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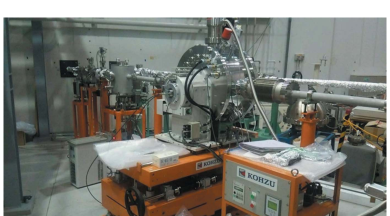
Performance of BL07A at NewSUBARU with installation of a new multi-layered-mirror monochromator

Shotaro Tanaka, Shuto Suzuki, Tomohiro Mishima and Kazuhiro Kanda*

Laboratory of Advanced Science and Technology for Industry, University of Hyogo, 3-1-2 Koto, Kamigori, Ako, Hyogo 678-1205, Japan. *Correspondence e-mail: kanda@lasti.u-hyogo.ac.jp

Soft X-rays excite the inner shells of materials more efficiently than any other form of light. The investigation of synchrotron radiation (SR) processes using inner-shell excitation requires the beamline to supply a single-color and high-photon-flux light in the soft X-ray region. A new integrated computing multi-layered-mirror (MLM) monochromator was installed at beamline 07A (BL07A) of NewSUBARU, which has a 3 m undulator as a light source for irradiation experiments with high-photon-flux monochromatic light. The MLM monochromator has a high reflectivity index in the soft X-ray region; it eliminates unnecessary harmonic light from the undulator and lowers the temperature of the irradiated sample surfaces. The monochromator can be operated in a high vacuum, and three different mirror pairs are available for different experimental energy ranges; they can be exchanged without exposing the monochromator to the atmosphere. Measurements of the photon current of a photodiode on the sample stage indicated that the photon flux of the monochromatic beam was more than 10^{14} photons s^{-1} cm^{-2} in the energy range 80–400 eV and 10^{13} photons s^{-1} cm^{-2} in the energy range 400–800 eV. Thus, BL07A is capable of performing SR-stimulated process experiments.



1. Introduction

Reaction processes originating from the excitation of inner-shell electrons of atoms are different from those originating from excitation of valence electrons, because the inner-shell electrons are localized within molecules. Specific atoms can be excited using inner-shell excitation because the binding energies are particular to each atom and the chemical bond environment (Eberhardt *et al.*, 1983; Bernd, 2012). The transition processes of these inner-shell excitations correspond to the high-energy X-ray region, so de-excitation occurs very early ($<10^{-14}$ s). In particular, inner-shell excitation of light elements, *i.e.* elements in the second and third periods, predominantly involves Auger electron emission, which is a non-radiative process, in the vicinity of the excited atom. This type of de-excitation usually generates a two-hole excited state. The two-hole excited state is unstable and undergoes a decoupling process of ionic dissociation or detachment triggered by Coulomb repulsion between two positive holes. This ionic dissociation originating from the inner-shell excitation contains information on the initial resonance excitation, namely, the transition from the inner-shell electron of a particular atom to an anti-bonding orbital and suggests the possibility of using inner-shell resonance excitation for site-selected chemical bond cutting. This reaction process is especially interesting from the perspective of cutting specific chemical bonds through the control of light, *i.e.* as a 'molecular scalpel'. In addition, the soft X-ray region has the largest reaction cross section in the light spectrum for such a cutting reaction. For these reasons, inner-shell excitation has attracted attention as a surface modification process at relatively low temperatures because it can cause a reaction without emitting extra energy (Wiedemann, 2010; Willmott, 2019). To excite inner shells of various elements, the energy of the light source must be tunable in the soft X-ray region, and synchrotron radiation (SR) is realistically the only available light source to serve this purpose. Furthermore, a beamline with a high-brightness beam is necessary to study inner-shell excitation reactions of a single color by using soft X-rays. Moreover, the beamline should be able to irradiate a sample's surface with high-photon-flux monochromatic light.

