BL-1

Beamline for X-Ray Diffraction and Scattering Experiments

This beamline is for X-ray diffraction and scattering experiments at wavelengths in the range from 0.25 to 0.15nm. The use of the radiation in the structural study of organic and/or inorganic crystals containing 3d metal atoms using phenomena of anomalous scattering is available. We have chosen simple beamline optics consisting of a pre-focusing toroidal mirror and a double-crystal monochromator with the 1:1 focusing geometry. With large curved aperture (460mm×256mm, 127.4mm radius) imaging plate as a detector, Weissenberg type camera provides rapid diffraction data collection. Partial-χ, 3-circle goniometer allows easy reorientation of crystal for complete coverage of reciprocal space. Data can be collected in standard oscillation mode or screenless Weissenberg mode.

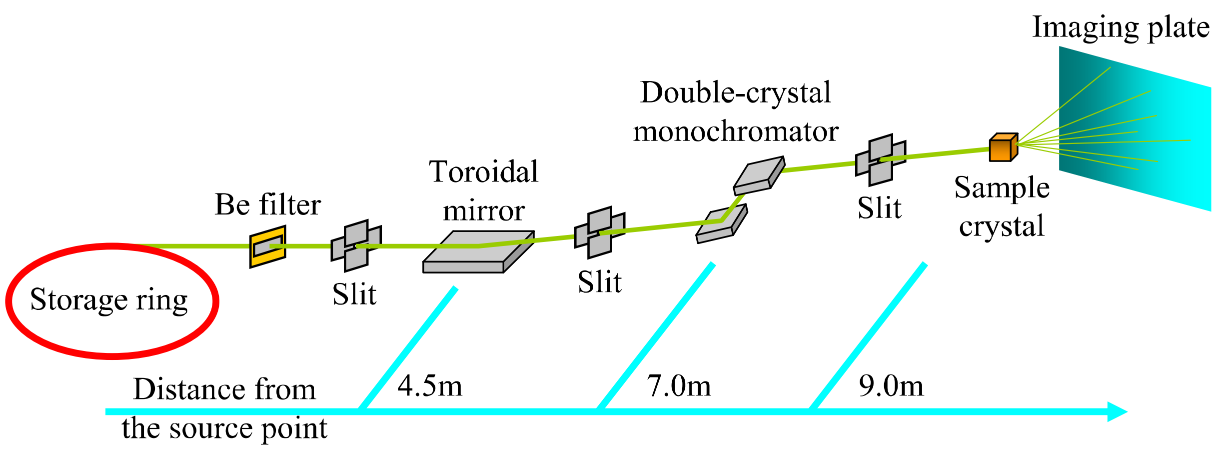


Fig. 1 Schematic view of the optical arrangement of the X-ray diffraction/scattering beamline.

Specification

Wavelength range	:0.25-0.15nm		
Resolution	:E/⊿E ～5000		
Pre-focusing toroidal mirror			
Substrate	:Silicon single crystal	Surface material	:Pt
Size	:L430mm×W70mm×T30mm	Grazing incidence angle	:0.55°
Double-crystal monochromator	:Ge(220) reflection		
Diffractometer	:Oscillation method / Weissenberg method		
X-ray detector	:Imaging plate (IP)		
2θ range	:-60～144°		

