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Multilayer polarization elements and their applications to polarimetric studies in vacuum ultraviolet and soft X-ray regions

WATANABE Makoto[1,*] HATANO Tadashi[2] SAITO Katsuhiko[2] HU Weibing[3] EJIMA Takeo[2] TSURU Toshihide[2] TAKAHASHI Masahiko[2] KIMURA Hiroaki[4] HIRONO Toko[4] WANG Zhangshan[5] CUI Mingqi[6] YAMAMOTO Masaki[2] YANAGIHARA Mihiro[2]

[1] *Shanghai Dianji University, Shanghai 200240, China; Institute of Composite Materials, Shanghai Jiaotong University, China and Venture Business Laboratory, Saga University, Japan*

[2] *Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, Sendai 980-8577, Japan*

[3] *Changchun Institute of Optics, Fine Mechanics and Physics, Changchun 130033, China*

[4] *Japan Synchrotron Radiation Research Institute, SPring-8, Hyogo 679-5198, Japan*

[5] *Institute of Precision Optical Engineering, Tongji University, Shanghai 200092, China*

[6] *BSRF, Institute of High Energy Physics, Beijing 100049, China*

Abstract Multilayer polarization elements and their applications to polarimetric studies in 20~400 eV region are mainly reviewed. General principle of selecting material combinations to get high linear polarizance multilayers of reflection type is given with practical examples, with periodic or non–periodic layer structures depending on the usage. Transmission type is introduced as linear polarizer and phase shifter. Their applications include polarization diagnosis of laboratory optical systems and synchrotron radiation beamlines of linear and circular polarization, magnetic rotation experiments such as Faraday rotation and magnetic Kerr rotation on magnetic films and multilayers, and ellipsometry to measure optical constants of thin films precisely. Polarization analysis of soft X-ray fluorescence using multilayer-coated grating is also mentioned. Finally this review is summarized with outlook of further developments.

Key words Vacuum ultraviolet, Soft X-ray, Synchrotron radiation, Multilayer, Polarizer, Phase shifter

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1 Introduction

Polarization of light is a character of great advantages that is utilized widely in optical science and technology in infrared, visible and ultraviolet regions. In vacuum ultraviolet (VUV) and soft X-ray (SX) regions, synchrotron radiation (SR) has manifested itself as an excellent polarized light source[1-5]. By the use of SR, polarimetric experiments such as optical studies of anisotropic crystals, magneto-optical experiments, ellipsometry, etc., have been developed. In the experiments, good polarization state of SR beamlines must be guaranteed by checking their polarization characteristics. Therefore, polarization elements such as linear polarizers and phase shifters are inevitable. From the visible to near VUV regions, polarizers are made from isotropic and anisotropic (dichroic, birefringent) transparent materials[6-8]. Phase shifters made from isotropic transparent materials operated under alternating pressure (photoelastic modulator) are used, too[8]. In deep VUV and SX regions, because of serious absorption, transparent polarizers cannot be applied and polarization elements of reflection or transmission type using absorbing materials were developed, and the developments are

* Corresponding author. *E-mail address:* watanabemakoto@163.com
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still under way. Therefore polarimetric experiments are not fully opened. In X-ray region, polarizers made of crystals have been established[9,10], and are popularly used.

In deep VUV and SX regions, a reflection type linear polarizer utilizes the difference between R_s and R_p (s- and p-reflectance), where s or p denotes electric field that is perpendicular or parallel to the plane of incidence of the light. From the Fresnel equation, s-reflectance is generally higher than p-reflectance for the same angle of incidence at the same wavelength. The exceptions are the reflections at 0º (exact normal incidence) and 90º (grazing incidence limit), in which both s- and p-reflectance are equal. The ratio of p-reflectance to s-reflectance is 0 at a Brewster angle for a transparent material. For an absorbing material, the ratio is the smallest at the angle called quasi Brewster angle. In deep VUV and SX regions, refractive indices of all materials are nearly equal to 1, so that the quasi Brewster angle is around 45º. Therefore around 45º incidence, high polarizance $(R_s–R_p)/(R_s+R_p)$ can be obtained. However, the reflectance is low, because of refractive indices $n≈1$ and small extinction coefficients k for short wavelengths.

There are two approaches to overcome the difficulty of low reflectance. One is to employ the reflection at incidence of grazing angles, at the cost of high polarizance, though. Multi-mirror reflectors with three- or four-time reflection enhance the polarizance[8,11,12]. In the short wavelength region, the angle of incidence should be large to obtain high reflectance, and the difference between s- and p-reflectance becomes small. Therefore useful wavelength range of the multi-mirror reflectors is fairly limited.

The other approach is to utilize multilayers, two materials piled periodically or non periodically, in which the wavelets reflected at each boundary interfere in phase with each other[13,14]. Multilayer polarizers are usable in wide wavelength range by choosing layer periodicity and material combinations. The useful wavelength range of a periodic multilayer is not wide, but this disadvantage can be overcome as described later.

