Activities of the Evaluation Beamline for Soft X-Ray Optical Elements (BL-11)

Takashi Imazono,¹ Kazuo Sano,² and Masato Koike¹

 Quantum Beam Science Directorate, Japan Atomic Energy Agency (QuBS/JAEA), 8-1-7 Umemidai, Kizugawa, Kyoto 619-0215, Japan

2) Shimadzu Emit Co., Ltd., 2-5-23 Kitahama, Chuo-ku, Osaka 541-0041, Japan

Abstract

We have been developing soft x-ray optical devices such as multilayer mirrors, gratings including multilayer-coated gratings, and polarizing elements. Ultra-thin films and multilayers have been designed and deposited by ion-beam sputtering (IBS) technique. The optical characteristics of these devices have been evaluated by using the BL-11 beamline at SR Center of Ritsumeikan University, which is an evaluation beamline for soft x-ray elements and gives us intense soft x-ray radiations form 0.65 nm to 25 nm by using two types of grazing incidence Monk-Gillieson monochromators. Most recent activities are as follows: development of multilayer gratings in the keV region; fabrication and evaluation of spherical normal-incidence multilayer mirrors for a soft x-ray laser, reflection mapping of multilayer mirrors with the contaminated surface used in EUV lithography instruments; determination of Stokes parameters of light by complete polarization analysis; comparative measurement of the diffraction efficiencies of the 1st and higher diffraction orders of mechanically-ruled and holographic gratings. Here, we report on establishment of a beam intensity monitor at just after the refocusing toroidal mirror of the beamline which is contribute to enhance measurement accuracy and improvements of the Soft X-Ray Polarimeter and Ellipsometer (SXPE), which is an apparatus used for the evaluation of polarizing elements and the polarization state of the incident light.

1. Introduction

Since 2000, the Evaluation Beamline for Soft X-Ray Optical Elements (BL-11)¹⁻³⁾ at SR Center of Ritsumeikan University has been operated to measure the wavelength and angler characteristics of the absolute reflectivity (or diffraction efficiency) of soft x-ray optical devices such as ultra-thin films, multilayer mirrors, gratings including multilayer-coated gratings, and polarizing elements. It provides intense soft x-ray radiations form 0.65 nm to 25 nm by two types of grazing incidence Monk-Gillieson monochromators. One is a conventional type equipped with three varied-line-spacing plane gratings (VLSPG's), allowing a choice of the included angle of either $88.0^{\circ} \times 2$ or $86.0^{\circ} \times 2$.⁴⁾ The other is a new type that employs a scanning mechanism based on surface normal rotation (SNR) of the grating.⁵⁾ This scanning scheme was proposed by Hettrick⁶⁾ in 1992 taking advantage of high grating diffraction efficiency in the manner of conical diffraction. The practical advantage of this scheme lies in its mechanical simplicity, requiring only a simple rotation of the grating around its normal for wavelength scanning.

It is necessary to calibrate wavelength of the beamline accurately to measure the reflectivity and diffraction efficiency as well as to evaluate other characters of the optical elements as the function of wavelength. For the wavelength calibration it is possible to utilize the positions of absorption edges (e.g., C-*K*, B-*K*, Si-*L*, and Al-*L*) of the elements usually used as thin film low-pass filters. Also for the case of the evaluation of Mo/Si multilayer mirrors applied for EUV lithography the valid wavelength range is limited to a very narrow range around 13 nm and absorption spectra of Kr gas $(3d_{5/2}^{-1} \rightarrow 5p (91.2 \text{ eV}))^{7)}$ may provide sufficient information which is enable to calibrate wavelength in the narrow spectral range.

On the other hand in the evaluation experiments of reflectivity and diffraction efficiency, either the intensity of zero-th order light of the monochromator or the ring current of the storage ring was utilized for the normalization of the beam intensity of the first order light which is incident to the reflectometer. These methods are valid for a measurement held in a relatively short time period. As well known, ring current in the storage ring decreases proportional to the passed time, however, the beam intensity is not reduce proportional to the time. Even though the beam intensity of the first order light introduced into the reflectometer has a collation with that of the zero-th order light but it is not a linear relation. To normalized the intensity of the incident beam it is appropriate to measure the intensity of the monochromatized first order light at a position after the exit slit at real time.

