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Article

Sc/SiC/Al Multilayer Optimization for Li K Spectroscopy

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Abstract: This paper presents an X-ray reflectivity study of a Sc/SiC/Al periodic multilayer deposited via magnetron sputtering and its possible adaptation to be used as a dispersive element in the crystal spectrometers equipping scanning electron microscopes and electron probe microanalyzers. This multilayer is designed for the spectral range of 45–60 eV. The results reveal a reflectance of 40.8% at 54.1 eV for a near-normal incidence angle of 7° with a narrow bandwidth of 2.6 eV. The measured and simulated reflectivity curves are very close, suggesting that this system has smooth interfaces and low interdiffusion. Owing to the growing importance of lithium and lack of spectroscopic data, we simulate a new Sc/SiC/Al stack based on the reflectivity data and optimize it to perform spectroscopy in the range near the Li K absorption edge around 55 eV, which is in the spectral range of the Li K α emission band. This optimization is achieved by tuning the thicknesses of the different layers and the number of periods of the multilayer using an in-house Python script. The optimization results are compared with the performances of other multilayers employed in the same energy range and at a working angle close to 30° grazing, including Be/Si/Al. This analysis indicates that the Sc/SiC/Al multilayer could be a good candidate for performing spectroscopy in the Li K range.

Keywords: periodic multilayer; X-ray reflectivity; EUV range; lithium; wavelength dispersive spectroscopy; synchrotron radiation; Sc/SiC/Al



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1. Introduction

X-ray wavelength dispersive spectroscopy (WDS) is a fundamental technique for the analysis of the electronic structure and chemical composition of materials essential for a wide range of fields, including materials science, electronics, and geology, etc. This is achieved by diffracting the characteristic X-ray emissions generated after the electron–matter interaction, making use of Bragg reflection by a crystal or a periodic multilayer [1,2]. WDS probes both the core–core (atomic lines) and valence–band–core (emission bands) radiative transitions. The valence–core transition provides information about the occupied valence states, making it sensitive to the local chemical environment of the emitting atom. Such a transition would help in better understanding the operation mechanism of lithium batteries and can be useful in their development, for instance, delivering information on the electrode phase transformation and probing the evolution of the key electronic states during battery operations [3]. The core–core emissions are much less sensitive to the chemical state of the emitting atoms, but are generally much more intense than valence-to-core transitions. Thus, these emissions can be useful in performing quantification, that is to say, in determining the weight fraction of an element in a solid material from the intensity of the characteristic emission emitted by this element.

In the extreme ultra-violet (EUV) energy range from 15 up to 124 eV (10–80 nm), diffraction conditions cannot be attained using natural crystals due to their interplanar

spacings being smaller than the long wavelength of the characteristic radiations emitted by light elements. This amounts to the relatively weak intensities emitted by such elements, arising from their low fluorescence yield [4], and the strong absorption of emissions within the specimen, raising the need for optical elements with a relatively high reflectivity at a working angle suitable for direct integration into scanning electron microscopes (SEM) and electron microprobes apparatus (EPMA) [5]. Let us remark that, at the present time, none of the periodic multilayers used in these commercial SEMs and EPMA are optimized to work in the photon energy range of Li K emissions. The use of non-optimized structures results in weak reflected intensities, leading to long acquisition times and broad emission peaks, making it difficult to disentangle spectral interferences.

To tackle this challenge, optical elements can be realized by using periodic multilayers made of alternating light and heavy layers with optimized periodicity d [6–8]. The constructive interference of multiple reflections from the interfaces is analogous to a Bragg reflection from the crystals. EUV multilayers are already used in astrophysics [9] and lithography [7,10], and they seem to be especially interesting for detecting lithium K emissions around 50 eV [11]. Indeed, the peak positions and shape of the Li K emission band depend on the chemical state of the Li atoms. The detection and quantification of such low-energy emission in WDS reveal the need for specific multilayers presenting a narrow bandwidth in order to obtain a good spectral resolution. If the quantification of lithium is the main objective, and not the determination of the chemical state of the Li atom, then the bandwidth of the diffraction pattern of the dispersive multilayer, which governs the spectral resolution, should be not too large, so that spectral interferences can be avoided, and not too small, so that the collected intensity is large.