In general, phases of s- and p-reflected light (δ_s and δ_p) differ from each other, so that mirrors can be used as phase shifters. In transparent materials, the phase difference ($\Delta=\delta_p–\delta_s$, phase retardation) suddenly changes by 180º when the angle of incidence exceeds the Brewster angle. In absorbing materials, the phase difference gradually changes by 180º when the angle of incidence exceeds the quasi Brewster angle. This effect can be used for phase shifter of reflection type, with different intensity ratio of s- and p-reflected light before and after reflection. When multi-mirror reflectors are used to change the phase retardation by 90º, it works as a quarter-wave plate[8,11]. In laboratory, linearly polarized light is obtained from unpolarized light with reflection type polarizers mentioned above, and circularly polarized light has been obtained[8]. The helicity is fixed (left or right) with a fixed azimuth angle of phase shifters.

Another kind of linear polarizer is transmission type. It is also used near the quasi Brewster angle, where the p-transmitted light is more intense than s-transmitted light. For a monolayer, the intensity difference between p- and s-transmitted light is small, and transmission multilayer polariazers are required to enhance the difference. The principle is similar to pile-of-plates polarizer[8]. The transmission type doesn’t need a change in optical path, though the output intensity is smaller than reflection type for the same polarizance. The phases are different between s- and p-transmitted light in general. The phase retardation changes remarkably, when angle of incidence exceeds the Bragg angle. The amount of retardation of each layer pair is not large. When the number of layer pair is chosen to make a 90º-phase retardation, the multilayer can be used as a quarter-wave plate.

Multilayers can be used as linear polarizers or phase shifters. Designed flexibly with respect to the wavelength region, they have potentials for versatile applications. A recent review was given by Schaefers, BESSY, Germany[15] on multilayers and their applications to polarimetric studies in deep VUV and SX regions mainly from 400 to 800 eV. This paper reviews mainly in the 20~400 eV region, referring the experimental results obtained in Japan and China. General principle of selecting material combinations to get high polarizance multilayer is given with practical examples. Estimation for the polarization state of light from a grating monochromator system of laboratory is described. Diagnoses of SR beamlines are given. Good polarization state of SR beamlines has to be kept, because optical elements have their own polarization effect and misalignment causes deterioration of the polarization characteristics. Magneto-optical rotation experiments on magnetic single films and multilayers, ellipsometry to measure optical constants of thin films, and polarization analysis of SX fluorescence using multilayer-coated grating, are described.

2 Multilayer polarization elements

Multilayers are usually fabricated by magnetron sputtering or ion beam sputtering on substrates of super-polished Si wafer, glass or quartz. For transmission type, multilayer is self-standing or deposited on transparent thin film such as Si_3N_4. The periodicity is checked by X-ray diffraction.

2.1 Reflection polarizers

To obtain reflection polarizer with high polarizance and reflectance, general principle to select material combinations of multilayers was studied[16]. First, as the polarizance is inversely proportional to the sum of k of two materials, k of both materials should be small. The second issue is to obtain high reflectance. To increase the number of reflections at the layer boundaries, k of both materials should be small again and to increase the reflectance at each layer boundary, the difference in n between the materials should be large[17].

Fig.1 shows plots of optical constants of several materials for He I (58.4 nm, 21.2 eV) and He II (30.4 nm, 40.8 eV) resonance lines in n-k plane which are available in Ref.[18]. For He I line, a layer pair of Si and Mg is the best for polarizer, because of their considerably smaller k value than other popular materials. For He II line, a layer pair of SiC and Mg is the best. Fig.2 shows incident angle dependence of polarizance and s-reflectance of SiC/Mg multilayer for He II line. The maximum polarizance is 0.98 at 40º incidence with a reflectance of 41%. For polarizer in 47~56 eV region, a combination of Al and YB_6 was chosen. Fig.3 shows s-reflectance and polarizance spectra of the polarizer of this layer pair for 41º incidence in 47~56 eV region, where the polarizance is higher than 0.96 and changes little, while the s-reflectance shows a peak (Bragg peak) up to 45%. In 80~150 eV region, many multilayers based on Mo/Si and other material combinations can be used for multilayer polarizers[15,19]. Above 150 eV, Mo/B_4C multilayer is often used, for which a calculated polarizance of 0.995 was obtained at angles of incidence between 44º and 47º in 175~190 eV region[20]. Around 400 eV, Sc/Cr polarizers are developed[15]. In the regions of L edges of Fe, Co and Ni (700~900 eV), W/B_4C multilayer are employed[15,21].