BL-2

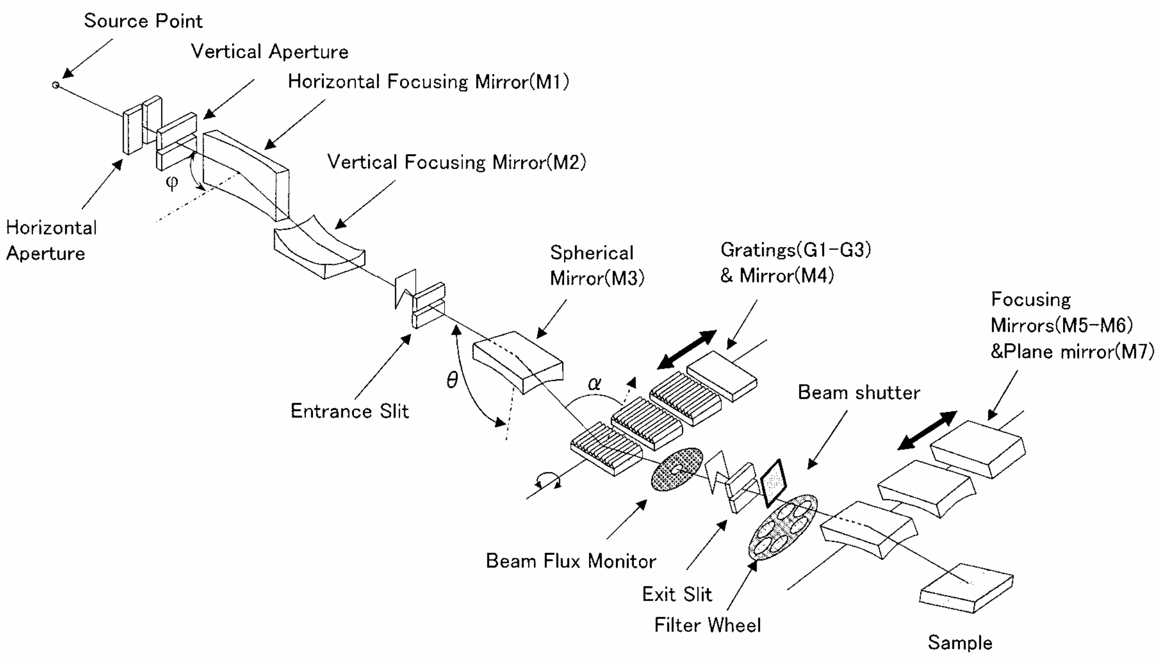
Ultra Soft X-ray (Vacuum Ultraviolet) Absorption Spectroscopy

The first BL-2 monochromator which has been active for almost ten years was closed December 2007. It was originally installed by Professor Kazuo Taniguchi of Osaka Electro-Communication University. It could provide photons covering from 50 to 600 eV. Its feature was unique so that the K-edge absorption data for light elements like lithium and boron in various compounds were well comparable with or of even higher quality than those reported from other facilities. Its specifications have been described in the previous copy of *Memories of the SR Center*, No.9 (2007), p.114.

We are now replacing the whole system with a new one. Its design is almost the same as the one installed at BL-11, cf. "Evaluation Beamline for Soft X-Ray Optical Elements" by K. Sano et al., *Memories of the SR Center*, No.9 (2007), p.67. The monochromator has three varied-line-spacing gratings: 300 l/mm for 20-120 eV, 600 l/mm for 80-120 eV, and 1200 l/mm for 140-600 eV. Expected resolution ($E/\Delta E$) is 1,000 ~ 3000.

The new beamline is under construction and will supply the light June, 2008.

Photons accepted : 5 mrad horizontally, 3 mrad vertically
Mirrors (M1) : 400 mmL x 50 mmW, spherical; r = 67694 mm, coated with 50 nm Au on Si
(M2) : 300 mmL x 50 mmW, spherical; r = 59098 mm, coated with 50 nm Au on SiO_2
(M3) : 150 mmL x 40 mmW, spherical; r = 20085 mm, coated with 50 nm Au on SiO_2
(M3' for the highest energy region) : 200 mmL x 40 mmW, spherical; r = 39643.8 mm, coated with 50 nm Au on SiO_2



BL-3

XAFS spectroscopy

This beamline is newly constructed by strong demand of users for XAFS spectroscopy. In principle, this beamline is similar to BL-4, but is aimed to provide higher photons. The beamline layout is shown in Fig. 1. Higher energy photons are filtered by two 100 μm thick Be windows and introduced into a Golovchenko-type double crystal monochromator. Si(220) and Si(111) are used as monochromatizing crystals and the photon energy from 3.4 to 11.0 keV is available, which is a little higher than that from BL-4. Beam size at the sample position is 25 mm^{H} and 2 mm^{V}, since no focusing optics is adopted. Conventional transmission mode is adopted with 5, 15 and 30 cm long ion chambers are used for the I_0 and I detectors.

It is also planned to insert a total reflection mirror at the sample position to focus the beam for XAFS spectroscopy of a small samples.

This beamline was funded by the nanotechnology supporting project of MEXT.

