Chapter 4

Multilayer Polarizer at the Energy of 50–1000 eV

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Additional information is available at the end of the chapter

http://dx.doi.org/10.5772/intechopen.70088

Abstract

Accurate evaluation of polarization states of the radiation is necessary for polarization-sensitive studies, which requires polarization optical elements, such as polarizer, analyzer, and phase retarder. In extreme ultraviolet (EUV) and soft X-ray region, the closeness of the real part of the refractive index to unity, coupled with high absorption, makes the realization of polarizers such as birefringence and dichroic polarizers impossible. Periodical multilayers are commonly used in polarization study working at the quasi-Brewster angle. To expand narrow spectral bandwidths of periodic multilayers, aperiodic and lateral gradual multilayer polarizers including reflective analyzers and transmission phase retarders are utilized. In this chapter, we demonstrate a series of periodic, aperiodic, and lateral gradual broadband multilayer polarizers with the material combinations of Mo/Si, Mo/Y, Mo/B_4C, Cr/C, Cr/Sc, Cr/Ti, Cr/V, WSi_2/Si, W/B_4C, etc. Different multilayer polarizers correspond to different energy ranges, covering 50–1000 eV totally, including "water window" and the L absorption edges of Fe, Co, and Ni. Polarization measurements are performed at BESSY II, Diamond Light Source, National Synchrotron Radiation Laboratory in Hefei and Beijing Synchrotron Radiation Facility. Some of the polarizers we have developed are applied to the polarization measurements of BESSY II UE56/1-PGM and Beamline 3W1B of Beijing Synchrotron Radiation Facility.

Keywords: polarization, extreme ultraviolet (EUV), soft X-ray, multilayer thin film, synchrotron radiation beamline

1. Introduction

For polarization-sensitive studies, such as circular dichroism spectroscopy, spin-polarized photoelectron spectroscopy, and spectroscopic ellipsometry [1–4], accurate evaluation of the polarization state of the radiation is clearly crucial. Traditionally, in the visible and ultraviolet regions of the spectrum, birefringence and dichroic polarizers are used. But, in the soft X-ray region and extreme ultraviolet regions, collectively known as the EUV, the closeness of the (real part of the) refractive index, coupled with high absorption, makes the realization of these kinds of polarizers impossible. As the one-dimension artificial crystal, multilayer can be utilized as polarizers and phase retarders in EUV and soft X-ray regions for its interference structures, which are based on Bragg diffraction theory [5, 6]. Periodical multilayers are commonly used in polarization study when they work at the quasi-Brewster angle. But the narrow spectral bandwidth of Bragg peak cannot fulfill the need of broadband spectrum applications. In order to expand the narrow spectral bandwidth of the periodic multilayer, the aperiodic multilayer polarizer including reflective analyzer and transmission phase retarder is utilized [7–10]. Complete polarization analysis has been performed by this new developed polarization system [11]. However, with the energy increasing, especially for the "water window" energy range, which is essential to biology researches, the aperiodic multilayer is no longer suitable because the period thicknesses in such energy region are ultra-thin (typically less than 1.0 nm). In this case, a lateral gradual multilayer is utilized and demonstrated with different material combinations. We also design and fabricate different types of multilayer polarizers at the energy range from 50 to 1000 eV covering the L absorption edges of metal magnetic materials Fe, Co, and Ni and also including the important energy range "water window." Measurements of the multilayer polarizers are performed at BESSY II, Diamond Light Source, Beijing Synchrotron Radiation Facility, and National Synchrotron Radiation Laboratory.

2. Design

The design of multilayer for polarization analysis requires determining the following parameters: materials combination, incident angle, and layer thickness distribution. The best material combination for multilayer is to form smooth and abrupt interface with low absorption and high optical contrast. The imperfections of interface as well as the absorption will influence the reflectivity of the multilayer. The absorption will be enhanced at a specific energy during the interaction between materials and incident light. To minimize the absorption, we choose material with the absorption edge slightly smaller than the working energy as the spacing layer. To maximize the interference in the structure of multilayer, the one with high optical contrast to the spacing layer is selected as the absorption layer. Here, we design periodical multilayer polarizers with different material combinations at the energy range from 50 to 1000 eV. **Table 1** shows the typical material combinations at different working energies.

Taking Mo/Y multilayer as an example, the calculated s-reflectivity, p-reflectivity, and R_s/R_p with the optimized parameters are shown in **Figure 1**. *Y*-axis is in log to make all curves clear. The corresponding parameters are shown in **Table 2**, where, D represents the period thickness, N for bilayer numbers, and γ for the ratio of spacing layer thickness to period thickness. From **Figure 1**, we can see that the bandwidth is quite narrow. Such polarizers are effective over a very small wavelength range and close to the Brewster angle (approximately 45° for EUV and soft X-ray radiation); the narrowband wavelength and angular properties may be disadvantages for some studies. In order to overcome this, Yanagihara et al. [12] developed the

Energy (eV)	Wavelength (nm)	Material combination
50–98	24–12	Mo/Si
98–155	8–12	Mo/Y
155–185	6.7–8	Mo/B_4C
177–282	4.4–6.7	Cr/C
284–543	2.3–4.4	Cr/Sc, Cr/Ti, Cr/V
540–1000	1.2–2.3	W/B_4C

Table 1. Typical material combinations for different energy ranges.