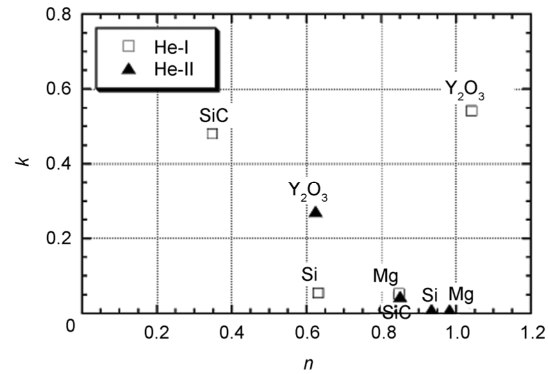

Fig.1 Plots of optical constants of typical materials for He I (58.4 nm) and He II (30.4 nm) lines on n-k plane[16].

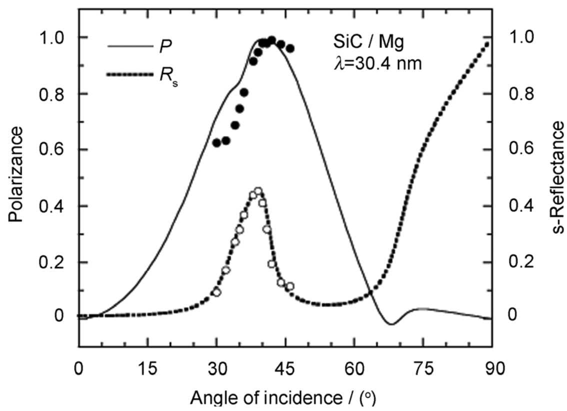

Fig.2 Incident angle dependence of polarizance and s-reflectance of SiC/Mg multilayer for He II line (30.4 nm)[16].

For periodic multilayer polarizers of reflection type, wavelength regions of high polarizance are wider than the width of their reflectivity peaks as seen in Fig.3. Therefore for polarimetric studies in wide regions, it is required to widen the region of high reflectance with high polarizance. There are several methods to widen it. The first is to change angle of incidence to the multilayers to choose high reflectance peak at different wavelengths. A simple way is to use one multilayer[22], and a complicated way is two multilayers[23], the mechanism of which is similar to that of double crystal monochromators. The second method is to utilize multilayers with laterally graded period[21]. The multilayer is shifted laterally to choose high reflectance peak at different wavelengths. The angle of incidence is fixed at 45°. The third one is to use non periodic multilayers of broad band, in which thickness of every layer differs and thickness of each layer pair distributes around an average value. Layer pairs of different thicknesses are situated randomly, rather than monotonously in order of thickness, to provide broad band characteristics[24]. As an example, s-reflectance and polarizance of Mo/Si multilayers are shown in Fig.4. It is clearly seen that the s-reflectance is high in a wide region having a flat top and that high polarizance is obtained in the same region. The measured s-reflectance and polarizance designed for 15~17 nm region are over 37% and 0.98, respectively. Those for 14~18 nm region are over 35% and 0.98, and for 13~19 nm, over 30% and 0.96. The range of incidence angle that gives almost the same polarizance is over 10°[24]. The Mo/Y combination has been used for 8.5~11.7 nm region[25]. With these polarizers, measurements can be made without rotation or lateral translation to choose appropriate angle or position. The linear multilayer polarizers of reflection type are widely used for polarimetric studies as polarization analyzer as described later.

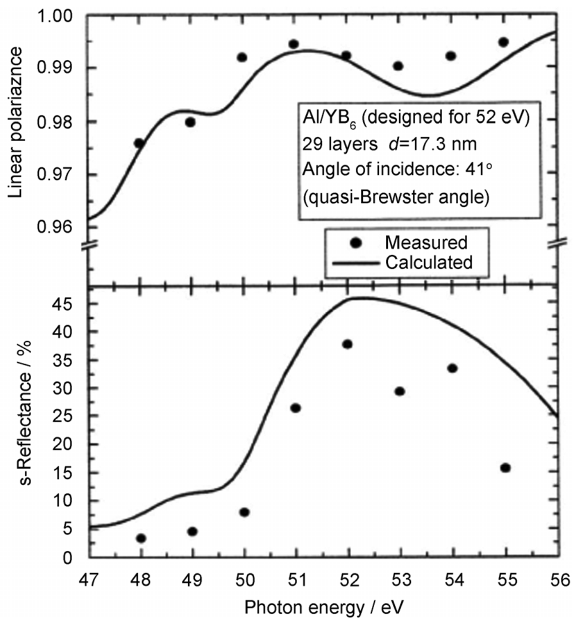

Fig.3 Polarizance and s-reflectance spectra of Al/YB_6 multilayer at angle of incidence of 41°.