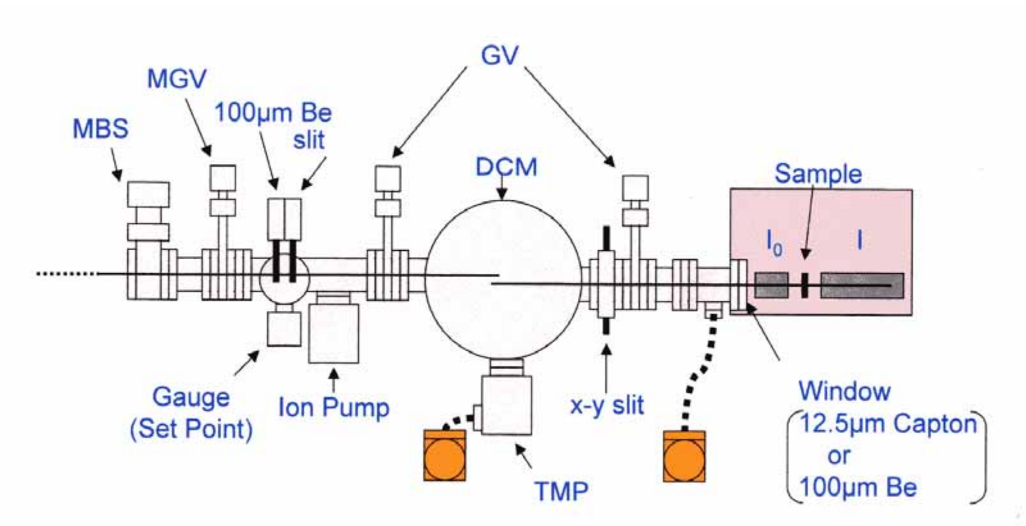


Fig.1 Beamline layout of the BL-3

BL–4

XAFS Spectroscopy

BL–4 consists of three windows, each being 50 μm in thickness, a slit, a double–crystal monochromator and two detectors. The outline of BL–4 is depicted in Fig. 1.

The double–crystal monochromator employed is of Golovchenko type. The mechanical movement of the monochromator is in a range from 15° to 75°. Thus, InSb(111) crystal covers the K–edge absorption energies of Si, P, S, Cl, Ar, K, Ca, Sc, Ti, and V. However, XAFS measurements for K, Ca, and Sc are difficult because L–edge absorptions of In and Sb appear in a region of 3.7 – 4.7 keV. The angular range corresponds to the X–ray energy range of 3.4 – 12.4 keV and 3.2 – 12.0 keV in the case of Si(220) and Ge(220), respectively. However, the compact SR source does not supply sufficient X–ray intensities above 10 keV. A practical use of Si(220) and Ge(220) crystals is thus limited to the elements K, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, and Zn. A quartz(1010) double–crystal was newly introduced for Al K-XAFS.

The incident monochromatic X–ray intensity I_0 and transmitted intensity I after passing through a sample were simultaneously measured by two ionization chambers (S–1329A and S–1196B, OKEN). He(100%), N_2(100%), N_2(85%) + Ar(15%), N_2(50%) + Ar(50%), and Ar(100%) gases are used for the detection. The output currents were amplified by a Keithley 428 current amplifier and changed to frequency by a 1 MHz V/F converter (NVF–02, Tsuji Denshi). The signal is then fed to an ORTEC 974 scaler and finally stored in an NEC PC–9801FA computer. For measurements around the K–edges of Co, Ni, Cu and Zn, where intensities of X–ray emitted from the SR source steeply decrease, a scintillation counter with 2 inches in diameter (SC–50, Rigaku) is used as an I detector. The scintillation counter is also employed for the fluorescent detection by using a suitable filter. An electron multiplier (R595, Hamamatsu Photonics) is used for total electron yield measurements.

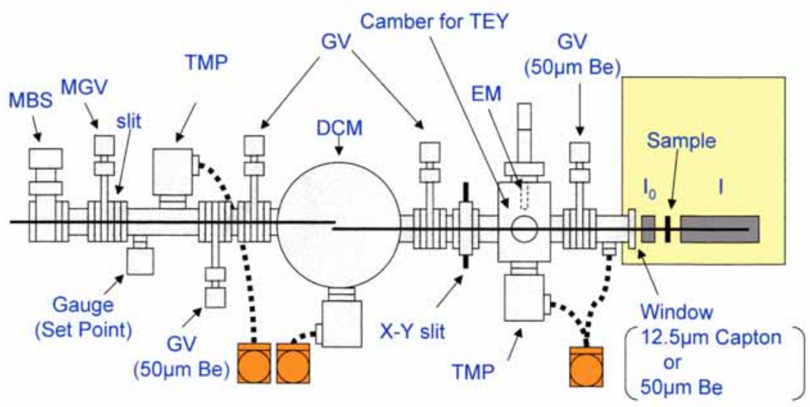


Fig.1 Layout of BL-4.