We report here the installation of a new multi-layered-mirror (MLM) monochromator at beamline 07A (BL07A) in the NewSUBARU Synchrotron Radiation Facility. This is designed for experiments on inner-shell excitation in which irradiation with high-photon-flux monochromatic light is produced by a combination of an undulator source and a multilayer-film-mirror monochromator.

The BL07A beamline was designed for investigating functional surfaces by using high-flux soft X-rays. It has a 3 m undulator to supply soft X-rays in the range 80–800 eV. The reflectance of many materials to soft X-rays is very low. Moreover, while a grating monochromator has high energy-resolving power, the monochromatic light it reflects is very weak. In contrast, an MLM can be used to improve the reflectivity index in the soft X-ray region. Higher reflectance can be gained using an MLM because the incident soft X-ray is reflected by the many interfaces of the MLM. Hence, an MLM monochromator is superior to a grating monochromator for the purpose described above; BL07A was originally equipped with an MLM monochromator that could be operated under ultra-high-vacuum conditions (Kanda *et al.*, 2001). However, this monochromator could not be used in actuality because of its low mechanical operability to withstand high temperatures during baking. In the previous studies reported by our group, we removed this monochromator and performed various studies on surface modification processes by directly irradiating samples with the undulator light (Heya *et al.*, 2017, 2018). Many research results were obtained, such as one showing the crystallization temperature of polysilicon is 200°C lower than what was previously believed (Heya *et al.*, 2013). However, there are two problems with irradiating a sample directly with undulator light, which includes multiple higher-order harmonics due to the undulator: (i) harmonics not at the desired energy become a heat source that needlessly raises the sample's surface temperature; (ii) the photon flux cannot be estimated because the sample is irradiated with multiple higher-order harmonics with different energies, and their intensity ratio cannot be evaluated. Therefore, we could not determine the reaction cross sections of inner-shell excitations at this beamline. To solve these problems, we installed a new MLM monochromator that can be operated under a high vacuum, by setting two differential pumping chambers upstream and downstream of the monochromator (Fig. 1). Here, we report the experimentally estimated photon flux when using the new MLM monochromator under each irradiation condition at BL07A.

Figure 1
Beamline 07A of NewSUBARU, where a new MLM monochromator was installed.

2. Beamline overview

NewSUBARU is a SR facility designed to generate bright light in the soft X-ray region with the goal of promoting advanced research and development of synchrotron light sources and their application. It is located at the SPring-8 site and has a 1.5 GeV electron storage ring 118 m in circumference (Ando *et al.*, 1997; Hashimoto *et al.*, 2001). The electron beams of the storage ring are currently delivered by the SPring-8 LINAC at an energy of 1.0 GeV; a new C-band LINAC will be installed in 2021.

The NewSUBARU ring is operated in two modes by selecting between two electron energies, 1.0 and 1.5 GeV. In 1.0 GeV operation mode, top-up operation (electron injection at any time) is performed to keep the stored current at 300 mA. In 1.5 GeV operation mode, beam energy is ramped up to 1.5 GeV after the accumulation of a beam current of 350 mA at 1.0 GeV. Therefore, the ring current in the storage ring decays in the 1.5 GeV operation mode.

Beamline 7 (BL07) at NewSUBARU was constructed for researching the creation of new functional materials. For new materials development using SR, two kinds of beamline are required in the soft X-ray region. One is a high-photon-flux and energy-tunable beamline for studying SR-stimulated processes, such as inner-shell excitation leading to selective photochemical reactions. The other is a high-resolution beamline for the analysis of the produced materials, such as by photoelectron spectroscopy and X-ray absorption spectroscopy. BL07 thus consists of two branch lines. BL07A is a SR-stimulated reaction beamline using high-photon-flux light; it has an MLM monochromator. BL07B is a material analysis beamline; it has a constant-deviation monochromator, consisting of a demagnifying spherical mirror and varied-line-spacing plane grating (VLSPG). This type of monochromator can provide high resolution through simple wavelength scanning with fixed slits (Haruyama *et al.*, 2007; Nishihara *et al.*, 2008).