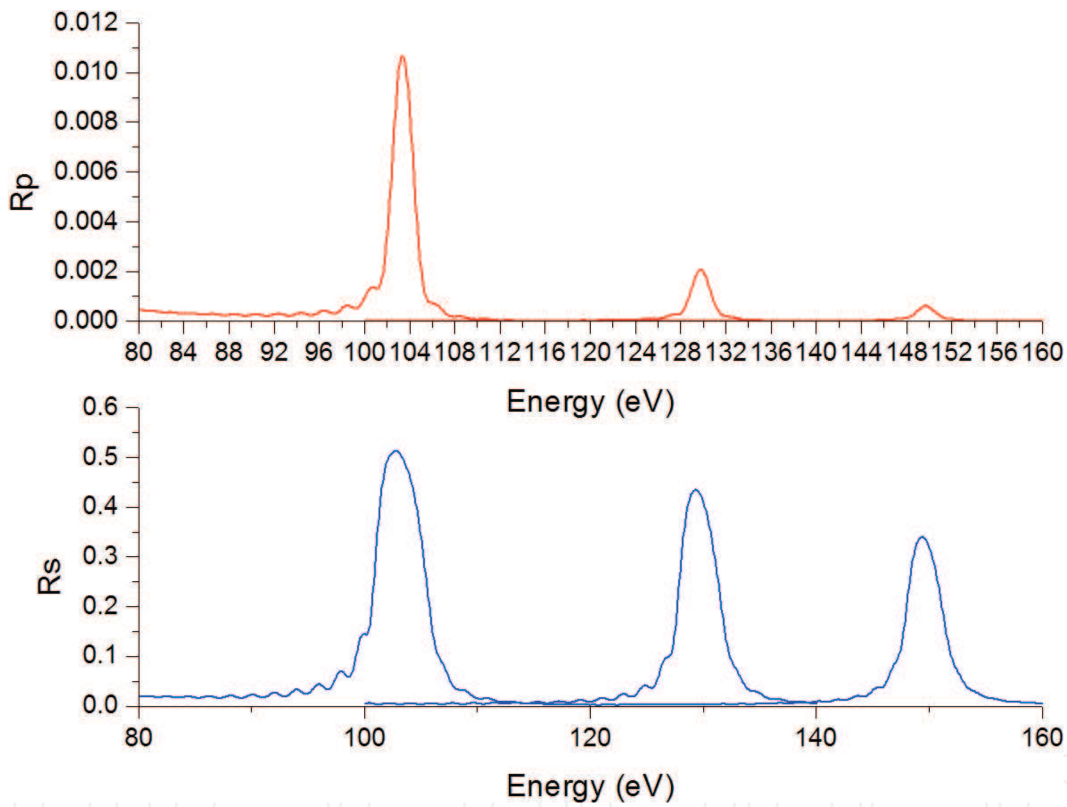

Figure 1. R_s, R_p, and R_s/R_p for Mo/Y periodic multilayers.

Energy (eV)	Material combination	*N*	*γ*	*D* (nm)	R_s
100	Mo/Y	40	0.56	9.60	0.58
130	Mo/Y	60	0.50	7.05	0.46
150	Mo/Y	80	0.53	6.03	0.34

Table 2. Parameters of Mo/Y periodic multilayers.

double-multilayer polarizer, which, however, requires a complicated experimental arrangement and degrades the throughput. By rotating and translating the multilayer polarizers, an s-reflectivity of >4% in the wavelength range of 8–15 nm has been achieved. Also, Kortright et al. [13] have constructed a continuously tunable, laterally graded multilayer-based polarimeter working over a wide energy range. Using a series of translatable laterally gradual multilayers, the polarization state of the beam can be determined over the range of 1.6–25 nm.

To simplify the use of broadband polarizers, the design and performances of aperiodic broadband multilayer polarizers are described here.

At the quasi-Brewster angle, the ratio of the s- to p-reflectivity reaches the largest, where the reflective analyzer can be obtained. Since the complex refractive indices of all materials are very close to unity in EUV and soft X-ray ranges, the quasi-Brewster is close to 45°. The polarization degree P of beam reflected from a multilayer is determined by usual manner,

$$P = \frac{R_s - R_p}{R_s + R_p} \tag{1}$$

where R_s and R_p are the reflectivities for s- and p-polarized radiation, respectively. In the design of a reflective multilayer analyzer, the optimization of layer thickness distribution is achieved by minimizing the merit function (MF) [14],

$$\mathrm{MF} = \frac{1}{m}\sum_{j=1}^{m}\left[1 - P\left(\lambda_j\right)\right]^2 \tag{2}$$

where the summation is over a selection of discrete wavelengths in the desired range. The layer thickness distribution is considered as independent variable. During the recursive optimization, only randomly selected layer thickness changes that decrease MF are retained, finally leading to an optimized layer thickness distribution that provides a minimum value of MF.

For the wide angular multilayer analyzer, the optimization is achieved by minimizing the following merit function (MF)

$$\mathrm{MF} = \frac{1}{m}\sum_{j=1}^{m}\left(1 - P\left(\theta_j\right)\right)^2 \tag{3}$$

where the summation is over a selection of discrete angles in the desired range.

The phases of the transmitted s (Φ_s) and p (Φ_p) electromagnetic fields can be calculated following the formalism of Vidal and Vincent [15], in which the phase shift $\Delta\Phi = \Phi_s - \Phi_p$ is evaluated as a function of the wavelength, the grazing angle, the optical constants of the materials, the number of layers, and their thicknesses. Initially, the published criteria for selecting material and the number of bi-layer were used to satisfy both the maximum phase shift and the transmission intensity [16]. Then, the grazing angle was set in the region between the Bragg peak and the total reflection [17], at which the phase shift is maximum. The numerical optimization method was based on the minimization of the usual merit function (MF)

$$MF = \left(\frac{1}{n} \sum_{j=1}^{n} \left(\Phi_0 - \Delta\Phi(\lambda_j) \right)^2 \right)^{1/2} \quad (4)$$

where $\Delta\Phi(\lambda_j)$ is the calculated phase retardation at wavelength λ_j and Φ_0 is the desired phase shift.

By using the above scheme, we have designed a series of aperiodic multilayers such as Mo/Y [7], Mo/Be, Ni/C, and Mo/Si [8, 9], which can be utilized as broadband reflection analyzer and transmission phase retarder [10]. The broad angular Mo/Si multilayer analyzer is described in detail as an example, which the layer thicknesses oscillate from 3 to 11 nm, a range that is feasible to manufacture using direct current (DC) magnetron sputtering machine. The calculated mean s-reflectivity in the 45–49° range at working wavelength of 13 nm is 64.99 ± 0.18% and the degree of polarization is up to 99.12 ± 0.71%.

However, with the increasing energy, especially in the "water window" energy range, period thicknesses are decreasing to around 1.0 nm. It is difficult for magnetron sputtering to deposit such thin layer accounting interface roughness and diffusion. In this case, a lateral gradual multilayer structure is utilized to fulfill the Bragg condition either for varying energy or varying incident angle. Each periodic thickness of the lateral gradual multilayer varies in the horizontal direction corresponding to different energy, so that the energy bandwidth can be broadened to a respectively large range.