BL-5

Exposure Beamline for LIGA Process

The beamline BL-5 is set up for exposure of deep X-ray lithography in the LIGA (German acronym for Lithographie, Galvanoformung, and Abformung) process. Fig. 1 shows a schematic drawing of the beamline BL-5. The whole length of the beamline is 6.12 m. A 200 μm-thick beryllium (Be) window is used to separate the ultra-high vacuum part from the low vacuum part, and a 100 μm-thick Be window is used to separate the low vacuum part from the exposure chamber.

This beamline has three exposure chambers. The 1st and the 2nd chamber can be used for the multi-layer LIGA process. The 3rd chamber can be available for fabricate the submicron three-dimension structures using PCT (Plane-pattern to Cross-section Transfer) technique.

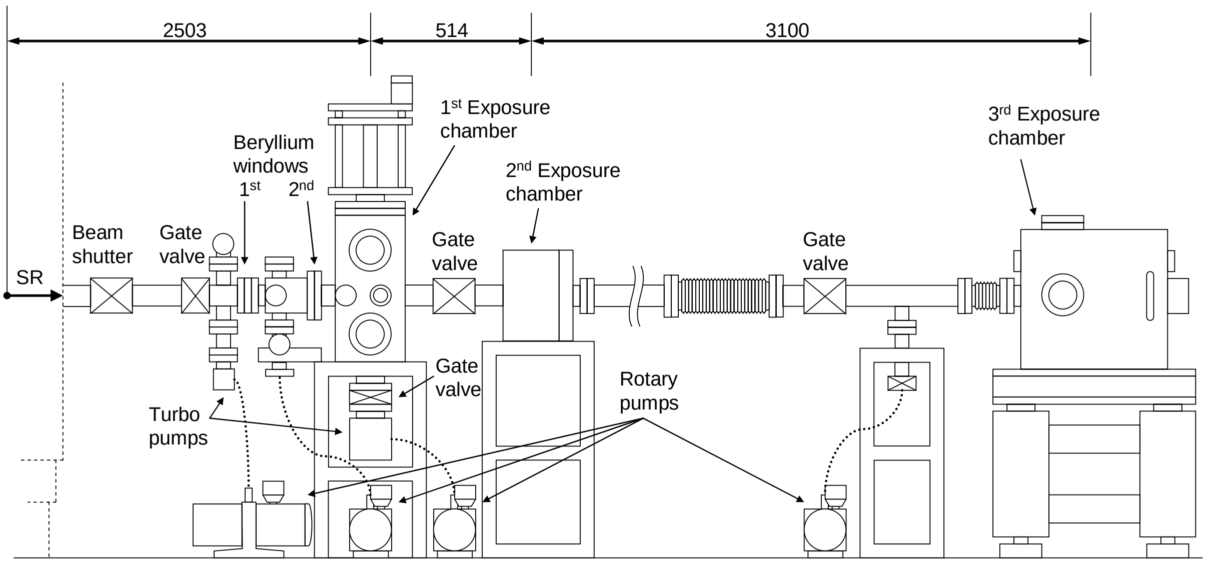


Fig. 1 Schematic drawing of the beamline BL-5

Specification

Energy range :2-10 keV

Chamber pressure :0.85 atm (He / Air ≥ 0.99)

	1st Chamber	2nd Chamber	3rd Chamber
Mask size	126 × 126 mm^2	φ 75 mm	4″ wafer size
Sample size	30 × 52 mm^2	φ 75 mm	4″ wafer size
Mask-Sample gap	100 – 2000 μm	100 – 2000 μm	Min. 50 μm
Sample scan area	Y: ±15 mm	Y: ±15	X: ±50 Y: ±25 mm

BL-6

Exposure beamline for LIGA Process

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

Figure 1 shows a drawing of the beamline BL-6. The whole length of the beamline is 1.43 m. A 200 μm-thick beryllium (Be) window which has a square aperture of 5 mm x 30 mm is used to separate the ultra-high vacuum part and to obtain hard X-rays which have the photon energy of more than 1.3 keV. A 50 μm thick Kapton window is used to separate the low vacuum part from the exposure chamber and to select harder 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 ±50 mm in both directions for a large area exposure. Wafer bound alignment system is available for the multi-layer LIGA process.