The light source of BL07 is a 3 m planar undulator, which consists of 29 sets of permanent magnets, the period length of which is 76 mm (Ando, 2000; Miyamoto *et al.*, 2000). The K parameter of the undulator ranges from 0.3 to 5.3, and the radiation wavelength from this undulator ranges from 66 to 4.4 nm when utilizing the fundamental and third through seventh higher harmonics. The brilliance of this undulator, which was calculated as 100 mA of the ring current (Miyamoto, 2001), is depicted in Fig. 2(*a*) for 1.0 GeV operation mode and Fig. 2(*b*) for 1.5 GeV operation mode. Brilliance steeply decreases at photon energies more than 400 eV in 1.0 GeV operation mode. On the other hand, brilliance in 1.5 GeV operation mode remains high enough out to 800 eV.

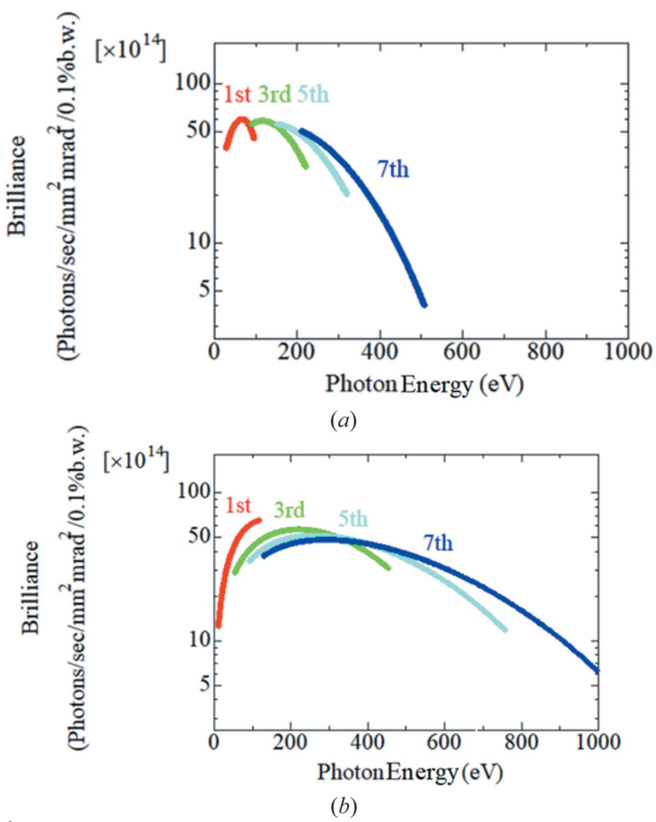


Figure 2
Calculated brilliance of the 3 m undulator in (*a*) 1.0 GeV operation mode and (*b*) 1.5 GeV operation mode.

The incident beam from the undulator is provided for the two branch lines, BL07A and BL07B, by translational switching of the first mirror.

Fig. 3 shows the overall setup of BL07A from the front end of the storage ring to the reaction chamber. The optical layout of BL07A has not changed from the design described by Kanda *et al.* (2001). The front end consists of a mask, absorber and four-quadrant slit. The acceptance angles are 2.2 mrad horizontally and 2.4 mrad vertically. The first mirror, the switching mirror, is spherical with a radius of curvature of 312 400 mm. The center of the first mirror is located 8174 mm downstream from the center of the undulator, and its incident angle is 3°. The first mirror is made of Si and its surface is coated with Pt. The induced undulator light is reflected by this mirror and propagates outside the protective wall as quasi-parallel light.

