BL3U

# Performance of a High-Precision Transmission Grating for High-Resolution Soft X-Ray Emission Spectroscopy

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The X-ray emission spectroscopy (XES) is to measure X-rays emitted as a photon-in/photon-out process arising from the decay of the inner-shell excitation and resonant Raman or inelastic scattering. The XES technique combined with the synchrotron radiation is a powerful method to study occupied partial density of states and element-specific excitations of materials. However, a serious problem exists in XES using a reflection grating, that is, a rather low efficiency in detecting emitted X-rays due to (i) the limited number of X-ray focusing optics in the Rowland circle mount (vertical direction only) and (ii) the low acceptance angle and low quantum efficiency at charge coupled devices (CCD) detector with the grazing-incident configuration in the soft X-ray region. Such problems are in general solved by the use of high brilliant X-ray beam of 10<sup>13</sup> photons/sec. In this case, however, one has to take care of radiation damage, in particular in soft matters.

In order to overcome the above problems, we have developed a new X-ray emission spectrometer based on a transmission grating (TG) at UVSOR BL3U. The spectrometer adopts a novel optical design with a Wolter type I mirror, a free-standing TG, and a back-illuminated CCD, which enables high efficiency and high energy resolution due to an omnidirectional focusing for emitted X-rays and the normal-incident configuration for the CCD detector.

Very recently, we have installed a new TG, which was fabricated by NTT-AT Co. Ltd. In order to achieve the high precision of the TG spectrometer, the new TG is focused on the stability and accuracy of the groove structure. In the high-precision TG, the SiC thickness is about 540 nm, the groove density is 5555 lines/mm (180 nm periods), and the size of the slit is 108 nm at the surface side and 64 nm at the backside as illustrated in Fig. 1. In this report, we show the result of evaluation of the performance for the high-precision TG spectrometer in the energy region of 60–300 eV.

Figure 2 shows an example of resonant XES in the elastic peak region of a LiF(100) single crystal, measured at an incident photon energy hv = 61.0 eV. We observed a very sharp elastic peak in the Li K $\alpha$  spectrum with the full-width at half-maximum of about  $\Delta E_{out} = 0.02$  eV, *i.e.*,  $E/\Delta E_{out} \sim 3050$ . Such a performance has been examined for various photon energies using Si(111), InP(100), CdS(0001), *h*-BN, and HOPG as summarized in Table 1. Note that, the emission intensity is reasonably high for all XES data, e.g., 0.5–1 spectrum/hr. As for the energy resolution, the high precision TG spectrometer shows a high

performance below hv = 100 eV with the resolving power of  $E/\Delta E_{out} \sim 3000$ , which is the advantage in our XES system; e.g., the present spectrometer may offer new insight into the field of Li-based functional materials such as energy-storage devices.



Fig. 1. Scheme of the high-precision transmission grating with the 5555 lines/mm groove density.



Fig. 2. Elastic peak of the Li K $\alpha$  XES spectrum (hv = 61.0 eV) for the LiF(100) single crystal.

Table 1. Performance of the transmission grating spectrometer with the 5555 lines/mm groove density.

hv/eV	$\Delta E_{\rm out}/{\rm eV}$	$E/\Delta E_{\rm out}$	Material
61.0	0.02	3050	LiF(100)
100.0	0.03	3333	Si(111)
139.6	0.08	1745	InP(100)
164.1	0.16	1026	CdS(0001)
196.0	0.20	980	<i>h</i> -BN
285.5	0.30	952	HOPG

#### BL3B

## New VIS-VUV Bending Magnet Beamline Design for BL3B

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The solid state optical devices in ultraviolet (UV) region will bring dramatic improvements in not only the telecommunication applications but also the illuminating, environmental and medical applications. To achieve both technological and scientific investigations of these device materials, the light sources which cover wide photon energy region from visible (VIS) to vacuum ultraviolet (VUV) are required. Then, BL3B normal incidence monochromator beamline has been planned for photoluminescence (PL) studies in VIS - VUV regions. This beamline will be constructed as the successor beamline of the BL1B (1-m Seya Namioka type monochromator beamline) which will be shutdown at the end of March 2011. In this report, we present the optical design of a new 2.5-m Eagle type monochromator beamline.