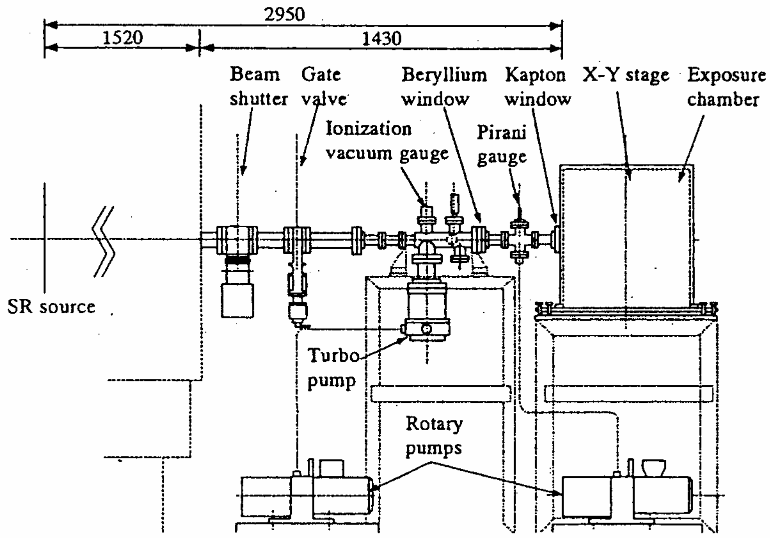


Fig.1 Schematic drawing of the BL-6

Specification

Energy range : 2 –10 keV Mask Holder : 126 x 126 mm^2

Beam size:5(H) x 30(V) mm^2 Mask-sample gap: 100 –2000 mm(adjustable)

Max sample size: 100 x 100 mm^2 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 (± 2.3 Å^{-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 $\pm 0.5°$.

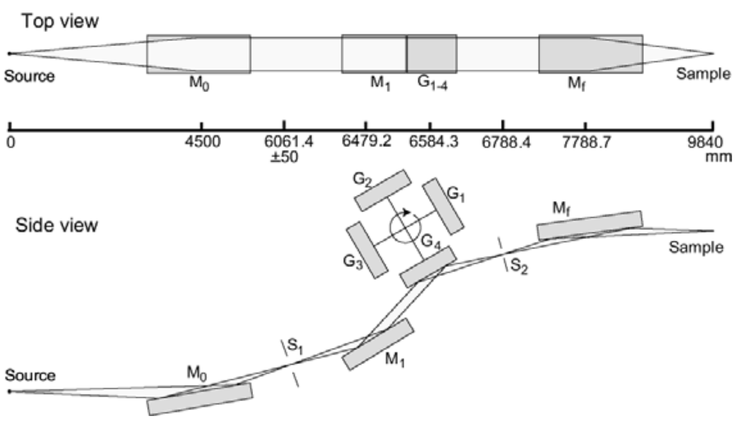


Fig.1. Optical arrangement of the new BL-7. M_0, M_1, M_f: mirrors; S_1, S_2: entrance and exit slits; G_{1-4}: spherical gratings.

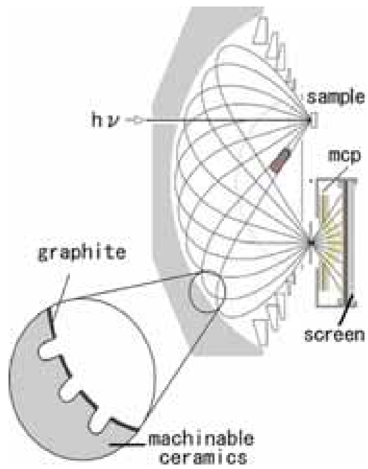


Fig. 2. Schematic diagram of DIANA with an electron gun. Incidence angle of electron beam is 45° off from the sample normal direction.

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 ΔE/E is 5500 at 10 eV, and 1500 at 700 eV. The photon flux at 100 eV was estimated to be 6×10^{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	: ΔE/E=5000−1500
Photon flux	: 10^{10}−10^{11} photons/s/300 mA
Beam size	: SR = 10.5(H)−3(V) mrad^2 Target = 2(H)−1(V) mm^2
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 bean	: In horizontal plane at 1238 mm high from the floor
Special notation	: A photoelectron spectrometer and others are usually connected to this beamline