We design a series of lateral gradual multilayer polarizers with the material combinations of Mo/Si, Mo/Y, Mo/B_4C, Cr/C, Cr/Sc, Cr/Ti, Cr/V, W/Si, WSi_2/Si, and W/B_4C, which the minimum lateral gradual reaches 0.01 nm/mm with the total length of 70 mm.

As an example, the designed parameters of WSi_2/Si multilayer polarizer are given in **Table 3**. The working energy range is 430–760 eV. The calculated s- and p-reflectivities at every 5 mm are shown in **Figure 2**.

Sample		**Position (mm)**						**Energy range (eV)**
		L0	**L5**	**L10**	**L15**	**L20**	**L25**	
S1-$[WSi_2/Si]_{400}$	Thickness (Å)	15.33	16.23	17.23	18.25	19.31	20.30	–
	Peak energy at 45° (eV)	578.9	547.3	516.0	483.0	456.8	434.7	434.7–578.9
S2-$[WSi_2/Si]_{400}$	Thickness (Å)	11.5	12.37	13.04	13.75	14.50	15.33	–
	Peak energy at 45° (eV)	764.2	715.0	679.0	644.6	611.7	578.9	578.9–764.2

Table 3. Designed parameters of WSi_2/Si lateral gradual multilayer.

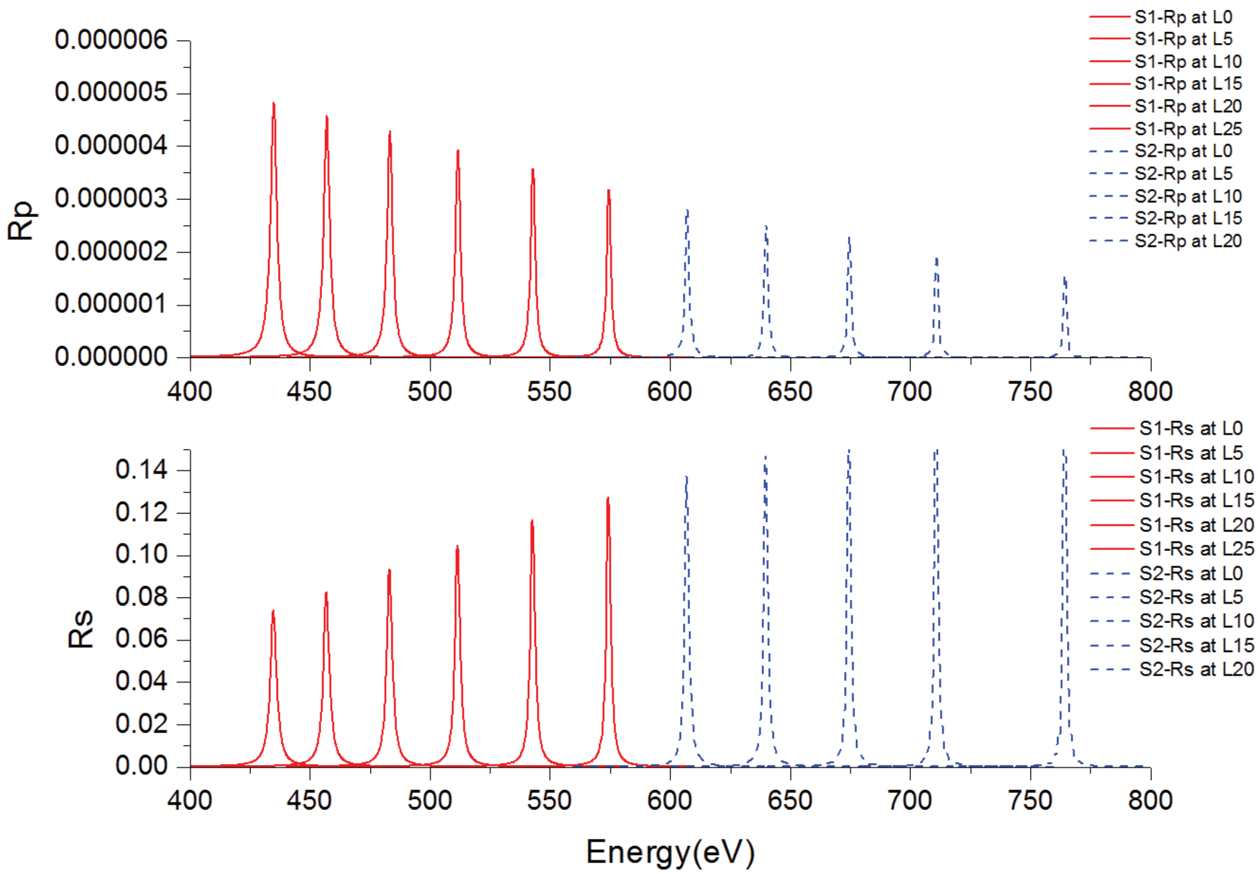

Figure 2. The calculated s- and p-reflectivities of WSi_2/Si lateral gradual multilayer.

3. Fabrication

The multilayer polarizers were deposited using a calibrated ultrahigh vacuum direct current (DC) magnetron sputtering system. They were deposited onto P(100) silicon wafer substrates at room temperature. Mask and speed control system were utilized in the fabrication of lateral gradual multilayers to control the lateral growth. The deposited multilayers were measured, for quality control, using a grazing incident angle X-ray reflectometry (XRR).

As an example, **Figure 3** shows the XRR curves for Mo/Si lateral gradual multilayer. Bragg peaks move evenly at different positions of the multilayer, which suggests a great homogeneity in lateral gradual variation. The appearances of all the fifth-order Bragg peaks prove the stability of fabrication technique.

The XRR result for W/B_4C lateral gradual multilayer with 150 layers is shown in **Figure 4**. The measured period thicknesses at the positions of every 5 mm (every 2 mm at beginning) are given in **Table 4**. As a result, period thicknesses of W/B_4C lateral gradual multilayer vary from 1.75 to 0.95 nm, and the multilayer covers the L absorption edges of Fe, Co, and Ni (600–900 eV) at quasi-Brewster angle, which is essential to the researches on magnetic materials.