A considerable amount of research has been published on the development of multilayers in the EUV range generally designed to work at normal incidence [7,12–14]. Al-based structures such as Al/Mo/B₄C and Al/Mo/SiC achieve a peak reflectance of more than 50% at normal incidence for a photon energy lower than 70 eV [15]. Another example is Be-based multilayers, which prove to be good candidates due to the low absorption of beryllium. For instance, Mo/Be multilayers were reported to measure a peak reflectivity of 58% at 94 eV (13.2 nm) and a spectral bandwidth of 2.5 eV (0.35 nm) [7]. Another interesting system is Be/Si/Al, with a reflectivity of 61% at normal incidence at an energy of 72.5 eV (17.1 nm) and a spectral bandwidth of 1.7 eV (0.4 nm) [7,16]. The same system was also designed, fabricated, and characterized for a grazing angle of 30° [11], which is more suitable for WDS applications in the Li K emission range. However, the use of Be is not recommended in practical designs due to safety precautions, because of the toxicity of inhaled beryllium-containing dust [17]. Similarly, Co/Mg multilayers were developed to work with 50 eV radiation at 45°, and this system reaches a reflectance of 40% [18].

In our research, we are studying the Sc/SiC/Al periodic multilayer. Multilayers containing Sc and Al have already been introduced for applications in attosecond physics, coherent light sources (synchrotrons, EUV lasers, and high-harmonic generation, etc.), and astrophysics [13]. These multilayers show a high reflectance, ranging from 42 to 56% for energies from 30 to 24 eV (40–51 nm) at near normal incidence (5°) with a bandwidth between 2.2 and 2.6 eV. The aging properties of Al/Sc multilayers were also studied, showing a good temporal stability after a period of time of nine months [13]. In the present work, we are interested in the design of multilayer systems for energies from 45 to 55 eV (wavelength between 27.5 and 22.5 nm), corresponding to the lithium K spectral range. We show that the new proposed Sc/SiC/Al multilayer can work efficiently for spectroscopic purposes in the Li K spectral range and avoid the use of previously designed Be/Si/Al multilayers, for which toxicity could be a problem during their preparation, owing to the use of beryllium.

Indeed, nowadays, lithium is a major element in the domain of energy [19,20], but its routine spectroscopic characterization on widely used apparatus, such as scanning electron microscopes or electron microprobes, is not yet possible. In fact, these kinds of apparatus are equipped with energy-dispersive spectrometers that cannot detect low-energy photons

as one of the Li K α emissions or wavelength-dispersive spectrometers (WDS) equipped with periodic multilayers [11,21]. However, up-to-date WDS apparatus is not available with multilayers optimized for the Li K α range. Thus, any progress to obtain spectroscopy in this spectral range is welcome, because it will make it possible to determine the chemical state of lithium atoms, as well as to perform quantification.

2. Experimental Details

In the present work, we analyzed Sc/SiC/Al multilayers of 20 periods of 11.85 nm. The first layer on the substrate is Al, followed by SiC and Sc. Scandium is proposed due to its low absorption in EUV above its M_{2,3} absorption edge at ~28 eV (44 nm), while aluminum presents its L_{2,3} absorption edge near 73 eV (17 nm), making them a good combination for the requested photon energy range. SiC was introduced into the multilayer structure in order to obtain a better reflectivity as compared with the two-component Al/Sc system. It turns out that such a design helps to smooth the interfaces and prevents the development of the Al₃Sc phase that was reported to form at the Al-Sc interface [13].