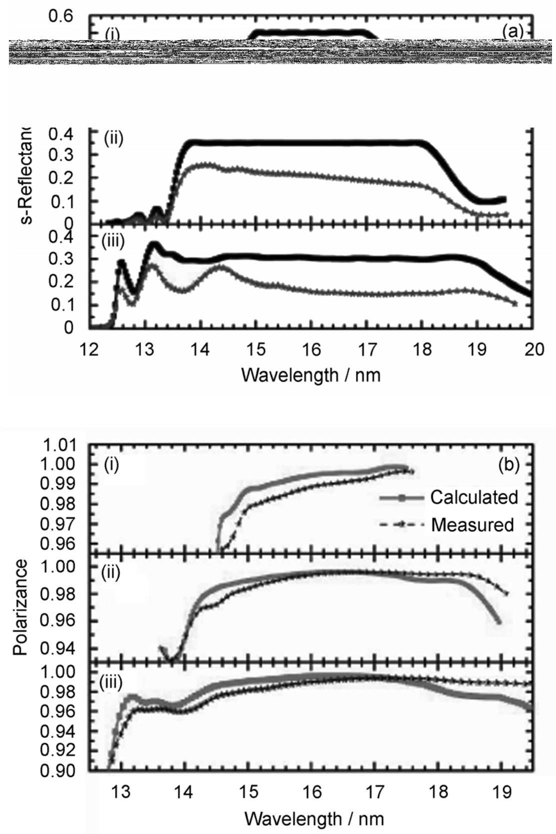

Fig.4 Calculated and measured s- and p-reflectivity (a) and polarizance spectra of non periodic Mo/Si multilayer polarizers designed to have broad band over wavelength regions of (i) 15~17 nm, (ii) 14~18 nm and (iii) 13~19 nm at 40° incidence (b)[24].

2.2 Transmission polarizers and phase shifters

An Al/YB_6 self-standing transmission multilayer was fabricated as linear polarizer in 55~69 eV region[26]. It was used to increase the degree of linear polarization of the light behind a monochromator. In Fig.5, polarizance $(T_p-T_s)/(T_p+T_s)$ (minus) was measured with 65 eV SR of two different polarization states. The two measured values are in good agreement. In comparison with calculated value of 0.94, the measured peak polarizance was 0.92, and the calculated and the measured transmittances were 7.9% and 3.4%, respectively[26,27]. For phase retardation $\delta_p-\delta_s$ of the transmission multilayer, the calculated peak value was 99°, but measured one was 68°. It can be used as a phase shifter[27]. A Mo/Si transmission multilayer was used to increase the degree of linear polarization of the light behind a monochromator in 80~100 eV region[26,28]. With a phase retardation of 90°, it can be used as a quarter-wave plate[28,29]. The phase difference is 180º, hence the use as a half-wave plate[30]. Around 400 eV, Sc/Cr transmission polarizer is used as a rotating analyzer[31], a phase shifter[15] and a quarter-wave plate[31,32]. In Fig.6, the polarizance and phase retardation of the quarter-wave plate are plotted against angle of incidence[32]. The phase retardation of 90° and polarizance of 0.19 at 59.77° incidence indicate that p-transmitted light is a little more intense than s-transmitted light. The p-transmittance is 0.4%. By the way, Fe film, with large Faraday rotation angle in the regions of its L edges, can be used to rotate the plane of electric field of light[15].

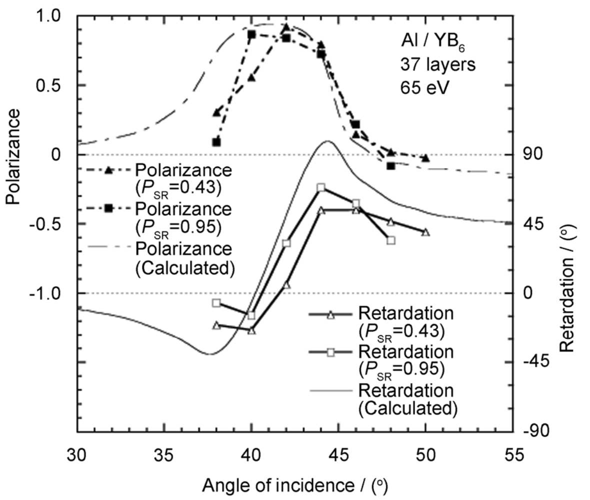

Fig.5 Calculated and measured polarizance and phase retardation of Al/YB_6 transmission multilayer at 65 eV[27].