The design parameters for the beamline monochromator dedicated to the PL are usually both the brilliance and the linear polarization degree together with the photon energy coverage, photon flux, resolution and purity. These design specifications of this beamline are as follows; **Photon energy coverage** :  $2 \sim 24$  eV, **Photon flux** ( $E/\Delta E = 1000$ ) :  $\geq 10^{10}$  photons/sec, **Resolution** ( $E/\Delta E$ ) :  $\geq 10000$ , **Beam spot size** (H × V) :  $\leq 0.8 \times 0.8$  mm<sup>2</sup>, **Linear polarization degree** (P) :  $\geq 0.7$ .

Figures 1 and 2 show the schematic side view and top view layouts of the BL3B, respectively. The first plane mirror  $(M_0)$  only reflects the synchrotron radiation up to increase the design freedom of this beamline optics in a limited area with the radiation shield wall which is located between  $M_0$  and  $M_1$ . The

photon flux specification requires 40 mrad horizontal acceptance angle. Then, a toroidal mirror  $(M_1)$  has ~ 210 mm width. S (source point) –  $M_1$  to  $M_1 - S_1$ (entrance slit) distance ratio is almost 1:1 to prevent the shape deformation of the focal spot. The  $M_2$  is a plane mirror reflects the light to the x direction (xyplane and y axis are defined as the plane of incidence and the light axis). The roles of  $M_2$  are to give wide space at the sample position (Q) and to cancel the source point (S) horizontal movement under the UVSOR feature version up. An off-plane Eagle type monochromator consists of  $S_1$ , G (gratings), and  $S_2$ (exit slit). The focal length is 2.5 m satisfies both the F number matching to the pre-mirror system ( $M_0$  – M<sub>2</sub>) and the resolution specification. Three gratings  $(G_1 \sim G_3)$  with the different glazing angles cover 2 ~ 24 eV region. The blazing angles and the additional optical filters prevent the higher order lights. The deviation angle of the G is 4 degree. The post-mirror system with Kirkpatrick - Baez optics consists of both spherical mirrors  $M_3$  and  $M_4$  which focus x and z directions, respectively. The reflected light by the M<sub>4</sub> becomes the parallel to the floor. Both the VIS - UV and VUV spectrometers with CCD sensors for the PL measurements will be installed into the sample chamber at Q.

The ray tracing results of this optical configuration are as follows; **Photon flux**  $(E/\Delta E = 1000)$  :  $1 \sim 4 \times 10^{10}$  photons/sec, **Resolution**  $(E/\Delta E)$  :  $12000 \sim 46000$ , **Beam spot size** (H × V):  $0.5 \times 0.8$  mm<sup>2</sup>, **Linear polarization degree** (P) :  $0.7 \sim 0.8$ . This beamline will be constructed within FY2011.



Fig. 2. Schematic layout of the BL3B (top view).

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#### BL5B

## Measurement for the Quantum Efficiencies of Microchannel Plates

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#### Introduction

We are developing the space-based imagers for plasma emissions from the plasmasphere that is torus-shaped dense region filled with plasmas flowing upward from the earth's ionosphere. The main targets of our instruments are the He II and O II ions in the plasmasphere. The He II and O II ions have resonantly scattered emission lines at 30.4 nm and 83.4 nm respectively. Column densities along the field of view of instruments can be obtained because intensities of emissions are proportional to them under the assumption that observational targets are optically thin. Therefore, the accuracies of the detective efficiencies for the instruments are critical for estimation of the densities and to achieve the science goals of our missions. The detective efficiencies are absolutely calibrated at UVSOR.

Microchannel plates (MCPs) detect photons of UV and high-energy particles with incident positions by using position encoder, with extremely low noises. MCPs produce single charge pulses of thousands of electrons via secondary emissions. They have heritages as detectors of the space-based optical instruments. It is essential to obtain the quantum efficiencies (QE) of the MCP for the calculation of the incident intensities. It is commonly used method that photocathodes are deposited to achieve high quantum efficiencies in the spectral range of the ultraviolet. Even in this case, relative QEs of MCPs with photoelectric materials to the bare one are easily measured. Therefore, absolute QEs can be estimated.

In this experiment, the QE of one bare MCP at the wavelength of 30.4 nm is measured. This result leads calibrations of the detectors for various space-based instruments.