All the multilayers were deposited via magnetron sputtering at Laboratory Charles Fabry (LCF) of Institut d'Optique Graduate School. The magnetron sputtering system at LCF can accommodate up to four material targets and the deposition process can run either in rf- (radio frequency) or dc- (direct current) mode, according to the nature of the material being deposited. The substrates were Si wafers in the (100) crystal orientation with a surface roughness in the 0.3 nm range. The targets used were pure (99.95%) scandium and silicon carbide, as well as a Si-doped (1.5 wt.%) aluminum. The base pressure in the deposition chamber was lower than 10⁻⁵ Pa. The working gas (argon) pressure was set to 0.27 Pa. We used the dc-mode operation with a 100 mA current for the Sc target and rf-mode for the SiC and Al targets with the powers of 150 and 200 W, respectively. All deposited samples were capped with a few nm thick protection layer of SiC in order to protect the multilayer from oxidation and preserve its reflectivity. In the spectral range of interest, the thickness of the capping layer can even be optimized for increasing the initial value of the peak reflectance by a few percent. A good temporal stability of this type of multilayer has been already proven previously [13]. All samples were initially characterized with a Bruker D8 reflectometer via grazing incidence X-ray reflectivity (GIXR) measurements at grazing incidence at the Cu K α emission line ($\lambda = 0.154$ nm; $E = 8.048$ keV).

EUV reflectivity measurements in the Li K range were performed at the bending magnet BEAR (Bending magnet for emission, Absorption, and Reflectivity) beamline [22,23] at the Elettra synchrotron facility, operating in the 2.8–1600 eV spectral region. An energy scan was performed in the range from 45 to 65 eV with a step of 0.1 eV. The measurements were conducted using the grazing incidence monochromator [22] of the beamline and using an exit slit aperture of 900 × 100 μm^2 . This setup allowed for delivering a spot dimension of about 200 × 100 μm^2 FWHM and an energy bandwidth ΔE of about 0.03 eV, well below the energy bandwidth of the multilayer structure. Higher-order purity was obtained using a 0.1 μm thick Al filter. EUV light was delivered with 0.9° linear polarization, as given by the setting of the aperture of the polarization selector of the beamline.

The reflectivity was measured inside the BEAR UHV end station [22], where the angular resolution and accuracy given by the manipulator and detector goniometers was 0.01°. The reflectivity was evaluated by measuring the current from a silicon diode (IRD SXUV-100 model), positioned in correspondence with the light beam and, in parallel, the current from the last mirror of the beamline, used as an intensity monitor. Both diode and monitor currents were read with two multimeters (Keithley 6517A), used with the appropriate range. The reflectivity was calculated after a standard procedure based on 4 measurements: a reflectivity scan and relative dark measurement of the current of the diode, positioned in correspondence with the reflected beam and in parallel with the monitor current, both functions of energy. These two measurements allowed for the extraction of the quantity $T_R(E)$ given by the ratio between the diode and monitor currents, once the dark current was subtracted.

The I_0 scan and relative dark measurement of the current of the diode positioned in correspondence with the incoming beam after removing the sample, and in parallel with the monitor current, are both functions of energy. Similarly, these two measurements allowed for extracting the quantity $T_0(E)$ given by the ratio between the diode and monitor currents, once the dark current was subtracted. The ratio between $T_R(E)$ and $T_0(E)$ gave the experimental reflectivity.

For the analysis of the reflectivity curves, we used software for modeling the optical properties of multilayer films: the commercial Leptos© package and IMD program [24]. The calculation of the optical functions (reflection and transmission, etc.) of multilayer films is based on the application of Fresnel equations. Fits to the GIXR data allowed us to determine the multilayer period, layer thicknesses, and average values of the interfacial roughness/diffusion. A simulation of the multilayer reflectivity was performed using the same software (IMD) that can be used for modeling in the X-ray range the optical properties, reflectance, transmittance, electric field, and intensities, etc., of multilayer films. Multiple layers can be specified in the software to create a multilayer structure with the desired thickness and roughness. The roughness is defined at each surface/interface to take into account the reduction in reflectance due to imperfections. Detailed information on the use of IMD and Leptos is found in [24,25]. The calculations use the optical constants database by the Center for X-ray Optics (CXRO) [26] in conjunction with a homemade Python script established using the trial and error method for the optimization part with pre-imposed constraints. The calculations are performed either at a fixed photon energy as a function of the glancing angle or at a fixed angle as a function of the photon energy.