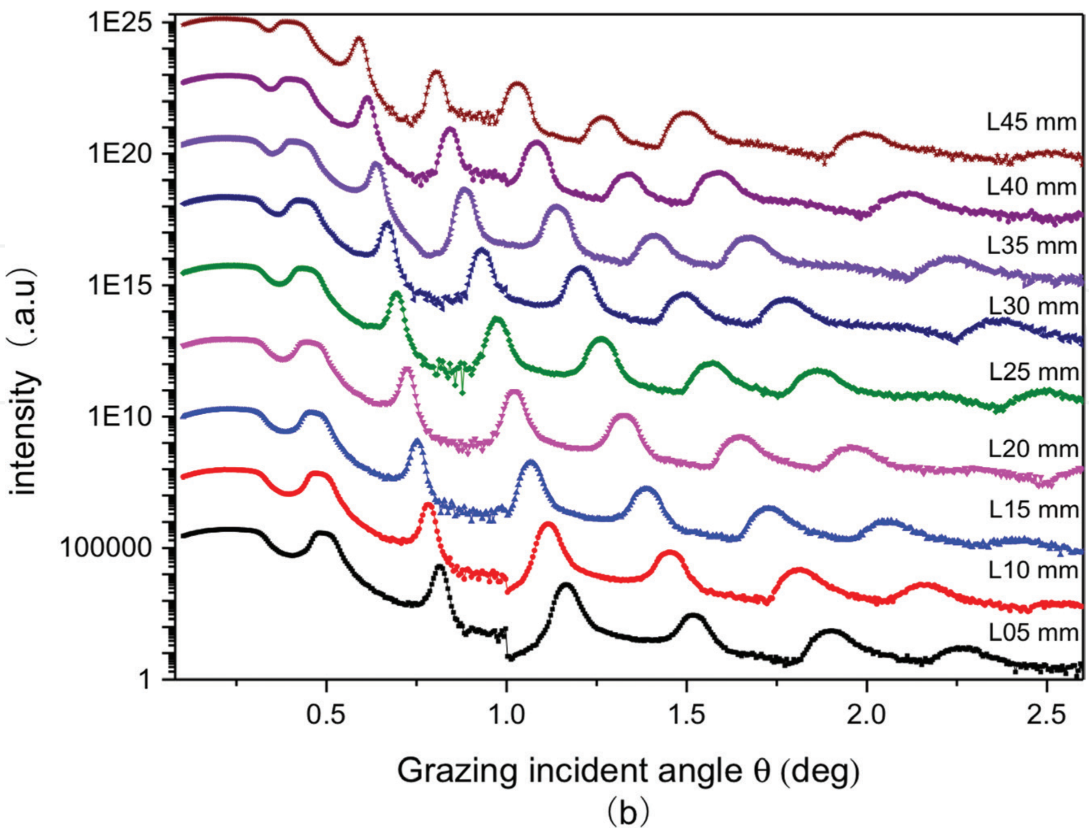

Figure 3. XRR results for Mo/Si lateral gradual multilayer at different positions (translations are set in *Y*-axis to make all the curves clear).

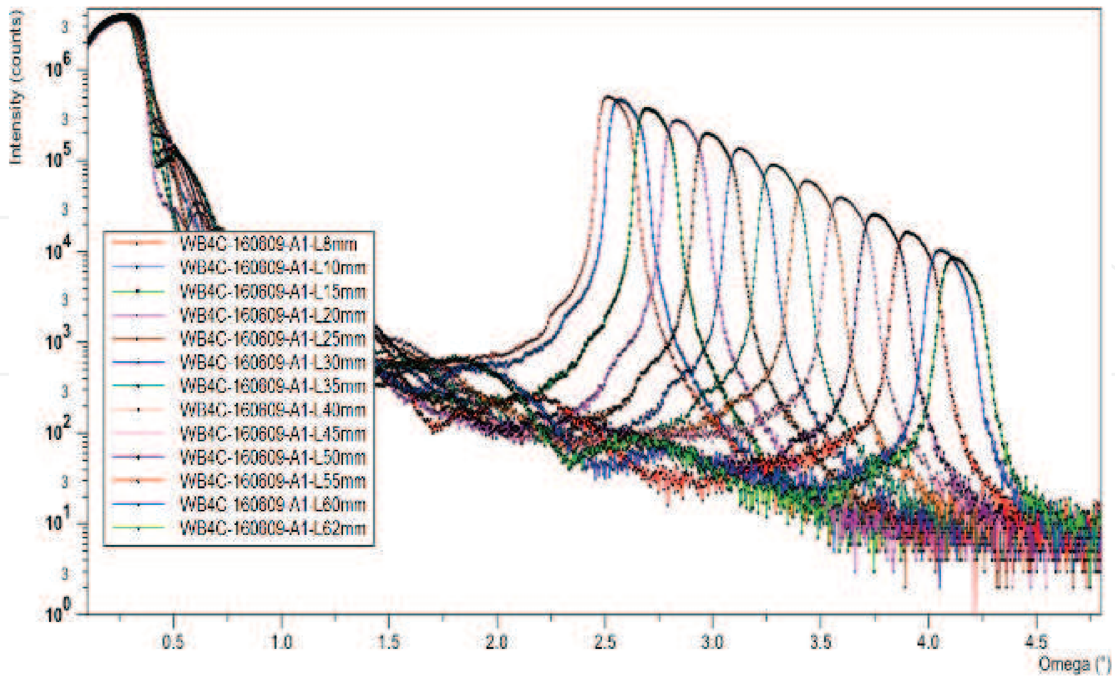

Figure 4. XRR results for W/B_4C lateral gradual multilayer at different positions.

Position (mm)	08	10	15	20	25	30	35	40	45	50	55	60	65
D (nm)	1.75	1.73	1.63	1.56	1.48	1.40	1.34	1.28	1.22	1.17	1.13	1.08	0.95
Energy at 45° (eV)	501	507	538	562	592	626	654	685	719	749	776	812	923

Table 4. Measured periods of W/B_4C lateral gradual multilayer at different positions.

4. Characterization and applications in synchrotron radiation measurement

The performances of Mo/Y and Mo/Si aperiodic multilayers have been evaluated using the high precision polarimeter on beamline UE56/1-PGM at BESSY II. The details can be found in our previous articles [7–11].

Taking as an example, measured mean s-reflectivities of Mo/Si transmission phase retarders with the bandwidth of 2.0 nm are 10.4% at 47° and the corresponding polarization efficiency is up to 98.9%, which is nearly four times of the periodic one. The analyzer also exhibits high s-reflectivity over the angular range of 10° at the fixed wavelength.