Furthermore, the source of the BL-11 beamline is a bending magnet and the generated light is polarized almost horizontally. Reflectivity and other properties of an optical element depend on the degree of linear polarization of the incidence beam and this phenomenon

becomes significant that the possibility of an under estimation of the optical performance especially at a grazing incidence condition. Therefore it is extremely important to disclose the polarization condition of the incidence beam in advance to measurements.

This report describes two major improvements of the beamline for the purpose of accurate evaluation of the performance of the optical elements: (1) the introduction of a beam intensity monitor in just front of the reflectometer (at the down side of the toroidal refocus mirror); (2) the improvements of the instrument to evaluate the polarization state (Soft X-ray Polarimeter and Ellipsometer for complete polarization analysis, SXPE⁸⁻¹¹). Also we describe a re-evaluation of degree of linear polarization of the beamline by use of Figure 1 shows a diagrammatic layout of BL-11 including the current soft x-ray



Fig. 1 Top and side views of the BL-11 and the improved SXPE (iSXPE). The chamber of SXPE has been rotated by 90°. BM1 means a newly developed beam monitor unit.



Fig. 2 Photograph of the BL-11 and iSXPE, as of 30 March, 2011.



Fig. 3 Schematics of a newly fabricated beam intensity monitor unit (a) and its main parts (b).

polarization analysis instrument (iSXPE) described in the third section. Figure 2 shows the photograph of the BL-11 and iSXPE.

2. Development of a beam intensity monitor

The beam intensity monitor (BM0) which has been used to normalize the incident beam intensity, i.e., the beam intensity of the first order light, for the reflectometer utilizes the current value of photoelectron yield obtained by a gold coated mesh irradiated by the zero-th order light in the monochromator. Recently we introduced a new beam intensity monitor (BM1) which can measure the intensity of the first order light in just front of the reflectmeter (at the down side of the toroidal refocus mirror). Hitherto a small turbo molecular pump (PT-50 compatible, Shimadzu Corp.) was located at this position with a T-style branch pipe. Therefore it is required that the newly fabricated beam intensity monitor unit is coexisted with the TMP. Figure 3 shows the blueprint of the fabricated beam intensity monitor unit. The measurement principle is the same as that of BM0 and based on



Fig. 4 Correlation between signal of photodiode and the beam monitor intensity, in voltage. The correlation efficient has been estimated to be 0.989.

the current value of photoelectron yield obtained by a gold-coated tungsten mesh (W-468098, Nilaco Corp.) irradiated by the first order light. The mesh is installed at the top of a linear feed through with the aid of a connection adapter in the ultra high vacuum atmosphere. Normally the mesh is not grounded. The stroke length of the linear feed through is 30 mm and it makes possible to realize the conditions of the mesh is irradiated or not by the beam.

Figure 4 shows the correlation between signal of photodiode and the beam intensity obtained by beam monitor. The correlation efficient has been estimated to be 0.989. It indicates that they have strong correlation and the new beam intensity monitor provides accurate real time measurement of the incidence beam intensity.

3. Improvement of SXPE (iSXPE) and linear polarization measurement

Reflectivity depends on the polarization state, i.e., the linear polarization degree, P_L , and azimuth angle of the major axis of the polarization ellipse, δ , of the incident light. In particular, it would be possible to underestimate the reflectivity at the angle of incidence |close to the pseudo-Brewster angle, which is approximately 45° in the soft x-ray (SX) region. Therefore, it is important to evaluate the polarization state of the incident light in advance of reflection measurements.



(a)



(b)

(c)

Fig. 5 Projection drawing (a) and photograph of the improved internal mechanism (b), and photograph of the vacuum chamber rotated by 90° and the control panel (c), of iSXPE.

Symbol of axis	Annotation of axis	Operation range	Resolution per pulse	Motorized stage [*]
χ	Azimuth angle of P	-90 ~ +360°	0.00125°	KS402-100-L
φ	Incident angle of P	$-10 \sim +70^{\circ}$	0.0003°	U11297 (KS401-40)
Н	Height of P	$-5 \sim +5 \text{ mm}$	0.0005 mm	U11298 (KS101-15)
ψ	Arm	-5 ~ +120°	0.001°	KS402-100-L
η	Azimuth angle of A	$-90 \sim +360^{\circ}$	0.00125°	U10698 (KS402-75)
ω	Incident angle of A	$-10 \sim +90^{\circ}$	0.0003°	U11297 (KS401-40)
Х	Height of A	$-5 \sim +5 \text{ mm}$	0.0005 mm	U11298 (KS101-15)
θ	Detection angle	$-5 \sim +125^{\circ}$	0.0003°	U11297 (KS401-40)
Т	Slit	$-20 \sim +20^{\circ}$	0.0015°	U12252 (KRW04360)

Table 1 Details of the motorized stages assigned to the drive shafts of the improved SXPE (iSXPE).

