BL-1

Beamline for X-Ray Diffraction and Scattering Experiment

This beamline is for X-ray diffraction experiments using a wide-band parallel X-ray beam. It consists of a pre-focusing toroidal mirror and a wide-bandpass multiplayer monochromator. The band width is 600eV and useful energy range is from 4000eV to 8000eV. It is possible to measure the energy-dispersive intensity profile of Bragg reflections of organic or inorganic crystals.

With large curved aperture (460mm × 256mm, 127.4mm radius) imaging plate as a detector, Weissenberg type camera provides rapid diffraction data collection. Partial- $\chi$, 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
Optical elements : pre-focusing toroidal mirror
wide-bandpass multiplayer monochromator
Photon energy : 4000eV – 8000eV
Energy width : 600eV
Pre-focusing toroidal mirror
Substrate : silicon single crystal Surface material : Pt
Size : L430mm × W70mm × T30mm Glancing angle : 0.55°
Wide-bandpass multiplayer monochromator
Substrate : silicon single crystal Layer material : Si / W
Size : L300mm × W40mm × T15mm Glancing angle : 1.0°
Diffractometer : Oscillation method / Weissenberg method
X-ray detector : Imaging plate
$\theta$ range : -60 $\degree$ – 144$\degree$
BL-2

Ultra Soft X-ray (Vacuum Ultraviolet) Absorption Spectroscopy

This beamline is equipped with a 10 m Vodar type grazing incidence monochromator. Available photon energy range is 50 – 600 eV, which covers the K-edge energies of light elements from Li to O. Accordingly, this beamline is useful not only for the study of advanced materials composed of Li and B, but also for the fundamental study of photochemical reactions.

The schematic sketch of the beamline is shown in Fig. 1. Synchrotron radiation passes through a gate valve, a focusing pre-mirror room and two dumper chambers, and is introduced in the monochromator, which consists of three parts mounted on a Rowland circle: an entrance slit, a concave diffraction grating (1200 grooves/mm) and an exit slit. The former two parts are fixed, but the latter part is movable with a sample chamber (10⁻⁷ Torr).

An absorption spectrum is measured with a total electron yield mode by using either a channeltron detector (Murata Celatron EMT-6081B) or a photomultiplier (Hamamatsu R595). This beamline is also possible for soft X-ray fluorescence spectroscopy by rotating the whole system by 90°.

Fig.1 Top (upper) and side (lower) views of the Beamline 2.
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 $I_o$ 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.
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%), N₂(100%), N₂(85%) + Ar(15%), N₂(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.
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, Galvanof ormung, 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.

Specification

<table>
<thead>
<tr>
<th></th>
<th>1st Chamber</th>
<th>2nd Chamber</th>
<th>3rd Chamber</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mask size</td>
<td>126 × 126 mm²</td>
<td>φ 75 mm</td>
<td>4&quot; wafer size</td>
</tr>
<tr>
<td>Sample size</td>
<td>30 × 52 mm²</td>
<td>φ 75 mm</td>
<td>4&quot; wafer size</td>
</tr>
<tr>
<td>Mask-Sample gap</td>
<td>100 – 2000 µm</td>
<td>100 – 2000 µm</td>
<td>Min. 50 µm</td>
</tr>
<tr>
<td>Sample scan area</td>
<td>Y: ±15 mm</td>
<td>Y: ±15</td>
<td>X: ±50 Y: ±25 mm</td>
</tr>
</tbody>
</table>
BL-6

Exposure beamline for LIGA Process

The Beamline BL-6 is set up for exposure of deep X-ray lithography in the LIGA (German acronym for Lithographie, 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

<table>
<thead>
<tr>
<th>Energy range</th>
<th>2 – 10 keV</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mask Holder</td>
<td>126 x 126 mm²</td>
</tr>
<tr>
<td>Beam size</td>
<td>5(H) x 30(V) mm²</td>
</tr>
<tr>
<td>Mask-sample gap</td>
<td>100 – 2000 mm (adjustable)</td>
</tr>
<tr>
<td>Max sample size</td>
<td>100 x 100 mm²</td>
</tr>
<tr>
<td>Chamber pressure</td>
<td>1 atm (He/Air &gt; 0.99)</td>
</tr>
</tbody>
</table>
BL-7

Linearly-Polarized Two-Dimensional Photoelectron Spectroscopy

VUV, soft X-ray beamline BL-7 dedicated for solid and surface science has been renewed. The range of photon energy covered by a monochromator has been extended to 10-160 eV. Two-dimensional display-type analyzer (DIANA) is newly 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 ΔE/E is 0.2 ~ 1% and the angular resolution is ±0.5°.