#### Measurement and result

The Al/Mg/Al (744 Å/3958 Å/747 Å) filter and the Sn (1730Å) filter are installed on the entrance of the beam line to achieve the pure 30.4 nm light. The purity is investigated from the consistency between the wavelength characteristics of the Al/C and the Al/Mg/Al sample filters for the light at UVSOR dispersed by the gratings and for the emission line of the RF excited helium gas light at the EUV facilities of Institute of Space and Astronautical Science (ISAS). Fig. 1 shows the transmittances of the Al/C sample filter measured at UVSOR and at ISAS, Fig. 2 shows those of the Al/Mg/Al filter. It is clear that both transmittances of the filter are consistent at 30.4 nm. Therefore, it is shown that the pure 30.4 nm light

is achieved at UVSOR with entrance filters.

With the pure lines, the QE of the MCP detector for the 30.4 nm light is measured. The QEs are calculated by the rate of the MCPs counts to the electron yield of the photo diode which is absolutely calibrated. As a result, the QE of the MCP detector for the 30.4 nm light is 9.83% with the accuracy of 0.41%.

The QE of the MCPs for the 83.4 nm light has to be measured as the future work in addition to the 30.4 nm light. The O II ions have emission line at 83.4 nm and also known as one of main components in the plasmasphere. The pure 83.4 nm light is essential for the measurements and is going to be achieved by using entrance filters and one more UV diffraction grating.



Fig. 1. The transmittances of the Al/C sample filter measured at UVSOR and at ISAS.



Fig. 2. The transmittance of the Al/Mg/Al sample filter. It is showed that the pure 30.4 nm light is at UVSOR achieved through Al/C filter and the Sn filter as the entrance filters.

## **Two-Dimensional Electron Spectroscopy on BL6U**

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A new project for constructing the undulator beamline BL6U has been started since 2007. A variable-included-angle Monk-Gillieson mounting has been selected, in order to cover a wide photon energy region (30-500 eV) with one single grating. It has been confirmed through its performance tests that the monochromator designed can cover the photon energy ranging from 40 to 400 eV with the resolving power higher than 5000 and the photon flux more than  $10^{11}$  photons/sec, when the storage ring is operated in the top-up mode.

A new electron spectrometer for gas phase spectroscopy has successfully been installed, in parallel with the construction program of BL6U. It is well known that high-resolution electron spectroscopy is a powerful tool to investigate electronic structures of atoms and molecules, especially when high-resolution electron spectra and their polarization dependences are measured as a function of photon energy in high-resolution mode. The ability of this two dimensional (2D) electron spectroscopy has been demonstrated in our recent work at SPring-8 [1, 2], where special attention is paid to detect slow electrons following core excitations. In order to apply high-resolution 2D electron spectroscopy to the investigation of the L-shell excitations of the second row elements, a new experimental setup for BL6U has been designed and constructed. As a high performance hemispherical electron energy analyzer, MBS-A1, developed by the MB Scientific AB company, has been selected.

In order to realize 2D electron spectroscopy, software development for controlling both the beamline monochromator and MBS-A1 analyzer has been performed. For the beamline monochromator, not only its output but the gap of the undulator should be controlled. After careful optimizations for the undulator gaps, 2D electron spectroscopy on BL6U has become feasible, thanks to the stable operation of the UVSOR-II storage ring.

Figure 1 and 2 demonstrate the 2D maps for the de-excitation spectra following the double excitations near the carbon 1s photoionization threshold in CO, measured in the horizontal and vertical directions, respectively, as examples of successful measurements. The exit slit opening of the monochromator was set at 30  $\mu$ m, which corresponds to the photon energy resolution of about 60 meV. The pass energy and slit width of the MBS-A1 analyzer were set to 100 eV and 0.2 mm, which results in the electron energy resolution of about 60 meV. The straight lines with a

slope of 1 in the high kinetic energy (KE) region in each 2D map are due to the valence photoelectrons with vibrational structures. The vertical lines around KE of 275 eV in Fig. 2 are assigned to the atomic Auger line from oxygen atoms after the dissociation of CO molecules. Some island-like structures are seen in the KE range of 267-270 eV in Fig. 1 and Fig. 2, which seem to be specific to the decay processes of the double excitations. The complicated photon energy dependences of the structures may indicate that the de-excitation processes of the doubly excited states are not so simple.

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Fig. 1. 2D map of de-excitation spectra following the double excitations around the carbon K-edge of CO measured in horizontal direction.



Fig. 2. 2D map of de-excitation spectra following the double excitations around the carbon K-edge of CO measured in vertical direction.