The polarization performance of Mo/Si transmission phase retarders is evaluated with broadband analyzer. A transmittance ratio T_p/T_s of 5–23 from 12.5 to 13.2 nm is achieved and a constant phase shift of 50° at wavelength of 13.8–15.5 nm with the relative transmittance is 0.8–1.2.

Using this broadband Mo/Si polarizing system, the polarization of BESSY II UE56/1-PGM at 13.1 nm has been determined for the first time to our knowledge. As a result, the light of monochromatic radiation is nearly completely circularly, where the Stokes parameter is $|S3| \geq 0.94$, and the linear polarization is the largest with −0.96 for in-plane radiation [11].

A flexible soft X-ray multilayer polarimeter has been designed and built on beamline I06 at diamond Light Source. This allows polarization measurements using multilayers with unprecedented reproducibility and accuracy. The polarimeter with its pinhole assemblies and hexapod is easy to align to a beamline. More detailed description about this polarimeter can be seen in Ref. [18].

Experiments to characterize polarization properties of broadband lateral gradual multilayers have been performed. The designed energy range is from 434 to 764 eV at quasi-Brewster angle for WSi_2/Si lateral gradual multilayer. The measurements are performed at every 2.5 mm along the horizontal direction. The corresponding multilayer periods are from 1.18 to 2.03 nm. The highest s-reflectivity reaches 2% in the wide energy range of 434–764 eV, i.e., a 330 eV-broadband polarizer has been achieved.

The optical properties of Mo/Si and Mo/Y lateral gradual multilayer are evaluated at NSRL in Hefei. The designed parameters are shown in **Tables 5** and **6**.

Reflectivities at different position of both Mo/Si and Mo/Y lateral gradual multilayer are measured at metrology station of NSRL in Hefei. As shown in **Figures 5** and **6**, the highest reflectivity of Mo/Si multilayer is more than 50% and obtained at the wavelength of 14.4 nm (86 eV). Mo/Y multilayer achieves the highest reflectivity of 32% at the wavelength of 13 nm (95 eV). The Bragg peaks of both multilayers distribute homogeneously in the energy-space at different positions, which shows a great stability in the gradient control during fabrication along the horizontal direction.

Position (mm)	L05	L10	L15	L20	L25	L30	L35	L40	L45
D (nm)	11.89	12.45	13.07	13.72	14.37	15.15	16.05	17.03	18.06

Table 5. Designed parameters of Mo/Si lateral gradual multilayer.

Position (mm)	L05	L10	L15	L20	L25	L30	L35	L40	L45	L50	L55	L60	L65
D (nm)	7.47	7.74	8.00	8.30	8.66	9.04	9.38	9.75	10.17	10.65	11.17	11.67	12.05

Table 6. Designed parameters of Mo/Y lateral gradual multilayer.

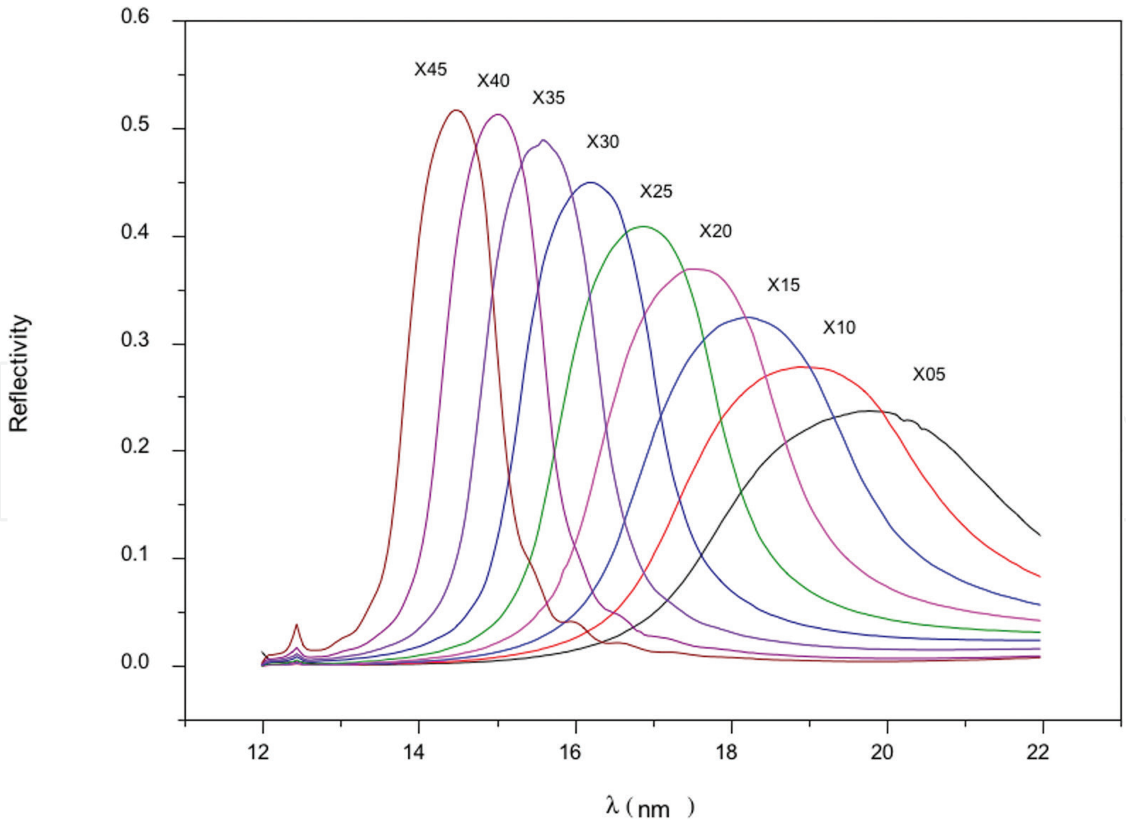

Figure 5. Reflectivities of Mo/Si lateral gradual multilayer at different positions.