* The basic model names of the motorized stages, made by Suruga Seiki Co., Ltd., are shown in the parentheses.

In our previous study, a dedicated apparatus for complete polarization analysis (Soft X-Ray Polarimeter and Ellipsometer: SXPE) had been developed.⁸⁻¹¹⁾ It was installed at the end station of the soft x-ray beamline (BL-11). It was successful that the linear polarization degree in the energy range of 12.5-14.8 nm was determined quantitatively along with the evaluation of the polarization characteristics of the polarizing elements, the reflectivity and polarizance of the SX polarizer. Unfortunately, there are some problems on SXPE as the following: the difficulty of the alignment; anxiety of the shortage of torque of rotational stages comparing with the imposed massive load; limitation of the size of a sample (polarizing element) up to $15 \times 15 \times 5t \text{ mm}^3$. To solve the above issues, the SXPE has been improved.

Firstly, the vacuum chamber of the SXPE has been rotated by 90° (see Figs. 1, 2, and 5). Also, a new drive shaft, T, was installed to change the position of a slit just before the detector, D, where T-axis had been used to change the horizontal position of the internal mechanism of the SXPE so far. The slit width and height are 0.2 mm and 8 mm, respectively. This slit is very useful to determine the incident angle and height position of optical component, and the optical axis. By this improvement, it has been able to be easily to adjust the position of the internal mechanism in the improved SXPE (iSXPE) and the optical axis.

The inside mechanism of the SXPE has also been modified extensively to rotate the vacuum chamber by 90°. As shown in Fig. 5, it consists of nine motorized stages and a large and a small rectangular frames supported by a U-shaped basement made of aluminum alloy with two bearings (φ 20 mm and φ 12 mm). In this figure, symbols of χ , φ , H, ψ , η , ω , X, θ , and T mean the azimuth angle of P, the incident angle of P, the height position of P, the arm, the azimuth angle of A, the incident angle of A, the height position of A, the detection angle, and the slit, respectively. The six of them, φ , H, ω , X, θ , and T, have been replaced

into more compact and light motorized stages along with the drastic improvements of the rectangular frames and the U-shaped basement. The details of the motorized stages assigned to the drive shafts of the iSXPE are shown in Table 1, where all motorized stages have been made by Suruga Seiki Co., Ltd. These drive shafts can be controlled by an exclusive program⁸⁾ based on LabVIEW[®] (National Instruments). By these improvements, it has been

able to reduce load by over at least 30%. In addition, as shown in Fig. 6, by reducing the number of wires for three sensors (CWLS, CCWLS, and ORG) and power supply $(DC\pm 5V)$, their load reduction has been achieved. The connector pin assignment itself of a 30-pin electrical feedthrough is not changed compared to before but the connector label of A1, C2, and C3 have been changed. The connector labels of A1, A2, A3, B1, B2, B3, C1, C2, and C3 are assigned to the axes of ψ , η , T, ω , θ , X, ϕ , H, and χ , respectively. Furthermore, the position of counterweight made of a stainless steel has been improved.

The linear stage, H (or X), to change the height position of P (or A) was connected just below the rotary stage, φ (or ω), to change the incident angle so far. In this case, it was impossible to coincide the height positions of P (or A), the center of the rotation axis of the incident angle, φ (or ω), and of the arm, ψ (or the detection angle, θ). Therefore, as shown in Fig. 5, the H (or X) stage and the φ (or ω) stage have been exchanged. It allows us to adjust the angle of incidence and the height position of the sample (polarizing element) by using the variable slit more accurately.