Fig.1. Optical arrangement of the new BL-7. M₀, M₁, M₂: mirrors; S₁, S₂: entrance and exit slits; G₁-₄: spherical gratings.

Fig. 2. Schematic diagram of DIANA with new 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$^2$
  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 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)](image)
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 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 (O₂ 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) mm² 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 $\frac{\Delta E}{E} = 9 \times 10^{-4}$. It has a wide inter-electrode 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⁺, H⁺ and Ne⁺, Beam Size: 0.18(horizontal) × 2.0(vertical) mm²
Toroidal Electrostatic Analyzer: $\frac{\Delta E}{E} = 9 \times 10^{-4}$, 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

The beamline is designed for absorption spectroscopic study of materials using the 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 and a Golovchenko-type double-crystal monochromator and a sample chamber. The radiation beam with 10 mrad (H) and 1 mrad (V) divergences is focused at the sample position 9 m apart from the source point with 1:1 focusing geometry. Six kinds of flat single crystals are used as the monochromating crystals, thus covering the photon energy range from 1000 eV to 4000 eV. Mg, Al, Si, P, S and Cl K-edge XAFS spectra can be measured with either in the transmission mode, total or partial electron yield mode. A high efficient detector for the fluorescence yield mode is designed and will be available in the near future.

**Specification**

**Optical elements**: pre-focusing toroidal mirror (nickel-coated silicon single crystal plate, water-cooled).
- double-crystal monochromator (Golovchenko-type) with the monochromator crystal of either Si(111), Ge(111), InSb(111), quartz(1010), beryl(1010) or KTP(110).

**Photon energy**: 1000 eV – 4000 eV

**Acceptance**: 10 mrad (horizontal) and 1 mrad (vertical)

**Resolution**:
\[
(\Delta E/E) = 5 \times 10^{-4}
\]

**Sample chamber**: kept in vacuum less than 10⁻⁶ Torr.
- Eight kinds of samples can be loaded in a sample holder.
  A simple dry box is designed to prevent samples from hygroscopic environment and oxidation.

**Detector**: Photodiode for the transmission mode, MCP with a retarding grid for the total and partial electron yield mode.
**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 prefocusing, a light microscope can be used, and (3) an X-ray image can be recorded on a CCD camera system.

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 (20µm), 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 50nm (25−75%) from the intensity gradient of its knife edge profile.

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. Imaging time is typically 10-60s.

**Specification**

<table>
<thead>
<tr>
<th>Specification</th>
<th>CZP</th>
<th>OZP 1</th>
<th>OZP 2</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Energy range</strong></td>
<td>0.28−1.2 keV ( Wavelength : 1.0−4.4nm )</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Optical element</strong></td>
<td>SiC Plane mirror, CZP, OZP</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Detector</strong></td>
<td>Cooled CCD camera (C4880-21-24WD: Hamamatsu Photonics)</td>
<td>512×512 pixel, each 24µm×24µm</td>
<td></td>
</tr>
<tr>
<td><strong>Spatial resolution</strong></td>
<td>46nm (25−75%) and 56nm (20−80%) at λ=3.2nm</td>
<td>49nm (25−75%) and 73nm (20−80%) at λ=2.4nm</td>
<td></td>
</tr>
<tr>
<td><strong>Scientific applications</strong></td>
<td>Biology, Medical, Polymers, Material sciences, Environmental sciences, Powder.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>CZP</th>
<th>OZP 1</th>
<th>OZP 2</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Diameter (µm)</strong></td>
<td>9,000</td>
<td>50</td>
<td>56</td>
</tr>
<tr>
<td><strong>Number of the zones</strong></td>
<td>41,890</td>
<td>277</td>
<td>311</td>
</tr>
<tr>
<td><strong>Outermost zone width (nm)</strong></td>
<td>53.7</td>
<td>45</td>
<td>45</td>
</tr>
<tr>
<td><strong>Zone material</strong></td>
<td>Ge (t=0.3µm)</td>
<td>Ni (t=0.13µm)</td>
<td>W (t=0.12µm)</td>
</tr>
<tr>
<td><strong>Support material</strong></td>
<td>SiN (t=0.1µm)</td>
<td>SiN (t=0.1µm)</td>
<td>SiN (t=0.1µm)</td>
</tr>
</tbody>
</table>
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 mm²
Beam size: 5(H) x 38(V) mm²    Mask-sample gap: 100 – 2000 mm (adjustable)
Max sample size: 100 x 100 mm²    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°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 3-dimensional processing (Fig. 2). Beam size is 30×5mm² and the maximum exposure area is 80×55mm².

Fig. 1 Outline of BL-15

Fig. 2 Stage system