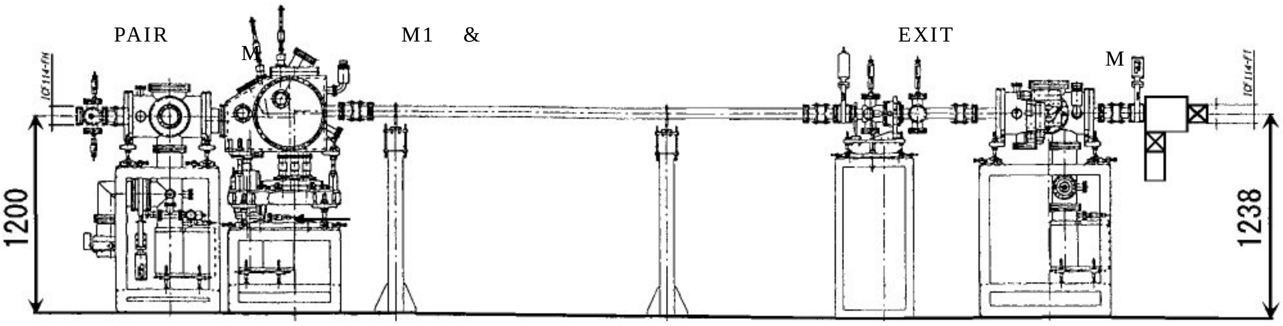


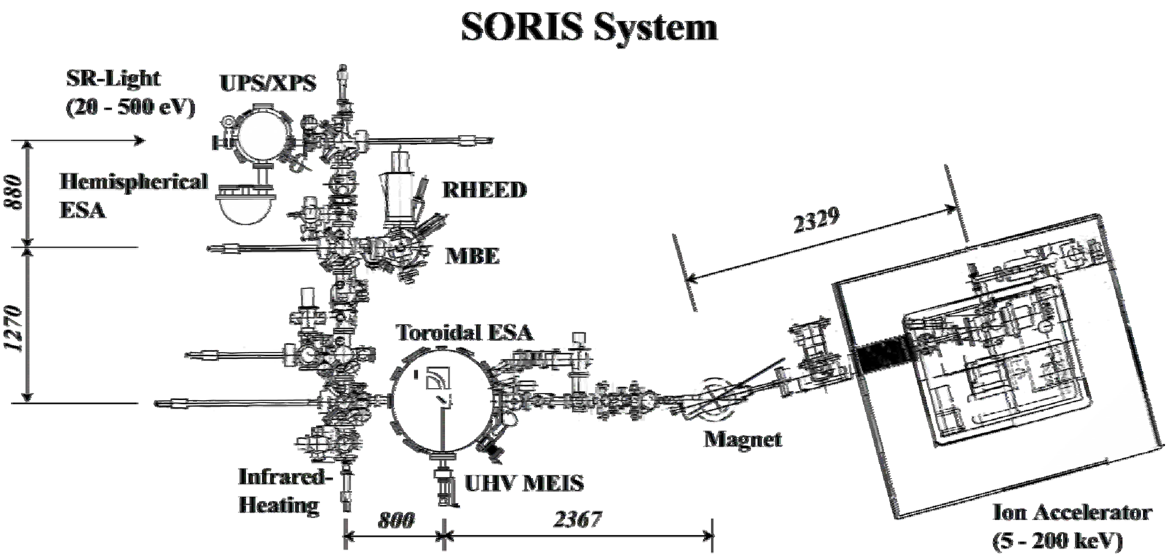
Fig. Outline of BL−8 (SR section only)

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(2×10^{-10} Torr) and various gas ambiance (O_2 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) × 2.0(vertical) mm^2 and the angular spread is ±0.2˚(vertical). Scattered ion energies are analyzed by a new toroidal electrostatic analyzer with an excellent energy resolution of $\Delta E/E = 9\times10^{-4}$. 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.



Specification:

Vacuum: $\leq 2\times10^{-10}$ Torr (Base)

Ion Beam: 5 – 200 keV He^+, H^+ and Ne^+, Beam Size: 0.18(horizontal)×2.0(vertical) mm^2

Toroidal Electrostatic Analyzer: $\Delta E/E = 9\times10^{-4}$,

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

This beamline 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 of a tungsten mesh evaporated with copper, and a sample chamber. The radiation beam with 6 mrad (horizontal) and 2 mrad (vertical) is deflected upward by 1.4 º and focused at the sample position 9 m apart from the source point with the 1:1 geometry. The available photon energy covers from 1000 to 4000 eV by exchanging several monochromatizing crystals. Na Mg, Al, Si, P, S, Cl, K, Ca K-edge XAFS spectra can be measured with several detection modes (see specification).