Undulator light reflected by the first mirror reaches the MLM monochromator, located 7100 mm downstream from the center of the first mirror, through a four-quadrant slit and differential pumping vessel 1. As described in Section 1, we installed a new MLM monochromator that can operate in a high vacuum. This monochromator, MKZ-7NS (Kohzu Precision Co. Ltd), is controlled with integrated computing, in which mechanically independent axes are programmatically controlled for keeping a fixed exit position. The Bragg angle can be varied from 9 to 65°. The beam offset of the monochromator is precisely 15 mm. This design reduces the convolution of mechanical errors, and the control mechanism is very compact. Three different pairs of mirrors can be selected and they can be exchanged without exposing the monochromator to the atmosphere. The details of the monochromator were reported by Okui *et al.* (2016).

A filter chamber and differential pumping vessel 2 are downstream of the MLM monochromator. The undulator light through a filter is introduced into the reaction chamber. A four-quadrant beam-forming slit is placed in front of the reaction chamber. The irradiation point of the sample in the reaction chamber is located at 11 927 mm downstream from the center of the first mirror. The reaction chamber has a load-lock system, and samples can be placed at the irradiation position without exposing the chamber to the atmosphere. The plane size of the sample holder is 70 mm long × 25 mm wide,

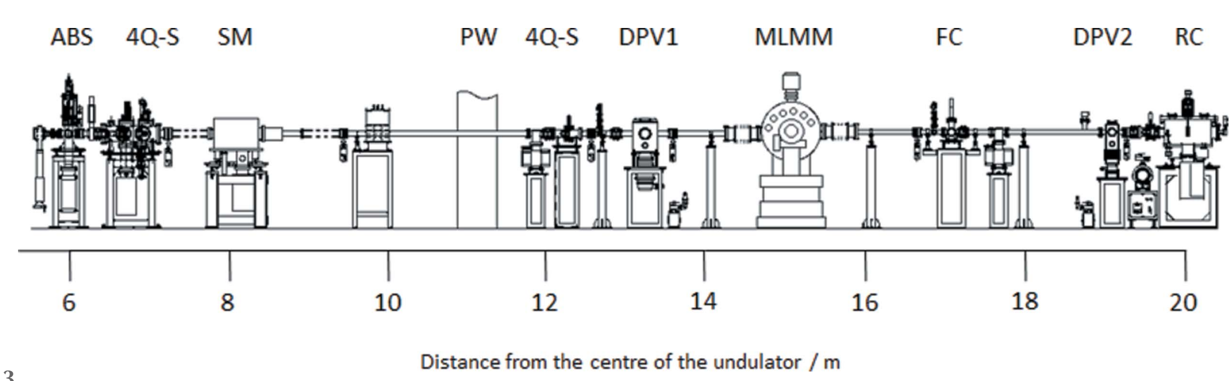


Figure 3
Layout of BL07A showing the position of beamline components. ABS: absorber; 4Q-S: four-quadrant slit; SM: switching mirror; PW: protective wall; DPV1: differential pumping vessel 1; MLMM: multi-layered-mirror monochromator; FC: filter chamber; DPV2: differential pumping vessel 2; RC: reaction chamber.

and both sides of the sample holder can be used. The beam spot at the irradiation point is about 25 mm × 25 mm. The intensity of light is made almost homogeneous in a 20 mm square around the center by checking with a 3 mm × 3 mm photoelectric board.

3. Experimental setup and results

The photon flux at the irradiation point was measured with a photodiode (PD), AUXV100 (International Radiation Detectors Inc.), whose light-receiving area was 10 mm × 10 mm. The PD was placed about 205 mm downstream from the irradiation point in the reaction chamber. To confirm the linearity of the photocurrent from the PD against the photon flux, we measured the output current of the PD while varying the current of the storage ring. The output current of the PD showed good linearity in the range below 1.7 mA. The data sheet of the AUXV-100 indicates that its quantum efficiency has a temperature dependence of 0.045% per °C. The temperature of the PD can be considered to depend on the quantity and energy of the irradiated X-rays. Unfortunately, we could not measure the surface temperature of the PD directly, because the reaction chamber does not have a window port for observing the PD's surface. Instead, we measured the surface temperature of amorphous Si (a-Si) thin film placed at the irradiation point through a ZnSe window by using an infrared radiation thermometer (CPA-T400A, Chino Co.). Under six energies of X-ray irradiation at the storage ring current of 300 mA, the surface temperature of the a-Si thin film varied from 20.5°C to 107.5°C. As a result, the error range of the photon flux due to temperature was estimated to be within 2%.