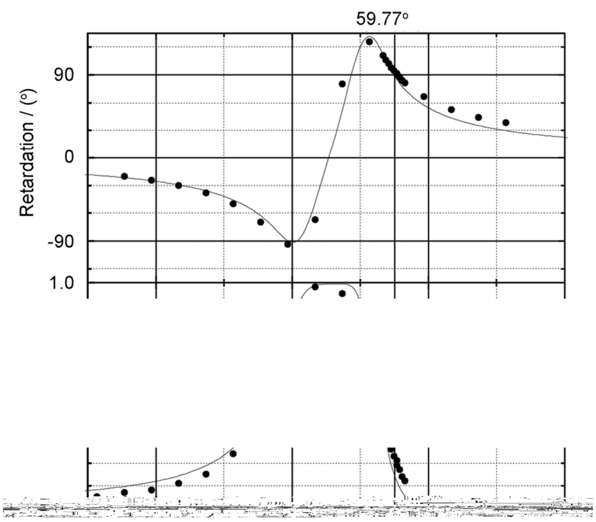

Fig.6 Polarizance and phase retardation of Sc/Cr transmission multilayer at 398.6 eV. •, measured data; —, fitting results[32].

3 Polarization diagnosis

3.1 Laboratory VUV system

Several polarization measurements using mirror polarizers have been made for laboratory VUV systems[8]. Introduced here is the polarization measurements using multilayer polarizers and photoelectron angular distribution measurement using He gas (Fig.7)[33]. The unpolarized light from a laboratory source is dispersed by a grating and refocused by a grazing incidence mirror. For polarization measurement, a rotating analyzer unit situated behind the refocusing mirror was used. The unit consists of a multilayer polarizer (analyzer) of reflection type and a detector. The unit was rotated as a whole around the optical axis of monochromatized light. The multilayers were made for He I and He II lines as described in §2.1. Fig.8 shows signals from the unit for He I line. The abscissa is rotation (azimuth) angle of the unit and the ordinate is relative intensity. Closed squares represent experimental results and a solid curve gives a fitting result by a sinusoidal curve. By finding amplitude and angle of the peak of the sinusoidal curve, it was found that the degree of linear polarization of monochromatized light was 0.68 and 0.33 for He-I and He-II lines, respectively. Here, the degree of linear polarization is defined as $(I_a - I_b)/(I_a + I_b)$, where I_a and I_b are intensities of major and minor axis components of dispersed light, respectively. The inclination angle of the major axis from the vertical plane was negligibly small. On the other hand, angular distribution of photoelectrons emitted from jet of He gas introduced in a measurement chamber was measured to check the polarization of the light. The degrees of linear polarization measured by both optical and photoelectron methods were in good agreement. It means that both measurements are consistent and the results are satisfactory. It was found that the present monochromator system has polarizing ability. It shall be remarked that the monochromatized unpolarized light is no more unpolarized. That is, the polarization characteristics of light source are not necessarily kept behind monochromator systems.

this purpose. The nominal circularly polarized light emerging from monochromator was converted to a combination of linearly polarized light and unpolarized light, which was observed like elliptically polarized light by rotation analyzer method. By analyzing signals from the rotating analyzer, the degree of circular polarization and inclination angle of polarization ellipse can be determined precisely. The results did not suggest a complete circular polarization. Whether this is due to misalignment or not is unclear at present.

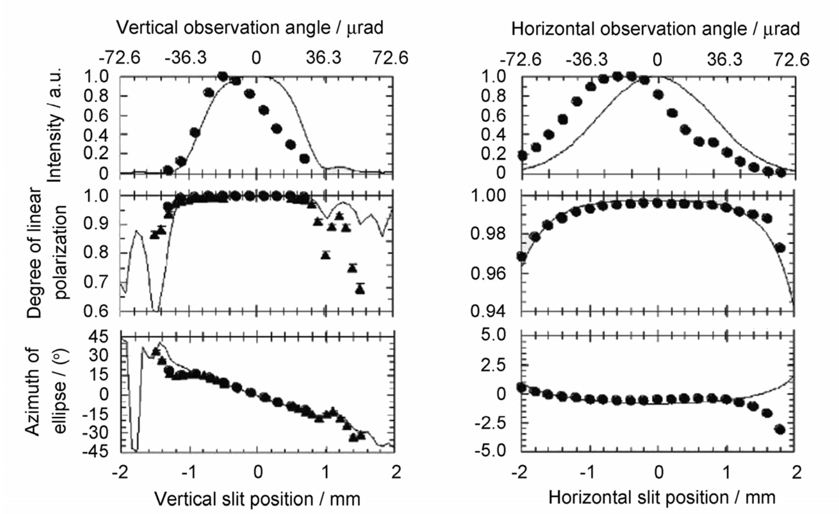

Fig.9 Polarization diagnosis of BL27SU, a figure-8 undulator beamline, SPring-8[35]. Experimental (dots) and theoretical results (solid curve) of distribution of intensity, degree of linear polarization and azimuth of ellipse are plotted against the vertical and horizontal positions of front end slits. The slit sizes were 0.2 mm×0.2 mm (●) and 0.1 mm×0.1 mm (▲).