3. Results and Discussion

After deposition, the Sc/SiC/Al multilayer was characterized using GIXR in the hard X-ray range at 0.154 nm. The corresponding reflectivity curve and its fit as a function of the thickness and roughness of the various layers are shown in Figure 1. The quality of the multilayer is demonstrated by the large number of Bragg peaks observed up to the 10th order of diffraction. Above the glancing angle of 4° , the Bragg peaks are too weak and cannot escape the background noise: there is no more reflectivity of the multilayer. The fitted layer thicknesses are $d_{Sc} = 1.9$ nm, $d_{SiC} = 1$ nm, and $d_{Al} = 8.95$ nm, with a capping SiC layer of 3 nm thickness. The interfacial roughnesses between the different layers, as well as between the stack and the substrate, comprising both physical and chemical aspects, are limited to between 0.3 and 0.5 nm. The fitted parameters are presented in Table 1.

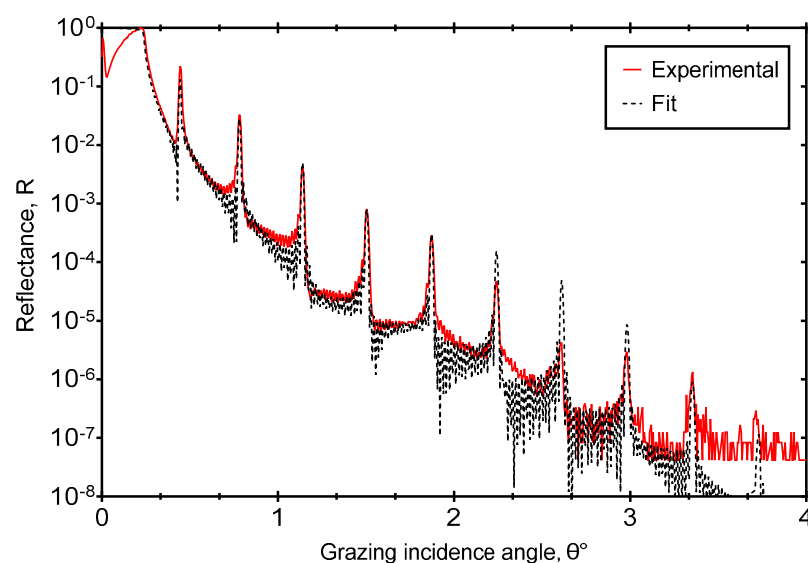


Figure 1. Experimental (red solid line) and fitted (black dashed line) X-ray reflectivity curves of the Al/SiC/Sc sample, measured at a photon energy of 8048 eV (0.154 nm wavelength).

Table 1. Fitted parameters of Al/SiC/Sc multilayer, with d being the layer thicknesses and σ being the interfacial roughnesses. The roughnesses on top of the Si substrate and SiC capping layer are 0.3 and 0.5 nm, respectively. The roughness between the last Sc layer of the multilayer and the SiC capping layer is 0.5 nm.

Multilayer: Al/SiC/Sc			Period: 20		
d_{Sc} (nm)	d_{SiC} (nm)	d_{Al} (nm)	$\sigma[\text{SiC-on-Sc}]$ (nm)	$\sigma[\text{Sc-on-Al}]$ (nm)	$\sigma[\text{SiC-on-Al}]$ (nm)
1.9	1.0	8.95	0.4	0.4	0.5

The reflectivity scan was performed as a function of energy in the EUV range of 45–65 eV (19–27.5 nm) at a fixed near-normal incidence angle of 7° , as shown in Figure 2. The reflectivity curve presents a main peak corresponding to the first-order diffraction of the incident radiation by the periodic structure of the multilayer stack. Some small oscillations can be seen at both sides of this main peak. Figure 2 reveals that the measured reflectance of the multilayer (solid line) presents a strong reflectivity of about 38% at 54.1 eV, and a narrow bandwidth, measured at full width at half maximum, of 2.6 eV. The dashed line represents the simulated reflectivity for an ideal multilayer (with no roughness and no interlayers) using the thicknesses determined above. The maximum of the simulated reflectance obtained is 40.8% at 54.1 eV. The reflectivity difference between the simulated and experimental values is ascribed to the interface imperfections, such as geometrical roughness and interdiffusion, leading to a loss of optical contrast between the different layers and thus to the deterioration of the optical performance of the stack. The experimental data are very consistent with the simulations in terms of reflectance and bandwidth and have been a starting point to evaluate new structures. The possible upgrades to make the multilayer adapted for the analysis of Li K emissions for grazing angles of 30° can be achieved by changing the number of periods, the individual layer thicknesses, and the order of deposition.