Sumbol	Assignment	Color	Connector label			
Symbol	Assignment	Color	index	Α	В	С
Α	Motor lead (Black)	Black				
В	Motor lead (Green)	Green				
С	Motor lead (Orange)	Orange				
D	Motor lead (Red)	Red				
E	Motor lead (Blue)	Blue	1			
F	CWLS Out (Brown)	N/A	· ·	Ψ	ω	φ
G	CCWLS Out (Yellow	CCWLS Out (Yellow N/A				
Н	ORG In (Gray)	N/A				
J	DC5V (+) (Violet)	N/A				
K	DC5V (-) (White)	N/A				
L	Motor lead (Black)	Black				
М	Motor lead (Green)	Green				
N	Motor lead (Orange)	Orange				
Р	Motor lead (Red)	Red				
R	Motor lead (Blue)	Blue	2		0	тт
S	CWLS Out (Brown)	N/A	2	η	9	п
Т	CCWLS Out (Yellow	N/A				
U	ORG In (Gray)	N/A				
V	DC5V (+) (Violet)	N/A				
W	DC5V (-) (White)	N/A	1			
Х	Motor lead (Black)	Black				
Y	Motor lead (Green)	Green				
Z	Motor lead (Orange)	Orange				
а	Motor lead (Red)	Red				
b	Motor lead (Blue)	Blue	3	т	х	~
С	CWLS Out (Brown)	N/A	Ĭ			λ
d	CCWLS Out (Yellow	N/A				
е	ORG In (Gray)	N/A	4			
f	DC5V (+) (Violet)	N/A	.			
g	DC5V (-) (White)	N/A				

Fig. 6 Connector pin assignment of a 30-pin electrical feedthrough. The connector labels of A1, A2, A3, B1, B2, B3, C1, C2, and C3 are assigned to the axes of ψ , η , T, ω , θ , X, ϕ , H, and χ , respectively. The connector labels of A1, C2, and C3 have been changed compared to before.

The iSXPE has been designed to be able to mount either a micro-channel plate (MCP) with a 18-mm outer diameter and a 14.5-mm effective detection area (F4655, Hamamatsu Photonics K.K.) or a silicon p-n junction photodiode (PD) with the sensitive area of a 10-mm square (AXUV100-Series, IRD Inc.). The PD makes it easy to detect the optical axis at the alignment. It is because the PD has a sensitivity to visible light, where BL-11 provides not only soft x-ray radiations but also very intense white light as the zero-th order light. In the experiment described below, a PD with directly deposited filter of Si/Zr (AXUV100Si/Zi, IRD Inc.) was used as the detector.

Finally, the sample holder of P (or A) has been modified to be able to mount a sample having a size of up to 2" diam. \times 10t mm (or 50 \times 30 \times 10t mm³), as seen in Figs. 5(a) and 5(b). It anticipates that characteristics of versatile optical elements can be measured by using the iSXPE.

As a performance test of iSXPE, reflection and linear polarization measurement were demonstrated by using two Mo/Si multilayer mirrors as a polarizer, P, and an analyzer. They were designed for the polarizers at the wavelength of 13.9 nm, where coherent x-ray radiation is generated by plasma excitation, and fabricated by an ion beam sputtering method. For the design period length of 10.0 nm the measured values of two multilayers by x-ray diffractomater (SXL2000, Rigaku Co.) were 9.9915 nm (or 10.005 nm) for the polarizer (or analyzer).

The experiment using iSXPE equipped with two Mo/Si polarizers was carried out at BL-11. The beam-shaping apertures, A, in Fig. 1, were set to 6 mm (H) \times 3 mm (V). The acceptance angles were estimated to be 3 mrad (H) \times 1.5 mrad (V). The conventional grazing incidence Monk-Gillieson monochromator with a laminar-type varied-line-spacing holographic gratings G1 (300 lines/mm) and a spherical mirror M5 was used at the included angle of 172°, where it covers a wavelength range of 4.5-25 nm.⁴⁾ The wavelength of the incident light was set at 13.9 nm. The slit widths of the entrance and exit slits were set to 200 µm and 220 µm, respectively, and the slit heights of both slits were set to 3.6mm. The resolution was estimated to be a couple of hundred. A thin Si film of ~0.5 µm thickness, indicated by F in Fig. 1, was used to reduce unwanted higher orders light. The refocusing toroidal mirror of M7 was used. The beam size at the sample position was restricted to 2 × 2 mm² or narrow by a four-quadrant slit, FS, set just in front of the iSXPE chamber. The beam intensity monitor, BM1, described in the previous section, was used for the normalization of the incident beam in the polarization measurement.

Figure 7 shows the reflection profiles for s- and p-polarization components (R_s and R_p) of P and A measured by the iSXPE, as a function of the angle of incidence. The vertical axis is in logarithmic scale. The reflection curves obtained by A almost agree with those of P,

where the maximum of R_s (or R_p) of A is 48.3% (or 2.3%) at the angle of incidence of 43.25°.