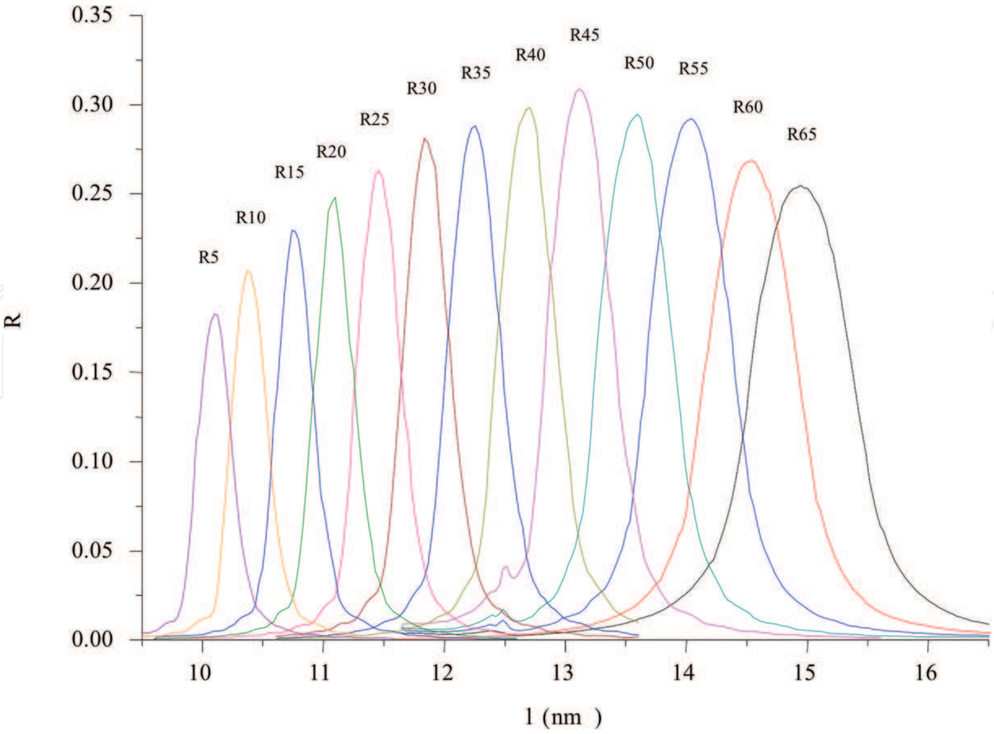

Figure 6. Reflectivities of Mo/Y lateral gradual multilayer at different positions.

5. Polarimeter in Beijing Synchrotron Radiation Facility (BSRF) and application

A compact high precision eight-axis automatism and two-axis manual soft-ray polarimeter has been designed, constructed, and installed in 3W1B at the Beijing Synchrotron Radiation Facility (BSRF). The beamline is a windowless full ultra-high vacuum (UHV) soft X-ray beamline utilizing a permanent-magnet wiggler source. It supplies focusing monochromatic soft X-rays. The photon energy region is from 50 to 1600 eV. There are four operational modes in this polarimeter device, which are double-reflection, double-transmission, front-reflection-behind-transmission, and front-transmission-behind-reflection. The schematic diagrams of four modes are shown in **Figure 7**. The chamber of the polarimeter links to the beamline by UHV ferrofluidics feedthrough on the upstream. It can be rotated around the axis of the incident beam. The chamber is supported on gimbals and installed in a rigid bench which can move in three dimensions. The schematic diagram and the photo of the polarimeter are shown in **Figures 8** and **9** [19].

The polarization properties of Mo/Y, Mo/B_4C, and W/B_4C lateral gradual multilayer have been characterized by this polarimeter. Results are shown in **Figure 10**.

Mo/B_4C lateral gradual multilayer polarizer is used to measure the polarization characteristics of the beamline 3W1B at BSRF. The measured parameters are given in **Table 7**. A highest s-reflectivity of 12% is achieved at the energy of 188 eV. **Figure 11** shows the measured reflectivities, where, p-reflectivities are not low enough comparing to s-reflectivities. That suggests the beam coming out from 3W1B is not completely linear polarized, which coincides with the result shown in **Table 7** that the average degree of polarization is around 72% for the beamline 3W1B.

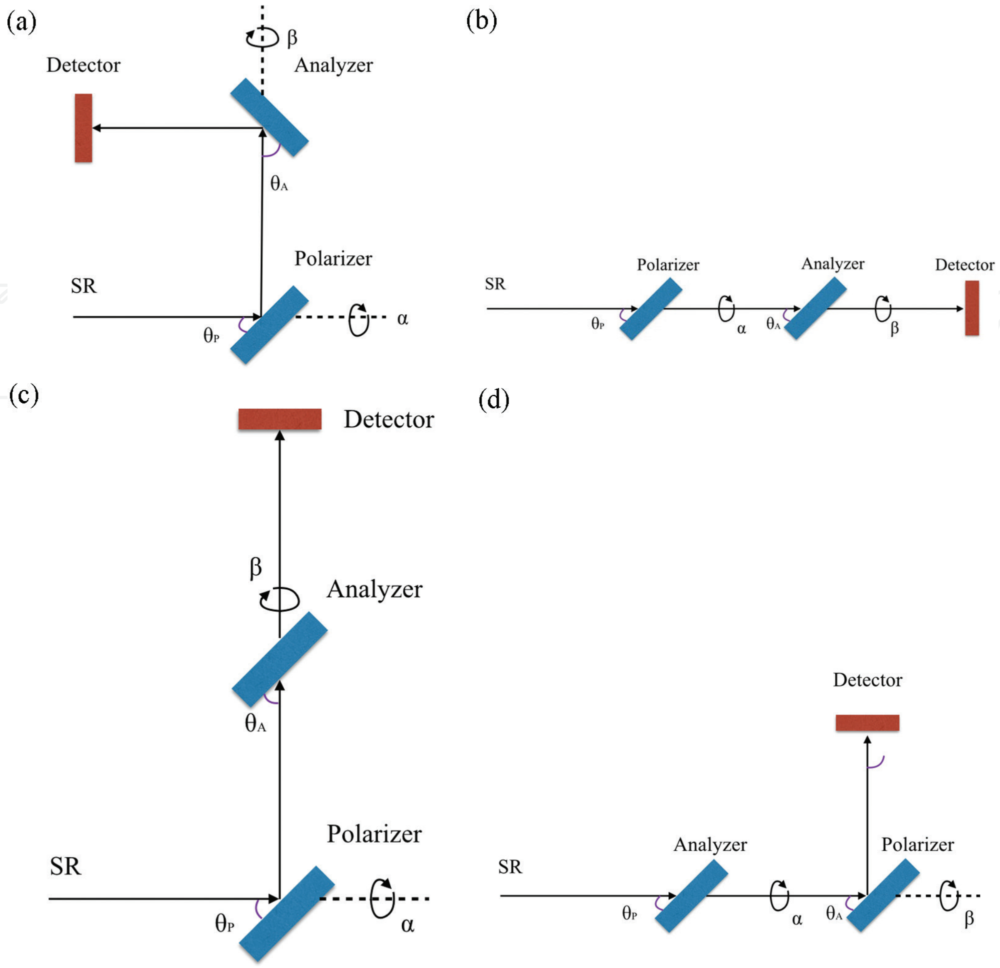

Figure 7. Schematic diagrams of four operational modes in polarimeter: (a) double-reflection; (b) double-transmission; (c) front-reflection-behind-transmission; (d) front-transmission-behind-reflection.