Specification

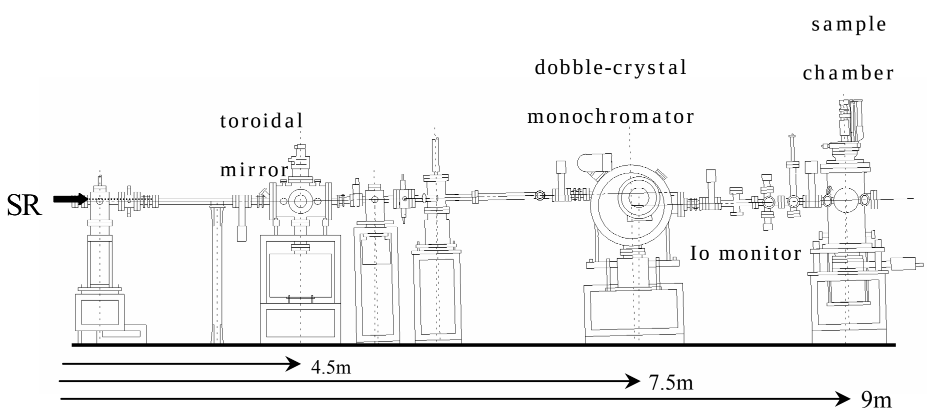
Optical system : pre-focusing toroidal mirror (1000 Å nickel-coated silicon single crystal, water-cooled) and Golovchenko-typedouble-crystal monochromator with a crystal pair of either Ge(111), InSb(111), quartz(10-10) or KTP(011).

Photon energy : 1000 eV – 4000 eV

Beam acceptance : 5 mrad (horizontal) and 1 mrad (vertical)

Sample chamber : kept in vacuum less than 10^{-8} Torr.
More than 10 kinds of samples can be loaded in a sample holder.

Detector : (1) Sample current for the total electron yield
(2) Silicon drift detector with 8 μm Be filter for the fluorescence X-ray yield
(3) MCP with a retarding grid for the total and partial electron yield mode, and the fluorescence X-ray yield
(4) Silicon photodiode for the transmission mode



BL-12

Soft X-Ray Microscope Station

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 systems are included in the project.

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 (20μmϕ), 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 λ=2.5nm. The resolution is estimated to be below 71nm (20－80%) from the intensity gradient of its knife edge profile at 2.4nm.

For a dry sample observation, almost any kind of holder can be mounted in the sample stage, and micro grids with carbon substrate are typically used. For a wet sample observation, 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×512 pixel, each 24μm×24μm
Spatial resolution	: 71nm (20－80%) at λ=2.4nm
Scientific applications	: Biology, Medical, Polymer sciences, Material sciences, Environmental sciences, Powders.

Table1 Specification of the CZP and OZP.

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

BL-13

Exposure Beamline for LIGA Process with high flexibility

This beamline has been newly 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 2D and 3D microstructures. Figure 1 shows a drawing of BL-13. The whole length of the beamline is 1.58 m. A 200 mm-thick beryllium(Be) window (5 x 30 mm) is used to separate the ultra high vacuum part from the low vacuum part and we get hard X-ray photons higher than 1.3 keV. 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 (δ-axis). On the other hand, we can also move X-ray mask in X-direction by computer and in Z-direction manually (See Fig. 2). Experiments can be done either in vacuum or in He gas. This beamline enable us to make many different 2D and 3D microstructures by changing the mask shape, sample moving speed and tilting direction.