Figure 8 shows the azimuth dependences of the intensities of the light reflected by only the analyzer, only the polarizer, and the analyzer after reflected by the polarizer. ^{8, 11)} The



Fig. 7 Respective reflection profiles for s- and p- polarization components (R_s and R_p) of the Mo/Si multilayer polarizer and analyzer measured by iSXPE, as a function of the angle of incidence. The vertical axis is in logarithmic scale.



Fig. 8 Azimuth angle dependencies of the intensities of the light reflected by only the analyzer, only the polarizer, and the analyzer after reflected by the polarizer, at the angles of incidence of 43.25°. The vertical axis is in logarithmic scale. The results of the curve-fitting analysis are also shown by solid lines.

angles of incidence of the polarizer and analyzer were set to 43.25° where the maximum reflectivities were obtained. The azimuth angles η and χ are measured clockwise to an observer from the horizontal plane. The vertical axis is in logarithmic scale. Solid lines in this figure stand for the results of the curve-fitting analysis^{8, 11)} and they are in good agreement with the respective measured data. Consequently, the polarizance, *Z*, defined as $(R_s - R_p)/(R_s + R_p)$, the linear polarization degree, *P*_L, and the azimuth angle of the major axis of the polarization ellipse, δ , of the incident light have been determined to be *Z* = 99.6% for A (or 99.9% for P) and *P*_L = 91%, and $\delta = -1.3^\circ$, respectively. The values of *P*_L and δ are in good agreement with our previous results.^{10, 11}

4. Summary

In this study we newly developed a beam intensity monitor and introduced in just front of the reflectometer. The correlation coefficient with the beam intensity measured by the detector (a photo diode) in the reflectometer is 0.989 and it is found that it works as an efficient beam intensity monitor. Also we improved the soft x-ray polarization analysis instrument and performed the polarization measurement by use of Mo/Si multilayer polarizers. As the result it was found that the degree of linear polarization is 91% at 13.9 nm. This research would enhance the accuracy of evaluations of optical elements at the BL-11.

Acknowledgement

This study has been conducted as part of a project of Collaborative Development of Innovative SEEDs (Practicability verification stage) by Japan Science and Technology Agency (JST). The collaborators are Tohoku Univ., JEOL Ltd., Shimadzu Corp., and JAEA.

References

- M. Koike, K. Sano, Y. Harada, O. Yoda, M. Ishino, K. Tamura, K. Yamashita, N. Moriya, H. Sasai, M. Jinno, and T. Namioka, *Proc. SPIE* 4782, 300-307 (2002).
- M. Koike, K. Sano, O. Yoda, Y. Harada, M. Ishino, N. Moriya, H. Sasai, H. Takenaka, E. Gullikson, S. Mrowka, M. Jinno, Y. Ueno, J. H. Underwood, and T. Namioka, *Rev. Sci. Instrum.*, 73, 1541-1544 (2002).
- (3) K. Sano, T. Imazono, M. Jinno, H. Sasai, Y. Harada, N. Moriya, M. Ishino, and M. Koike, *Memoirs* of *The SR Center*, *Ritsumeikan Univ.*, 9, 67-78, (2007).

- (4) M. Koike and T. Namioka, Appl. Opt., 36, 6308-6318 (1997).
- (5) M. Koike and T. Namioka, *Appl. Opt.*, **41**, 245-257 (2002).
- (6) M. C. Hettrick, Appl. Opt., 31, 7174-7178 (1992).
- (7) G. C. King, M. Tronc, F. H. Read, and R. C. Bradford, J. Phys. B., 10, 2479-2490 (1977).
- (8) T. Imazono, K. Sano, Y. Suzuki, T. Kawachi, and M. Koike, *Rev. Sci. Instrum.*, 80, 085109 (8 pages) (2009).
- (9) T. Imazono, Y. Suzuki, K. Sano, and M. Koike, *Spectrochimica Acta B.* **65**, 147-151 (2010).
- (10) T. Imazono, K. Sano, Y. Suzuki, T. Kawachi, and M. Koike, *AIP Conf. Proc.*, **1234**, 347-350 (2010).
- (11) T. Imazono, K. Sano, Y. Suzuki, T. Kawachi, and M. Koike, *Memoirs* of *The SR Center*, *Ritsumeikan Univ.*, **12**, 87-100 (2010).