4 Magneto-optical rotation experiments

One of main polarimetric experiments is the magneto-optical rotation experiment using linearly polarized light on magnetized materials. This kind of experiments in VUV and SX regions were firstly carried out on Fe, Co and Ni around their $L_{2,3}$ edges (700~900 eV) under magnetic field[15,21]. Here, just the experiments around Co and Ni $M_{2,3}$ edges (~60 eV) are introduced. The experiment set-up is shown schematically in Fig.10. The transmission experiment is called Faraday rotation experiment and the reflection one, magnetic Kerr rotation experiment.

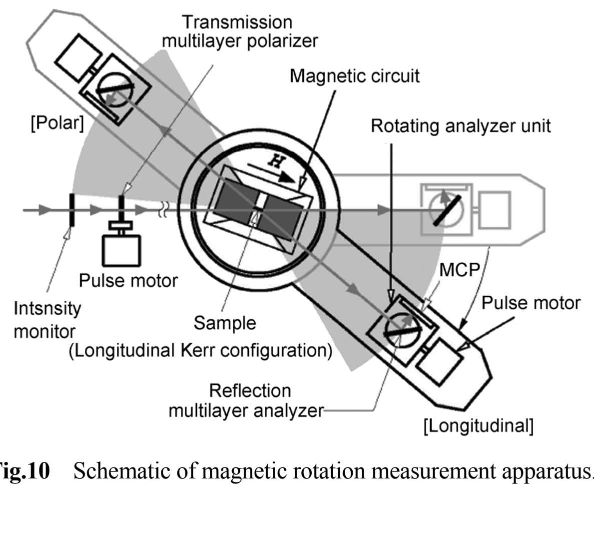

Fig.10 Schematic of magnetic rotation measurement apparatus.

4.1 Faraday rotation

In Faraday rotation experiment, the direction of applied magnetic field is parallel to the propagation direction of light. When linearly polarized light passes through a sample under magnetic field, the plane of electric field rotates from the initial plane, which is usually horizontal in SR experiments. The rotation correlates with the magnetic circular dichroism (MCD), which gives magnetic momentums in the case of core absorption experiments.

Fig.11 shows the Faraday rotation spectra obtained for Ni around its $M_{2,3}$ edges using a broad band multilayer[36]. It is clearly seen that the sign of rotation angle changes oppositely, when the direction of magnetic field is inversed. In MCD experiment, total electron yield (TEY) measurements are usually made rather than transmission measurements. However TEY measurements include considerably information near the surface, especially oxide layer, while the transmission measurements reflect the bulk properties[22].

Faraday rotation angle spectra of Co/Pt multilayers have been obtained using Al/YB_6 multilayer[37]. The Faraday rotation is observed around the Co $M_{2,3}$ edges and Pt $N_{4,5}$ edges. Pure Pt does not show magnetism, but in Co/Pt multilayers, it shows magnetism. Such effects have been found also in MCD experiment.

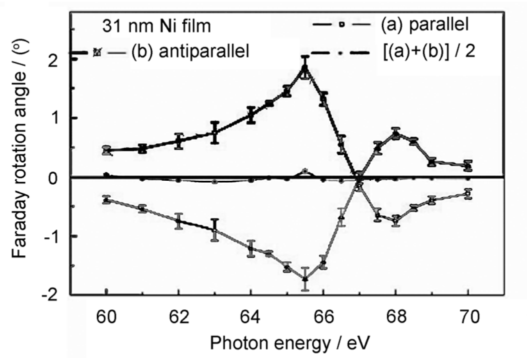

Fig.11 Faraday rotation spectrum of Ni film around its $M_{2,3}$ edges[36].

4.2 Magnetic Kerr rotation

For magnetic Kerr rotation experiments with reflection configuration, measurements are done on longitudinal Kerr effect and polar Kerr effect under longitudinal magnetic field, or transverse Kerr effect under transverse magnetic field. Here the measurement on longitudinal Kerr effect is demonstrated. Fig.12 shows the longitudinal Kerr rotation spectra of Co/Cu multilayers. The magnetic field is parallel to the direction of the light. The Kerr rotation is found around the Co $M_{2,3}$ edges and Cu $M_{2,3}$ edges. Pure Cu does not show magnetism, but in Co/Cu multilayers, it shows magnetism. Such experimental results are consistent with those of MCD-TEY measurements.

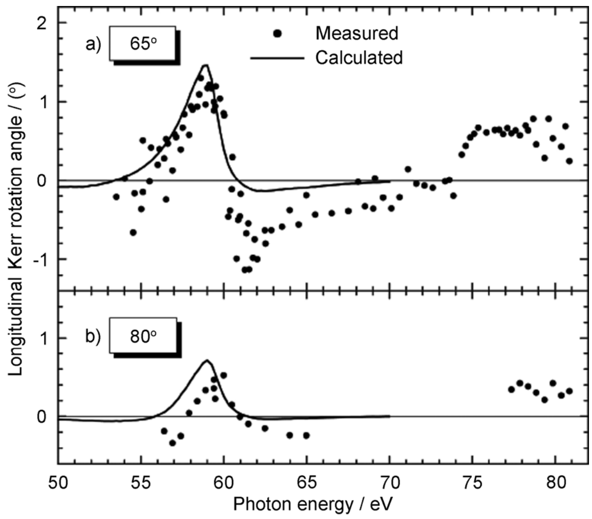

Fig.12 Magnetic Kerr rotation spectrum of Co/Cu multilayer around $M_{2,3}$ edges of Co and Cu[37].

