamber pressure: 1 atm (He/Air $>$ 0.99)

## BL-14

### SR Micro Lithography and Etching for Micro/Nano Fabrication

This beam line was built as an object for SMILE (SR micro lithography and etching for micro/nano fabrication) in 2000. Unlike other beam lines for LIGA, this beam line is equipped with the heater stage for substrate heating, and the turbo pump in the chamber (Fig.1). The temperature of a substrate can be raised to about 240°C, and the degree of vacuum in a chamber can be raised to less than  $10^{-5}$  torr. Therefore, it is possible to perform not only the usual LIGA process but a TIEGA process. Moreover, the stage system with high flexibility is carried and it can respond also to three-dimensional processing (Fig.2). Beam size is  $30 \times 5 \text{ mm}^2$  and the maximum exposure area is  $80 \times 55 \text{ mm}^2$ .

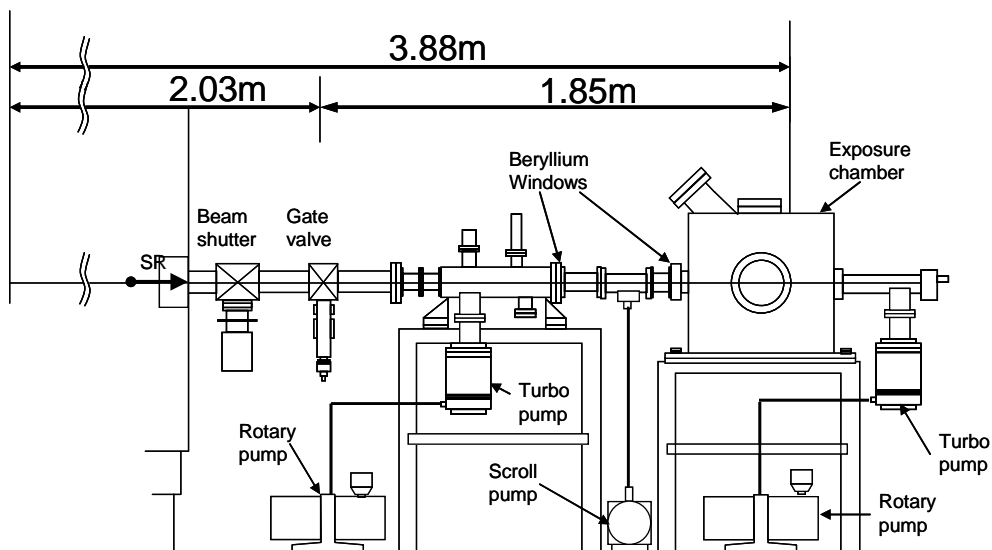


Fig.1 Outline of BL-14

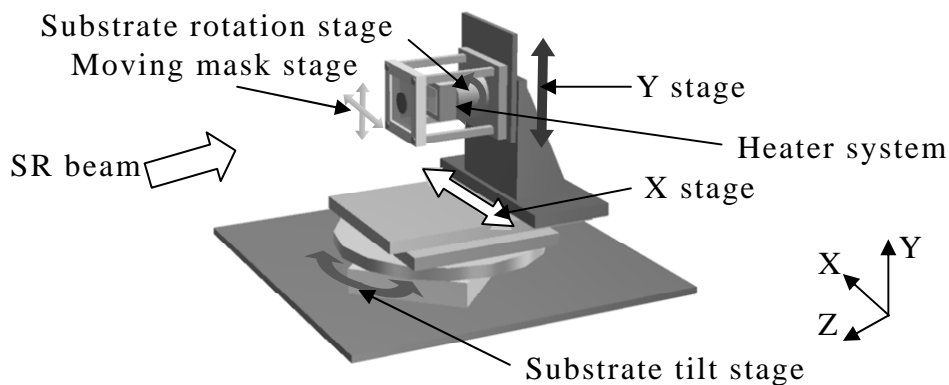


Fig.2 Stage system

## BL-15

### Infrared Microspectroscopy

Infrared microspectroscopy (IRMS) with an SR light source is designed for infrared (IR) spectroscopic studies of a microscopic region in various materials since SR light is a higher brilliance than laboratory sources in commercial IRMS. IR photons are collected by a toroidal mirror installed in the ring chamber, whose acceptance angles are 250 mrad (horizontal) and 63 mrad (vertical). The beam is once focused inside a ring port to travel through the narrow port. Another toroidal mirror outside the chamber focuses the beam again in the air part of the beamline via a window (NIR-MIR: BaF<sub>2</sub> or KRS-5, FIR: Diamond). The diverging beam is shaped into a parallel beam with a parabolic mirror, and then introduced into a commercial IRMS equipment. As a result, our IRMS with the SR light (SRMS) has a spatial resolution of ca. 5 $\mu$ m though that of a commercial IRMS is 10 $\mu$ m.

We can also measure using an ATR method with which the IRMS can have a much higher resolution.

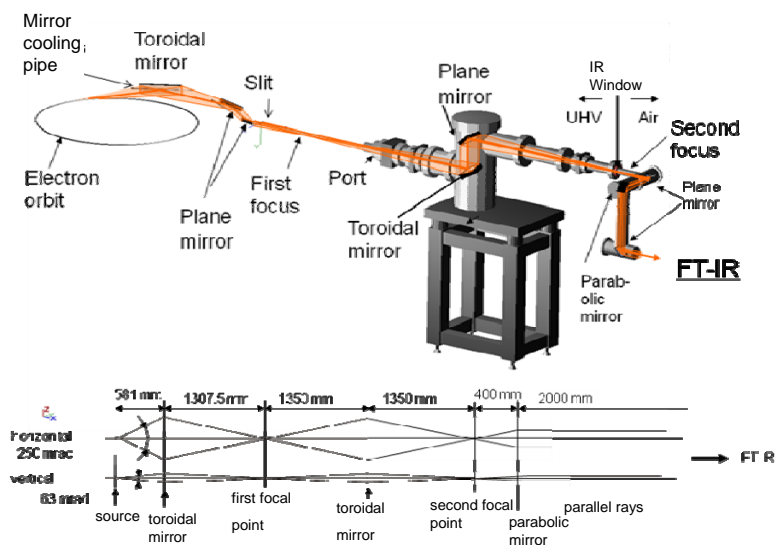


Fig. Schematic diagrams of the BL-15 and the optical system. IR window is BaF<sub>2</sub> (NIR to MIR) or Diamond (FIR).

**FTIR Spectrometer:** Nicolet 6700 (Thermo Fisher Scientific Inc.)

Beam splitter: XT-KBr (11000 - 350 cm<sup>-1</sup>) for NIR to MIR, Solid (700 - 20 cm<sup>-1</sup>) for FIR.

Energy Resolution (typ.): 4 cm<sup>-1</sup>.

Detector: DLaTGS/KBr (7800 cm<sup>-1</sup> - 350 cm<sup>-1</sup>), DLaTGS/PE (700 cm<sup>-1</sup> - 50 cm<sup>-1</sup>).

**FTIR Microscope:** Nicolet Continuum XL (Thermo Fisher Scientific Inc.)

Optics: 15x (N.A. 0.58), 32x (N.A. 0.65) Cassegrain objectives and condensers.

Detector: 50 $\mu$ mMCT (11000 - 650 cm<sup>-1</sup>), One-dimensional arrayed MCT (11000 - 800 cm<sup>-1</sup>).

ATR unit: Ge hemisphere.