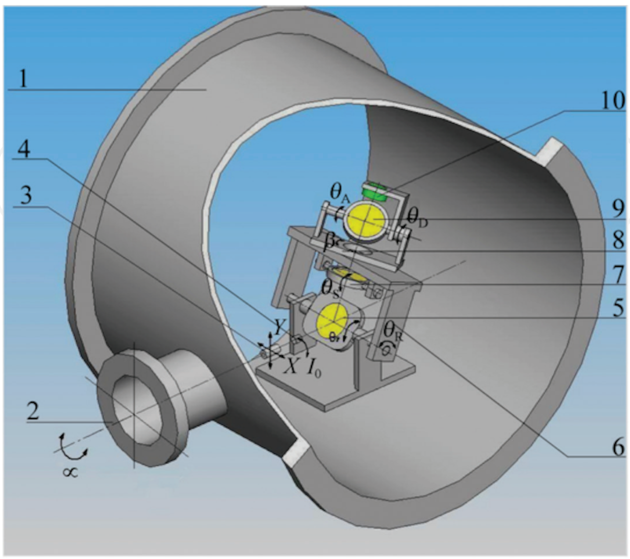

Figure 8. Schematic diagram of the polarimeter at BSRF: (1) chamber; (2) azimuthal angle of polarizer; (3) collimator translation x-y stage; (4) I_0 detector; (5) polarizer; (6) moving rocker; (7) sample stage; (8) rotational stage to change azimuth angle of analyzer; (9) analyzer; and (10) main detector.

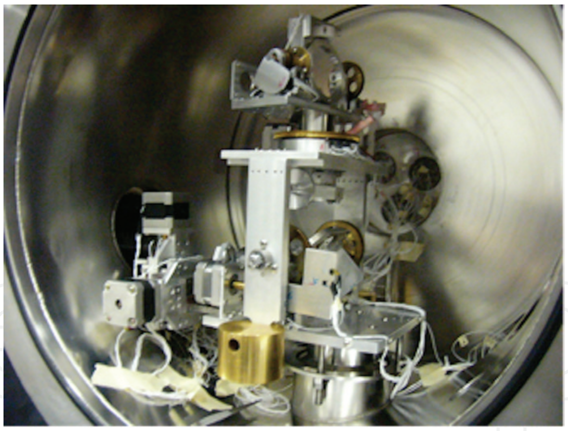

Figure 9. Photo of the polarimeter device.

To obtain complete liner polarization at 3W1B, two twins-born Cr/C multilayers are used as polarizer and analyzer at the energy of 206 eV. Both Cr/C multilayers have the exactly same optical characteristic with bi-layer number of 100 and period thickness of 4.318 nm. Using the double-reflection mode of the soft X-ray polarimeter, a nearly complete linear polarized beam is achieved. The schematic diagram is the same as shown in **Figure 7(a)**. Both multilayers are set at their quasi-Brewster angles. As a result, the degree of linear polarization is raised from 0.585 to 0.995 after polarized by Cr/C multilayer at 206 eV. The detailed results of the polarization optimization at other energies are shown in **Table 8** [20].

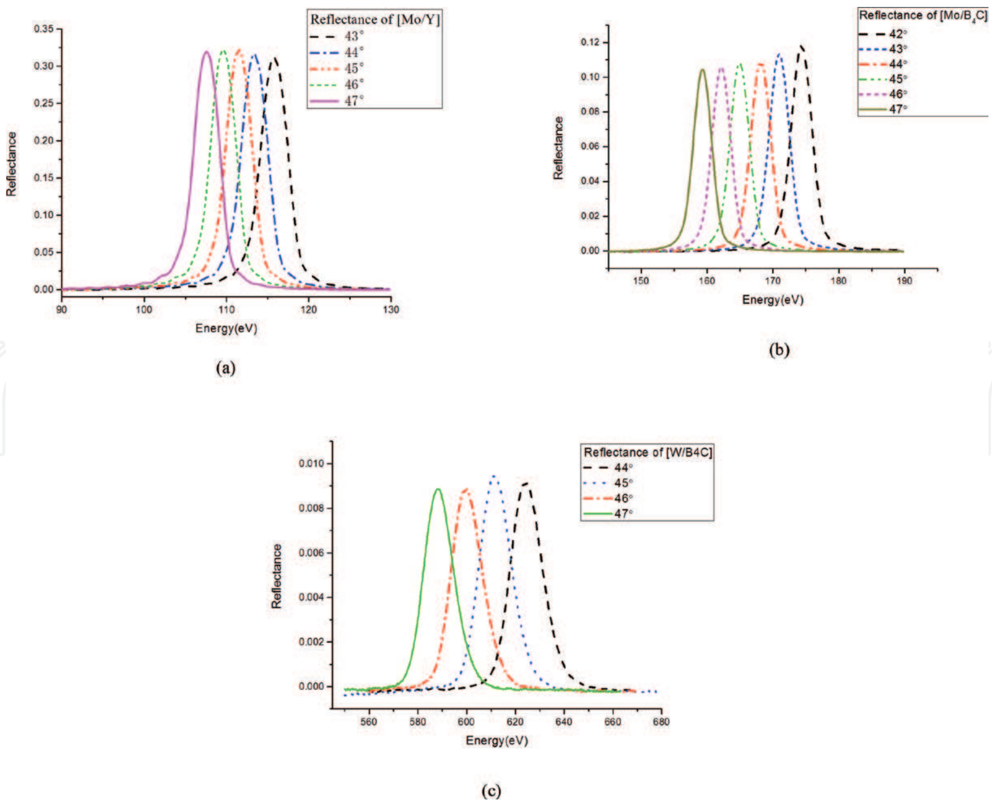

Figure 10. S-reflectivity as a function of energy: (a) Mo/Y; (b) Mo/B_4C; (c) W/B_4C.