5 Ellipsometry

In the visible region, the most popular ellipsometry employs the null-method, in which light of a wavelength is converted to linearly polarized light with a polarizer and is incident on a sample obliquely at a certain angle of incidence. Being reflected by the sample, the polarization state changes, i.e. the plane of electric field rotates and linearly polarized light becomes elliptically polarized light, which is changed into linearly polarized light using a quarter-wave plate. The angle of inclination of electric field plane is observed by an analyzer at crossed Nichol angle where light cannot pass. In another method, linearly polarized light is incident on a sample and the change of polarization state (polarization ellipse) is measured by a rotating analyzer. The configuration is similar to the one in Fig.10. In deep VUV and SX regions, ellipsometry using the latter method is adopted, because quarter-wave plates are still not popular.

The ratio of amplitude reflectance is defined as $r_p/r_s=\tan\Psi e^{i\Delta}$. The Ψ and Δ are measured by ellipsometry, which gives precise information of optical constants of samples, especially thin film or surface. Change in optical constant at growth process is detectable by this method of high sensitivity. In visible region, the method is used for *in situ* determination of film thickness in growth process. Similar experiment was made in SX region by multiple angle of incidence ellipsometry. Fig.13 shows the experimental and simulated results of Ψ and Δ at 97 eV for a Mo film deposited on Si substrate by magnetron sputtering[38]. The ellipsometry result was analyzed with the aid of the result of incident angle dependence of reflectance. The result could not be explained by a single layer model, but explained well by a double layer model of MoX/Mo/Si-substrate. It was found that the Mo film was not a simple monolayer, but was covered by unknown layer of MoX, of which optical constants are precisely determined.

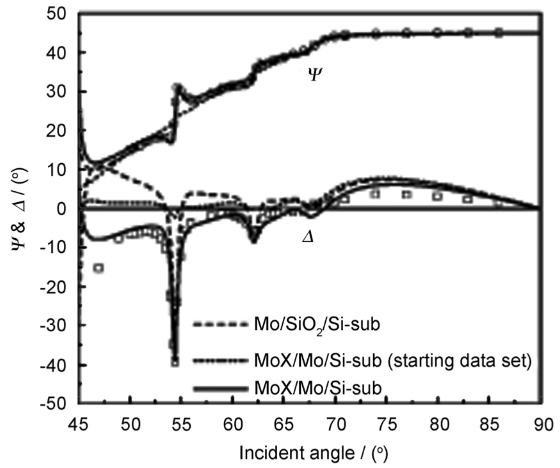

Fig.13 Ellipsometric parameter *Ψ* (open circle) and *Δ* (open square) measured by multiple angle of incidence ellipsometry at 97 eV. Solid, broken and dotted curves show simulated results[38].

6 Polarization measurements of SX fluorescence

When a grating is coated with a multilayer, its diffraction efficiency is enhanced in a certain wavelength region[39]. When the multilayer is fabricated for the use at an angle of incidence around 45°, the grating acts as not only a dispersive element, but also a polarization analyzer. Here, development of such grating and its application around 180 eV are described[40]. A laminar concave-grating with a radius of curvature of 1 m and a groove density of 2400/mm was coated with a Mo/B_4C multilayer. The 0^{th} order was suppressed by choosing the land-groove duty ratio as 1:1 and an appropriate depth of the laminar grating. Spectral diffraction efficiency of the 1^{st}, 0^{th} and -1^{st} orders of the multilayer grating and reflectance of the Mo/B_4C multilayer on a witness flat were measured. Efficiency of the $+1^{st}$ order is 3.4%. The wide wavelength band of good polarization was ensured by gradation of multilayer period in the grating surface area due to peculiarity of the present sputtering apparatus. The polarizance was 0.989.

The spectrometer to measure polarized SX fluorescence (Fig.14) consists of an entrance slit, the grating and a position sensitive detector. Angles of incidence and diffraction for the grating were chosen as about 45°, and the deviation angle was chosen as 90°. Monochromatized undulator light irradiates the sample with the electric field perpendicular to the plane of incidence. Materials such as borides with layered structure have σ- and π-bonds originated from the hybridized B 2s and 2p orbitals. The σ- and π-bonds are parallel and perpendicular, respectively, to the layer, whose normal is consistent with the *c*-axis. The lowest core level is 1s (K) state, which is spherically symmetric. Therefore when the K state is excited, valence band emission is observed, which reflects polarization character of the bond, that is, polarization character of the valence band. As an example, polarization dependence of the B K emission spectrum of CrB_2 is shown in Fig.15[40]. π-emission was observed with a configuration in which the *c*-axis was perpendicular to the plane of incidence of the multilayer grating and σ-emission was observed when the *c*-axis was parallel to it. The shape and energy position of both the σ- and π-emission spectra were well explained by the calculated density of states.