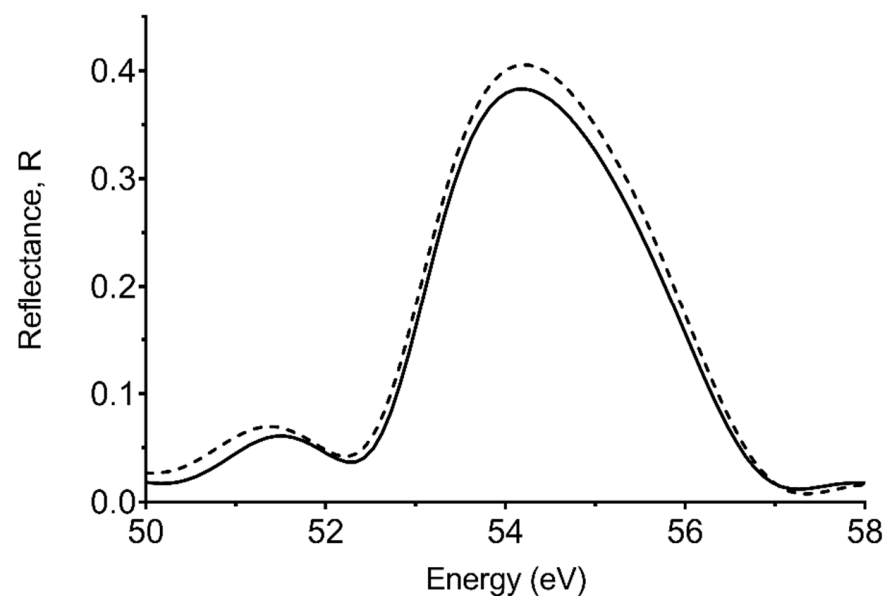


Figure 2. Reflectivity of the Sc/SiC/Al multilayer measured as a function of the incident photon energy at near-normal incidence (solid line) and calculated for the ideal stack (dashed line).

A previous study explored Al/Sc-based EUV multilayers as a function of the number of periods, individual layer thicknesses, and order of deposition [13]. We optimized the Sc/SiC/Al multilayer structure in order to maximize the reflectivity of the multilayer in the vicinity of the Li K-edge. For simulations, we used a sample with 20 periods, which leaves some room to improve the reflectivity by depositing multilayers with a bigger number

of periods up to a saturation value of about 40. As can be seen in Figure 3, displaying reflectivity curves in the 50–58 eV photon energy range and for an incidence angle of 7° , the peak reflectivity increases and the bandwidth decreases as the number of periods in the stack increases. This allows us to obtain a peak reflectance of about 49% and narrower bandwidth (about 2.0 eV), as shown in Figure 3.