Position (mm)	I_s/I_p	I_s-I_p/I_s+I_p	Peak energy (eV) R_s/R_p	R_s	R_s/R_p
L9	5.98	0.71	187.7/187.6	0.12	5.98
L26	6.55	0.74	163.4/163.2	0.10	6.54
L44	6.80	0.74	140.7/140.5	0.10	6.80
L61	6.68	0.74	121.7/122.4	0.09	6.69

Table 7. Measured parameters of Mo/B_4C lateral gradual multilayer at different positions.

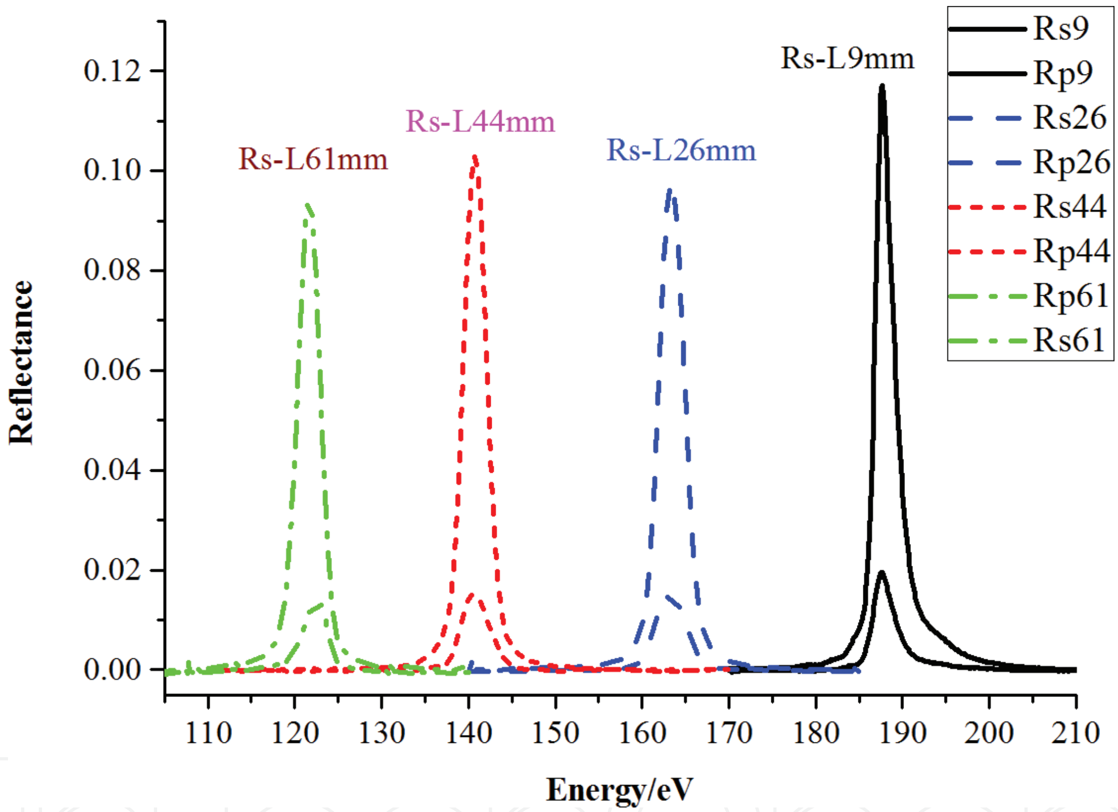

Figure 11. S-reflectivities and p-reflectivities at different energies of the beamline 3W1B.

Energy (eV)	P_L (linear polarization degree) before polarizing	P_L (linear polarization degree) after polarizing
206.0	0.585	0.995
92.5	0.443	0.99
77.5	0.373	0.985
65.0	0.400	0.950

Table 8. Measurement results of linear polarization at different energies.

6. Conclusion

The multilayers can be utilized in EUV and soft X-ray polarization applications. A series of multilayers with periodic and aperiodic thicknesses are designed to broaden the bandwidth. Measurements are performed at BESSY-II, Diamond and Beijing Synchrotron Radiation Facility. For Mo/Y aperiodic multilayer, polarization efficiency is up to 20% over a 90–120 eV (10.5–13.5 nm) energy range. Mo/Si broadband transmission phase retarder is also realized. Combination using the polarimeter, complete polarization characteristics of the synchrotron radiation beam is performed. The results show that broadband aperiodic multilayer greatly simplifies polarization experiments. For further development of multilayer polarizers in soft X-ray region, especially the "water window" range, a series of lateral gradual multilayers are utilized. The performance of WSi_2/Si lateral gradual multilayer polarizer is evaluated at diamond light source, which, as a result, covers the energy range of 434–764 eV. In addition, polarization measurements of Mo/Si, Mo/Y, Mo/B_4C, W/B_4C, and other lateral gradual multilayers have been performed at Beijing Synchrotron Radiation Facility and National Synchrotron Radiation Laboratory in Hefei. These researches create some novel experimental methods, such as soft X-ray magnetic circular dichroism, element resolution Faraday effect, and Kerr effect measurements, which provide the powerful tools for biology, medicine, information, material science, etc.

Acknowledgements

The authors are indebted to Dr. Igor V. Kozhevnikov for useful discussions. Special thanks are due to Dr. Franz. Schäfers, Dr. Hongchang Wang, Matthew Hand, Hongjun Zhou, Tonglin Huo, and Dr. Lijuan Sun for their kind assistance during the measurement at BESSY, Diamond, NSRL, and BSRF. The authors are grateful to Dr. Alan Michette, Dr.Keith Powell, Dr. Slawka Pfauntsch, Dr. Sawhney Kawal, and Prof. Zhanshan Wang for their great support during this work. The author would also like to thank Miao Li, Shengming Zhu, Bin Ji, Jinwen Chen, and Jiayi Zhang for the participations during multilayer design, fabrication, and measurement processes. This work was supported by the National Natural Science Foundation of China (contract numbers 11375131 and U1432244), by the Royal Society—NSFC International Exchanges funding (reference IE141043 and 11511130051), and by the Fundamental Research Funds for Central Universities.

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