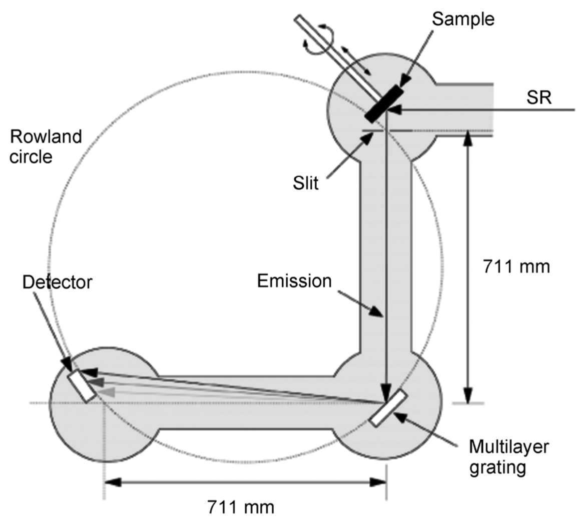

Fig.14 Side-view schematic of polarization spectrometer mounting a multilayer-coated grating[40].

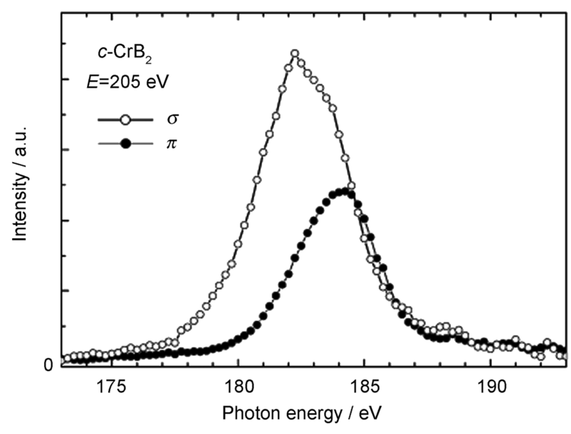

Fig.15 B K σ- and π-emission spectra of CrB_2 single crystal[40].

7 Summary and outlook

For the polarimetric study in VUV and SX regions, multilayer polarizers of reflection type with high polarizance and reflectance are needed. It was suggested that the first important item of selection of material combinations to get such multilayer polarizers is to choose materials with small extinction coefficients. The second item is to choose materials with large difference in refractive index. Some practical examples were given for a 20~400 eV region. The multilayer structure is periodic or non periodic. Non periodic one has an advantage of wide reflection band. Transmission type is also useful for polarizers and phase shifters. It may be challenging to develop non periodic transmission multilayers for use in a wide band. Conversion of linearly polarized light to circularly polarized light by the use of quarter-wave plates made of transmission multilayers has not been practically performed because of large absorptions. However it is possible to make elliptically polarized light to circularly polarized light. Furthermore in the case of strong SX laser[41], the conversion will be in practical use. Switching helicity (switching left and right circularly polarized light) is further a subject.

By the use of multilayer polarizers, several kinds of polarimetric studies have been intensively made. Some of them made in 20~400 eV were introduced. Polarization diagnosis for laboratory optical system and SR beamline of linear and circular polarization were described. It was noticed that gratings and mirrors have polarization action and it was emphasized that polarization characteristics behind monochromator systems have to be checked. The polarization diagnosis is a powerful tool to evaluate alignment of SR beamlines. The magnetic rotation experiments such as Faraday rotation and magnetic Kerr rotation on magnetic films and multilayers around M edges of transition metals were mentioned. Circularly polarized light with high polarization degree is not always obtained, so that exact circular dichroism experiments are not always possible. On the other hand, linearly polarized light with high polarization degree is easily obtained for optical rotation experiment. Furthermore optical rotation experiment for small dichroic materials such as materials with natural chirality will be possible. Ellipsometry using VUV and SX was demonstrated as a method to characterize the material surfaces precisely by measuring optical constants with high sensitivity. The sample was Mo film. The *in site* VUV and SX ellipsometry on thin film growth studies will be promising. Polarization analysis of SX fluorescence from a boride layer crystal using multilayer-coated grating was introduced. There are many kinds of useful layer materials including B, C, N and O, so that this method can be extended to these materials for clarification of their bonding nature and electronic structure.

In making a summary, the multilayer polarizers will be steadily developed and provide new opportunity of polarimetric studies in VUV and SX regions.

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