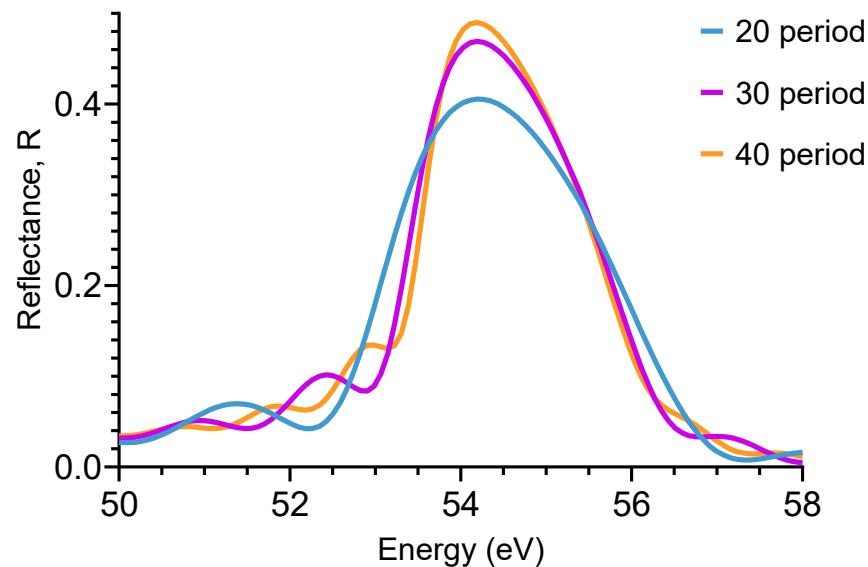


Figure 3. Effect of period number on the Sc/SiC/Al multilayer reflectivity (calculated) in near-normal incidence condition.

The oscillating features observed on both sides of the main peak (50–53 eV and 56–58 eV) are known as Kiessig fringes. They were first reported in 1931 [27] and result from interference between X-rays reflected at the different interfaces (from the vacuum/top layer to the bottom layer/substrate interfaces) of the multilayer stack. The position and spacing of the fringes can be used to determine the thickness of the various layers.

To work in the Li K energy range at a grazing angle of 30° , we increased the multilayer period to $d = 25.2$ nm. This configuration was extracted from a homemade Python script, able to compute X-ray reflectivity for a given set of parameters and geometry and to find the optimal thicknesses of the layers (in our case Sc and Al layers). Three constraints were imposed to reduce the number of combinations:

1. The SiC intermediate layer thickness was restricted to two values: 1 and 1.5 nm;
2. The number of periods was limited to a range between 15 and 40;
3. The thicknesses of Al layers could vary between 1.5 nm and 23 nm, while Sc layers were allowed to vary between 22.8 nm and 1.5 nm, both with an increment of 0.25 nm.

The simulations were conducted considering an ideal multilayer without roughness and interdiffusion. As observed in Figure 2, we expected that the real deposited multilayer would lose a few percent of reflectance with respect to the ideal multilayer. The code gave, as a result, a number of periods of 40, a period equal to 25.2 nm with $d_{\text{Sc}} = 1.8$ nm, $d_{\text{SiC}} = 1.5$ nm, and $d_{\text{Al}} = 21.9$ nm. Figure 4 presents the calculated reflectivity at a fixed glancing angle of 30° as a function of the energy of the incident radiation: the structure maintains a peak reflectance of 35.6% at 55.2 eV, with a bandwidth of 3.9 eV. The latter is wider by 1.3 eV compared to the near-normal incidence design. Additional simulations with an interface roughness of 0.5 nm were conducted, but were not incorporated into the figure due to their negligible impact on the multilayer reflectance (decrease of less than 0.2%).