The photon energy of the monochromatic light of BL07A cannot be determined precisely, because the installed MLM monochromator has low resolving power. Instead, we assessed the relationship of the energies of the undulator harmonics with the gaps of the 3 m undulator at BL07B, which has a grating monochromator with a high resolving power, and we set the energy of the MLM monochromator to the peak positions of the harmonics estimated from this relationship.

3.1. Multi-layered mirrors and energy resolution

Undulator light includes the fundamental and multiple higher harmonics. To eliminate undesired undulator harmonics, undulator light is monochromated by a combination of an MLM monochromator and three different metal foils prepared as filters. Three different MLMs are used to cover an energy range from 80 to 800 eV. The MLMs are composed of multi-layered mirrors made from Mo and Si, Mo and B_4C, and Ni and C. Their spacing, thickness ratio, number of layers, ideal energy resolution calculated from the specifications and energy range are summarized in Table 1. The filters are made from Ag, Cr and Ni; the film thickness and energy range of each film are listed in Table 2. The transmittances of the filters, which were calculated using the X-ray database of the Center for X-ray Optics (Gullikson, 1995), are shown in Fig. 4.

Table 1
Specifications of MLMs and their usable energy ranges.

Multi-layered mirror	Spacing (nm)	Thickness ratio	Number of layers	Designed $\Delta E/E$ (%)	Energy range (eV)
Mo/Si	20	0.8	20	6.2	80–200
Mo/B_4C	11	0.5	25	3.3	125–360
Ni/C	5	0.5	60	2.5	260–800

Table 2
Specifications of filters and their usable energy ranges.

Material	Thickness (nm)	Energy range (eV)
None		80–140
Ag	100	140–400
Cr	500	400–560
Ni	500	560–800

Energy resolution was estimated by measuring the PD current against the photon energy of the MLM monochromator. The electron energy and current of the storage ring were 1.0 GeV and 300 mA, respectively. The third harmonics of the undulator light were used with the undulator gap of 56.2 mm. The Mo/B_4C MLM and Ag filter were used. The observed spectrum is shown in Fig. 5. The resolution, $\Delta E/E$,

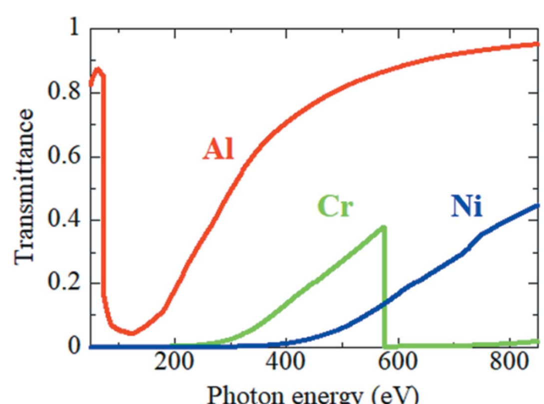


Figure 4
Calculated transmittances of Ag, Cr and Ni filters.

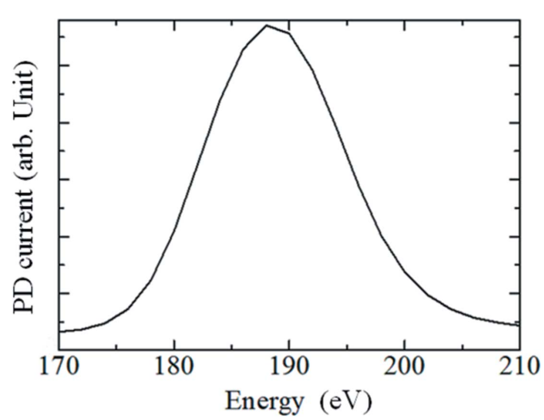


Figure 5
Observed spectrum of third harmonics of undulator light with an undulator gap of 56.2 mm. The MLM and filter used were Mo/B_4C and Ag, respectively.

estimated from this figure is 7.9%, which is about twice the ideal energy resolution calculated from the specifications of the MLM. The resolution is sufficient for picking out a specific harmonic from other harmonics of the undulator with a certain gap.