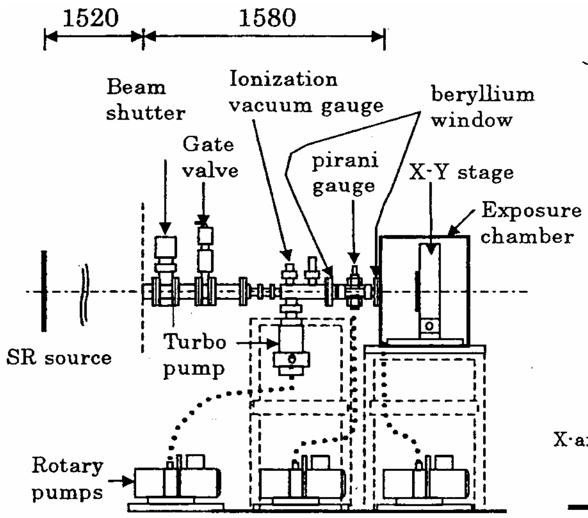


Fig.1 Schematic drawing of BL-13

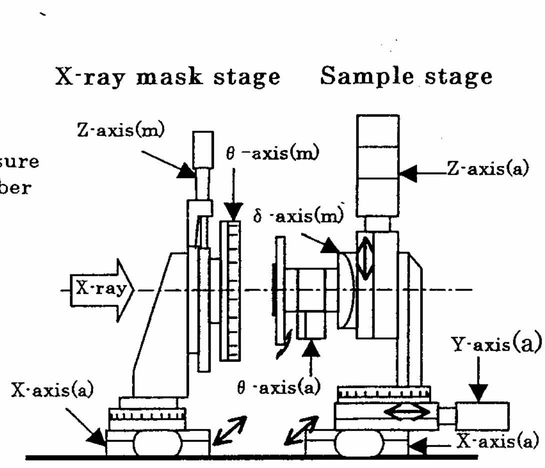


Fig.2 Sketch of the stage
(a: automatic, m: manual)

Specification

Energy range : 2 –10 keV Mask Holder : 126 x 126 mm^2

Beam size:5(H) x 38(V) mm^2 Mask-sample gap: 100 –2000 mm(adjustable)

Max sample size: 100 x 100 mm^2 Chamber pressure : 1 atm (He/Air>0.99) or vacuum

BL-15

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□, 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 3-dimensional processing (Fig.2). Beam size is $30\times5mm^2$ and the maximum exposure area is $80\times55mm^2$.

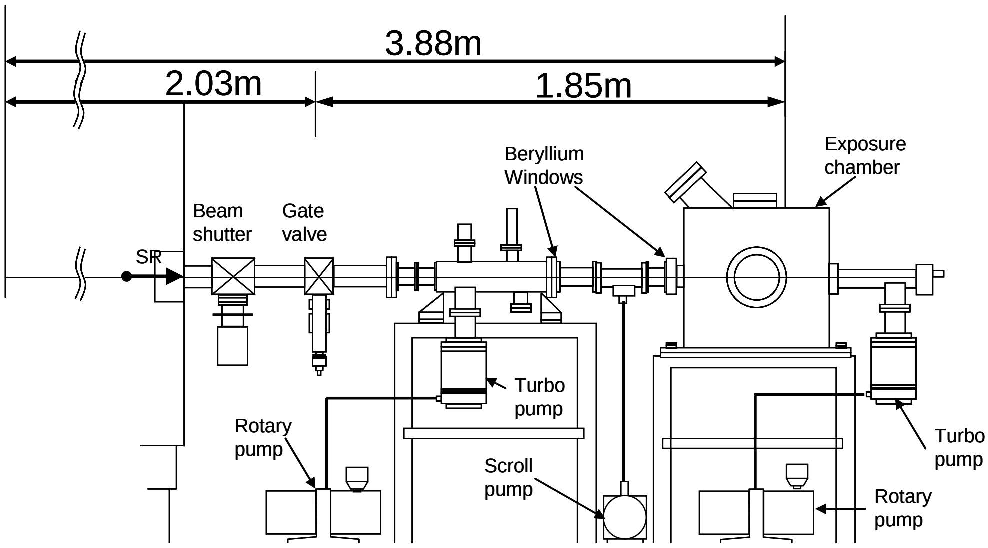


Fig.1 Outline of BL-15

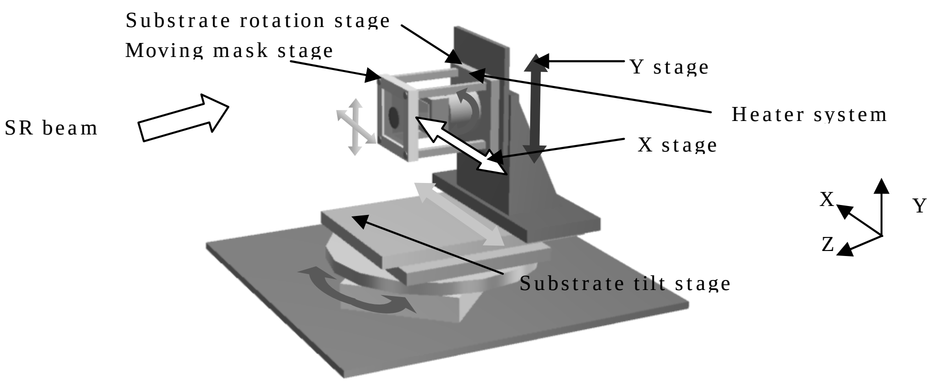


Fig.2 Stage system