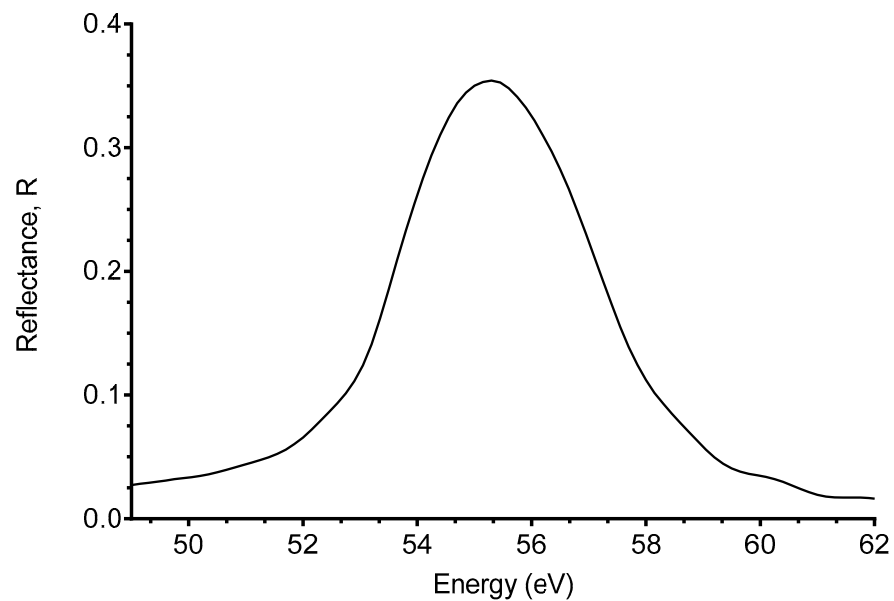


Figure 4. Simulated reflectivity at a 30° grazing angle of the Sc/SiC/Al multilayer optimized for operation in the Li K emission spectral range (for an ideal multilayer) as a function of the incident photon energy.

Figure 5 shows the reflectivity curve of the Sc/SiC/Al multilayer simulated at the fixed incident photon energy of 55.2 eV, which is the position of the maximum observed in Figure 4, as a function of the glancing angle. The same structural parameters of the multilayer as those of the simulation presented in Figure 4 were used. The reflectivity maximum is 35.1% and the bandwidth 2.7° . This reflectivity curve, complementary to the previous curve obtained as a function of the photon energy, confirms the good performance of the multilayer in terms of its reflectance in the spectral range of the Li K emission band.

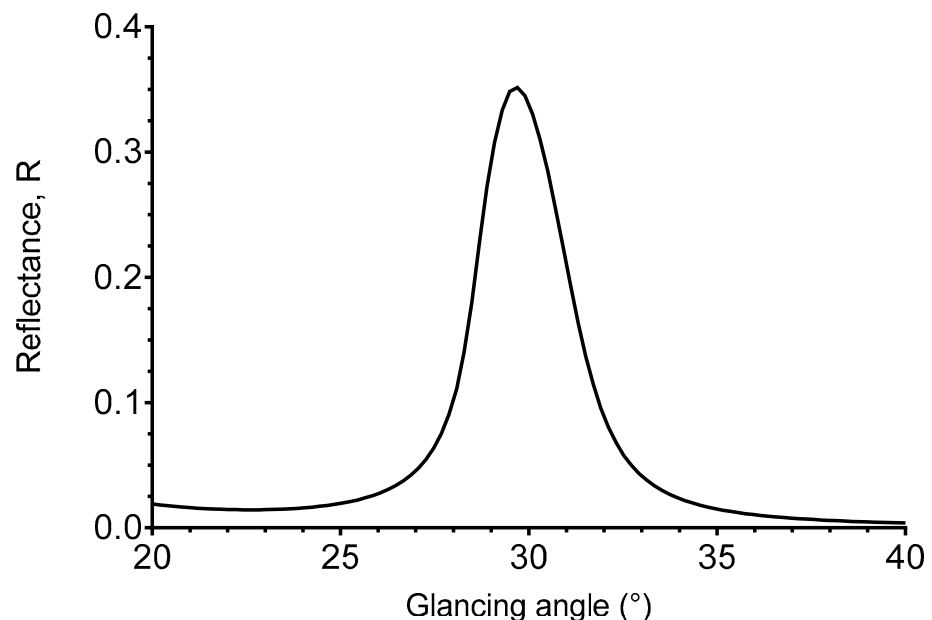


Figure 5. Simulated reflectivity at a 55.2 eV incident photon energy of the Sc/SiC/Al multilayer optimized for operation in the Li K emission spectral range (for an ideal multilayer) as a function of the glancing angle.

Table 2 presents a comparison of the reflectance of various multilayer systems designed to work in a spectral range close to the one of the Li K emission band. The multilayer periods range from 17 to 29 nm. As one can see, the Sc/SiC/Al multilayer has a calculated reflectivity of 35.4% with a bandwidth of 3.9 eV. The Be/Si/Al multilayer [11,16] has a reflectivity of 31.8% with a bandwidth of 3.5 eV; its measured reflectance is 14% lower than the value obtained from the calculations. In the case of the Co/Mg multilayer [18,28], the measured reflectance is reported to be 42.6%, which is 14% less than the calculated value, with a bandwidth of 8 eV. The reflectivity drop for Sc/SiC/Al is expected to be small based on the measurements at near-normal incidence, where the drop did not exceed 4%. This suggests that this system has smoother interfaces and less interdiffusion compared to the other presented multilayers.