3.2. Photon flux

Photon fluxes in 1.0 GeV and 1.5 GeV operation modes were estimated as follows. The photon flux in 1.0 GeV operation mode was calculated from the PD current measured when the ring current was 300 mA. The photon flux in 1.5 GeV operation mode was obtained after the PD current was normalized by the ring current of 300 mA. After normalization, the photon fluxes were obtained for each irradiation condition, with the quantum yield corresponding to the photon energy listed in the data sheet for the AUXV-100.

Figs. 6 and 7 show the photon fluxes of monochromatic SR at the reaction chamber of BL07A measured in 1.0 GeV operation mode and 1.5 GeV operation mode, respectively. The colors of the dots indicate the type of MLM used: red, green and blue dots, respectively, indicate measurements made with Mo/Si, Mo/B4C and Ni/C MLMs. Moreover, the shapes of the dots correspond to the harmonics of the undulator: circular, square, triangular and diamond-shaped dots correspond to the fundamental, third, fifth and seventh harmonics of the undulator light, respectively. An adequate filter corresponding to the energy as listed in Table 1 was used for each measurement. The intensity unit of photon flux was defined as photons s^{-1} cm^{-2}. The error range for the photon flux in these figures was estimated to be 20–30%.

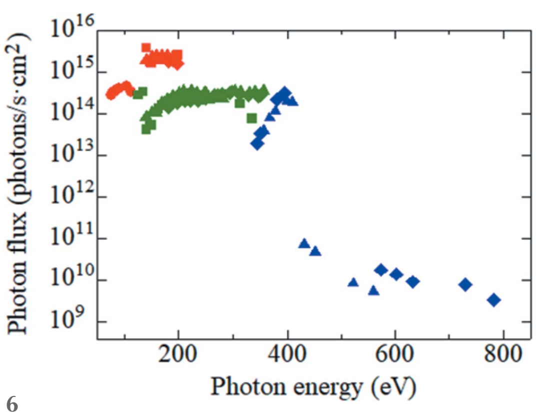


Figure 6
Photon flux at the irradiation point of the sample stage of BL07A in 1.0 GeV operation mode. Ring current was 300 mA. Red dots, green dots and blue dots were measured with Mo/Si, Mo/B_4C and Ni/C MLM pairs, respectively. Circular, square, triangular and diamond-shaped dots represent fundamental, third, fifth and seventh harmonics of undulator light, respectively.

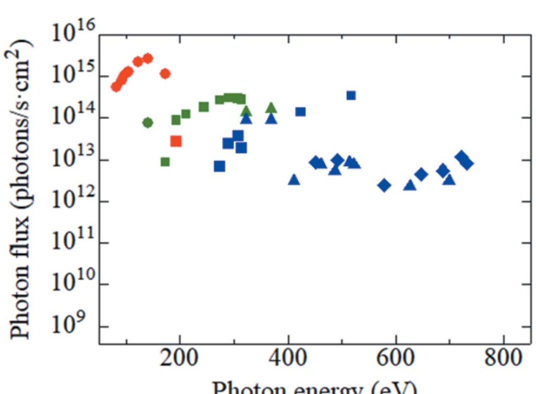


Figure 7
Photon flux at the irradiation point of the sample stage of BL07A in 1.5 GeV operation mode. Flux was normalized by a ring current of 300 mA. Red dots, green dots and blue dots were measured with Mo/Si, Mo/B_4C and Ni/C MLM pairs, respectively. Circular, square, triangular and diamond–shaped dots represent fundamental, third, fifth and seventh harmonics of undulator light, respectively.