BL6U

BL7B

## Reflection Measurement on [SiC/W/Co]<sub>2</sub> and SiC/W/Ir/Ti Sub-Quaterwave Multilayers for Use in 50-110 nm Region

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Traditional multilayers consisting of high- and low-Z materials alternatively having high normal incidence reflectance with broad bands in 50-110 nm region have been developed [1]. In this region, however, because of the strong absorption of all material, the reflectance is rather low. In order to improve this situation, a theory of sub-quarterwave multilayer has been developed [2], basing on the multi-material combination. The layer thicknesses of sub-quarterwave multilayer are less than a quarter of the wave-length. Comparing with traditional multilayer, sub-quarterwave multilayer provides more interfaces per constant optical path. This provides a high reflectance with a broad band by the interfaces close to the outer surface, thus reducing absorption.

Preliminary measurement of reflectance on such multilayers of [Si/W/Co]2 and [Si/W/C]2 had been performed [3]. The subscript "2" means 2 trilayers of different layer thicknesses. In the present study, the measurements on the multilayers, [SiC/W/Co]<sub>2</sub>, [SiC/W/Ir]<sub>2</sub>, SiC/W/Ir/Co and SiC/W/Ir/Ti have been performed. The multilayers were designed to have high reflectance of 40-50% in this region at incident angle of 20° for p-polarization. In the multilayers, the top layers were SiC layers and the substrates were Si wafers. Here we report the reflectance measurement on the [SiC/W/Co]<sub>2</sub> and SiC/W/Ir/Ti multilayers. In the former, the designed value of layer thickness was respectively 10.59, 11.06, 12.67, 17.51, 10.02 and 8.69 nm from the top SiC layer, and in the latter, 10.42, 5.97, 9.49 and 9.18 nm. They were deposited by using magnetron sputtering method in Tongji University. Near normal incidence reflectance was measured at BL7B, which is equipped with a 3 m McPherson type monochromator. The p-reflectance was measured at incident angles of 20°.

The measured reflectances are shown in Figs.1 and 2 with the calculated results. It can be seen that all the measured reflectances are lower than those of the calculated ones. The possible reasons are as follows. (1) In design, the optical constants used were those from the optics handbook. There is a possibility that the optical constants used in the calculation are different from the actual ones. (2) During and after deposition, the materials reacted with atmosphere and oxidized. Some improvement should be required in next research, such as higher base vacuum condition

and capping layer to protect the multilayers from oxidization when they are exposed to air.



Fig. 1. Calculated and measured p-reflectances of [SiC/W/Co]<sub>2</sub> multilayer at incident angle of 20°.



Fig. 2. Calculated and measured p-reflectances of SiC/W/Ir/Ti multilayer at incident angle of 20°.

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BL7B

# <sup>21/D</sup>Diffraction Efficiency Measurement on Sub-Quarterwave-Multilayer -Coated Grating for Seya-Namioka Mount in 50–110 nm Region

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The multilayer-coated gratings can provide high diffraction efficiency. They have been widely used in X-ray region and vacuum-UV (VUV) region below 50 nm. However, multilayer coated gratings have been precluded in the VUV region of 50-110 nm because of the strong absorption of materials in this region. As reported in this issue sub-quarterwave multilayers having high normal incidence reflectance with broad bands in 50-110 nm region have been developed. In this study, one of these multilayers, SiC/W/Ir/Ti designed to enhance diffraction efficiency in the 50-110 nm region for Seya-Namioka mount at the -1st order is coated on a grating. Its efficiency was compared with those of the gratings coated with SiC and Ir monolayers. In Seya-Namioka mount, the deviation angle between incident and diffracted rays is constant and about 70°.

The gratings of laminar type were designed to have high efficiency in the 50–110 nm region using the modified integral method [1]. The gratings were fabricated holographically by ion-beam etching. Their line density was 1200/mm. The SiC/W/Ir/Ti multilayer, and SiC and Ir monolayers were deposited on the gratings using magnetron sputtering at Tongji University. Figure 1 shows the surface profile of the multilayer-coated grating. It can be seen that the grooves are still sharp after multilayer deposition. The diffraction efficiency for p-polarization was measured at BL7B equipped with a 3 m McPherson type monochromator in the 45–120 nm. The measurement was made keeping the deviation angle of Seya-Namioka mount.