Table 2. Comparison of multilayers' characteristics, simulated (R_{sim}) and experimental (R_{exp}) peak reflectance in the range of the Li K emission. θ is the grazing angle.

Multilayer	E, eV	θ , °	d, nm	R_{sim} , %	R_{exp} , %	Bandwidth, eV	Ref.
(Sc/SiC/Al) ₄₀	55.2	30	25.2	35.4	–	3.9	This work
(Be/Si/Al) ₃₀	51.6	30	29	45.8	31.8	3.5	[11]
(Co/Mg) ₃₀	49.6	45	17	56.5	42.6	8.0	[18]

Thus, such a Sc/SiC/Al periodic multilayer can be introduced into the spectrometers equipping commercial SEMs and EPMA. Following the Bragg law, $n\lambda = 2d \sin(\theta)$, where λ is the wavelength of the radiation, d is the period of the stack, θ is the glancing angle of the incident beam on the sample surface, and n is the diffraction order, each emitted wavelength will be dispersed in one direction of the goniometer, corresponding, for the Li K emission, to the middle of the accessible angular range of EPMA. By scanning the angles, the various wavelengths will be sequentially detected and their various intensities collected, enabling the determination of the Li K emission band.

4. Conclusions

We showed that, by optimizing the thickness of the layers constituting a periodic multilayer, as well as the number of periods of the stack, it is possible to turn a structure initially designed to work at near-normal incidence for imaging purposes into a structure working at much lower glancing angles and use it for spectroscopy purposes. The multilayer was first characterized via laboratory-based reflectivity measurements performed in the hard X-ray range and then studied with the use of both synchrotron-based reflectometer and theoretical calculations. The GIXR curve demonstrated the good structural quality of the stack and its fit showed smooth interfaces with roughnesses that did not exceed 0.5 nm.

The reflectivity measurements of the Sc/SiC/Al multilayer in the lithium K emission range revealed a relatively high reflectance of the proposed multilayer and its good spectral selectivity. Indeed, the peak reflectance was nearly 40% at the photon energy of 54 eV at near-normal incidence and the bandwidth was about 2.6 eV. This system optimized for the WDS analysis of Li K emission bands shows that the periodic Sc/SiC/Al multilayer can provide a reflectance of up to 35.4% at 30° and a bandwidth of about 4 eV. The main parameter for the optimization was the change in the multilayer period, fixed to 25.2 nm to work in the Li K spectral range.

Thus, such a new periodic multilayer could be inserted as the dispersive element in bent crystal spectrometers working following the Bragg law and equipping electron microprobes or scanning electron microscopes. They could provide a cheap and simple way for the analysis of lithium in various solid samples: Li-ion batteries, lithium ores, minerals, and light alloys, etc. Owing to the combination of peak reflectance and bandwidth, the proposed Sc/SiC/Al periodic multilayer mirror can be used for the elemental quantification

of the Li K emission band, that is, for the determination of the Li mass fraction, where only the measured intensity is important.

Author Contributions: Conceptualization, K.H. and P.J.; methodology, K.H. and P.J.; software, K.H. and E.M.; validation, K.H., E.M., F.D., A.G. and P.J.; formal analysis, K.H., E.M. and A.G.; investigation, K.H., E.M., F.D., A.G. and P.J.; resources, K.H., E.M., F.D. and A.G.; data curation, K.H., E.M. and A.G.; writing—original draft preparation, K.H., E.M., F.D., A.G. and P.J.; writing—review and editing, K.H., E.M., F.D., A.G. and P.J.; visualization, K.H., E.M. and P.J.; supervision, F.D. and P.J.; project administration, F.D., A.G. and P.J.; funding acquisition, P.J. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The raw data supporting the conclusions of this article will be made available by the authors on request.

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