In the range 80–200 eV, the photon flux went up to more than 2×10^{14} photons s^{-1} cm^{-2}. The highest photon flux in the range 125–200 eV was about 4×10^{15} photons s^{-1} cm^{-2}. The photon fluxes monochromated by the Mo/Si MLM were higher than those monochromated by the Mo/B_4C MLM in the range 125–200 eV. The photon fluxes in the range 200–400 eV were about 2×10^{14} photons s^{-1} cm^{-2}. The photon fluxes monochromated by the Mo/B_4C MLM were higher than those monochromated by the Ni/C MLM in the range 280–360 eV, while they were almost the same value in the range 360–400 eV. In the range above 400 eV, photon fluxes measured in 1.5 GeV operation mode were about 10^{13} photons s^{-1} cm^{-2}. However, those measured in 1.0 GeV operation mode were lower than 10^{11} photons s^{-1} cm^{-2}. Therefore, the photon-flux-to-sample in 1.5 GeV operation mode was clearly much higher than that in 1.0 GeV operation mode in the energy range higher than 400 eV. The above results indicate that BL07A can be used to investigate the SR process for monochromatic soft X-rays with photon fluxes higher than 10^{13} photons s^{-1} cm^{-2}. In particular, irradiation experiments using high photon fluxes exceeding 10^{14} photons s^{-1} cm^{-2} can be performed in the energy range 80–400 eV.

Now let us compare the photon fluxes of BL07A with those of several other beamlines at NewSUBARU. All values described below are for 1.0 GeV operation mode with a ring current of 300 mA. Beamline 03 has seven multi-layered mirrors and uses a bending magnet as a light source (Watanabe *et al.*, 2005). The flux at 91.8 eV (13.5 nm) is 2×10^{13} photons s^{-1} cm^{-2}, and the beam spot is 4 mm × 4 mm. Beamline 10 has a grating monochromator and its light source is a bending magnet (Kuki *et al.*, 2015). The flux at 150 eV is 1×10^{11} photons s^{-1} cm^{-2}, and the beam spot is 0.1 mm high × 0.8 mm wide. Beamline 09 is a high-flux beamline with high energy resolution; it has an 11 m undulator as a light source and a grating monochromator (Niibe, 2004). The flux at 91.8 eV (13.5 nm) is 3.4×10^{14} photons s^{-1} cm^{-2}, and the beam spot was 0.5 mm high × 2.4 mm wide. Therefore, BL07A offers a higher flux of monochromatic light over a larger area compared with these other beamlines.

4. Conclusions

A high-photon-flux beamline (BL07A) for research and development of new functional materials was constructed at NewSUBARU by combining an undulator and MLM mono-

Figure 8
New sample holder for irradiation at low temperatures.

chromator. A combination of three different MLM mirror pairs and three different metal thin-film filters enables the selection of specific harmonics of undulator light in a range from 80 to 800 eV. The photon fluxes irradiating the irradiation point were measured using a PD for different experimental conditions, *i.e.* the electron energy of the storage ring, undulator gap, MLM pair used and filter used. The incident beam at the reaction chamber was more than 10^{13} photons s^{-1} cm^{-2} in the energy range 80–800 eV. This flux is sufficient for irradiation experiments, and the energy range covers the ionization energies of useful light elements, such as the C *K* edge and Si *L* edge. BL07A is thus a useful beamline for the investigation of SR processes.

In addition, we fabricated a new holder made of copper for irradiation at relatively lower temperatures to enable low-temperature processes to be studied (Fig. 8). This holder can suppress the temperature rise of the sample surfaces by circulating water supplied from a chiller. We performed a preliminary test on this cooling system and observed that the surface of the target polysilicon substrate was cooled from 350°C to 80°C. This sample holder will enable modification processes to be studied at lower temperatures.

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