The measured diffraction efficiency spectra of the gratings coated with SiC/W/Ir/Ti multilayer, and SiC and Ir monolayers are shown in Fig. 2 with calculated ones. The relative trend of the measured spectrum of each grating agrees with that of calculated one. However, the measured efficiencies are lower than the calculated ones except the result at 56 nm for the grating coated with Ir. The plausible reasons of this fact are the deviation of the actual groove depth from the designed one, which causes a wavelength shift of efficiency peak and the deviation of performance of the coating materials. In spite of this, the diffraction

efficiency of the grating coated with SiC/W/Ir/Ti multilayer is more than 10% in the 55-100 nm region. This value seems to be satisfactory for practical use in VUV range. Further analysis and experiments are necessary to find the factors that affect the efficiency and to obtain higher efficiency.



Fig. 1. AFM-measured profile of the grating after SiC/W/Ir/Ti multilayer deposition.



Fig. 2. Measured and calculated –1st order efficiency spectra of the gratings coated with three kinds of materials (G1#: SiC/W/Ir/Ti, red squares; G8#: SiC, green circles; G15#: Ir, blue upper triangles).

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#### BL7B

## **Development and Verification of CLASP Polarimeter**

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We developed а polarimeter for the "Chromospheric Lyman-Alpha Spectro-Polarimeter (CLASP)" rocket experiment, which is an international project in collaboration with solar researchers in U.S., Spain, Norway, and Japan. CLASP will detect the weak linear polarization signal produced by the Hanle effect [1] and directly measure the chromospheric magnetic field using Lyman-alpha emission line (121.567 nm) for the first time. Since no practical polarimeter for Lyman-alpha has been developed, we designed and developed the polarimeter, and verified its system performance with an engineering model.

To measure the linear polarization with a high accuracy of 0.1%, we will take the spectra of two orthogonal polarization states simultaneously with a rotating half waveplate and two MgF<sub>2</sub> plates at Brewster's angle. The first MgF<sub>2</sub> plate ("beam splitter" in Fig.1) is a polarization beam splitter that reflects pure s-polarized light to Channel 1. The transmitted light is purified to be p-polarized by the second MgF<sub>2</sub> plate ("polarization analyzer" in Fig.1) and reflected to Channel 2. The rotating waveplate allows measurement of any direction of linear polarization with the fixed beam splitter.

Based on the measurements of reflectivity, transmissivity, and retardation for MgF<sub>2</sub> plate performed in 2009 at UVSOR, components of the polarimeter are designed as follows: The half waveplate is composed by stacking two MgF<sub>2</sub> plates with slightly different thicknesses (14.7 µm) in such a configuration that the slow axes are perpendicular to each other. The beam splitter is a 2-mm thickness MgF<sub>2</sub> plate with its slow axis parallel to the s-polarized light. The reflectivity for s-polarization is 22% and the transmissivity for p-polarization is 78%. The polarization analyzer at Channel 2 is a 15-mm thickness MgF<sub>2</sub> plate with its slow axis parallel to the p-polarized light, i.e., perpendicular to that of the beam splitter.

Using these components described above, the engineering model of the CLASP polarimeter was developed as shown in Fig. 1. By using a pair of autocollimated theodolites, we confirmed that both the beam splitter and the polarization analyzer were mounted at the Brewster's angle (59 degree) to the beam within 3 arcmin accuracy.

The engineering model of the CLASP polarimeter was tested with the synchrotron radiation at the UVSOR BL7B beamline. We input a highly linearly polarized light to the polarimeter and observed the output signal at both channels with a CCD camera. The expected results of the designed polarimeter are the followings: (1) the output signals show a  $\cos 4\theta$ modulation with respect to the rotation angle of the waveplate, and (2) the phases of modulations at Channel 1 and Channel 2 are shifted by a half cycle each other, i.e., the maximum at Channel 1 corresponds to the minimum at Channel 2. We indeed observed such modulation patterns as shown in Fig. 2, and verified that the polarimeter for CLASP has been developed.

We note that the wavelength of BL7B beam was calibrated with the absorption lines of molecular oxygen (O<sub>2</sub>) around Lyman-alpha (121.567 nm), at a pressure of 0.3 Torr and with a beam line slit width of 50 µm. This method can determine the wavelength within 0.1 nm accuracy.



Fig. 1. Photo of the polarimeter engineering model



Fig. 2. Observed modulation patterns in Channel 1 (black) and Channel 2 (orange).

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