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Development of Al-based multilayer optics for EUV

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Abstract We report on the development of multilayer optics for the extreme ultra-violet (EUV) range. The optical performance of Al-based multilayer mirrors is discussed with regard to promising reflectivity and selectivity characteristics and the problems of the interfacial roughness for this type of multilayers. We demonstrate a possibility to reduce the average roughness by introducing additional metal layer (W or Mo) rather than depositing a buffer layer at each interface. We have prepared and tested Al/SiC, Al/W/SiC and Al/Mo/SiC multilayers of various periods for the spectral range from 15 to 40 nm, which is the range of increasing interest for high-order harmonic generation, synchrotron radiation and astrophysics. The structure of the three-component systems has been optimized in order to obtain the best reflectivity for each wavelength within the spectral range. We have shown that introduction of refractory metal in Al-based periodic stack can improve the optical performance of multilayer reflecting coatings designed for the EUV applications.

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1 Introduction

The use of multilayer reflecting coatings is practically responding to the need for normal incidence optics in the EUV range. These structures, made of a number of alternatively deposited thin layers of different materials, provide for high normal reflectance due to constructive interference of small fractions of incident light reflected by each interface in accordance with Bragg's law [1].

Remarkable technological progress of multilayer fabrication was mainly inspired by the development of a technology for projection lithography at 13.5 nm, for which Mo/Si multilayers proved to be the most appropriate coating due to high reflectivity (∼70%) and stability of optical characteristics [1, 2]. While further progress of the EUV-lithography is postponed until the problem of an efficient light source at 13.5 nm is solved, the multilayer development is expanding to longer wavelengths. In the wavelength range from 15 to 40 nm, multilayer optical elements and systems are essential for various applications, such as synchrotron radiation, free electron laser, high-order harmonics generation sources, plasma diagnostic, etc. They are also of particular interest for astrophysics, which is running an analysis of numerous emission lines of solar plasma within the EUV range. For instance, the multilayer reflecting coatings were used in a number of instruments for some recent space missions (SOHO/EIT, STEREO/SECCHI, etc.) [3, 4].

Among the reflecting multilayer coatings available for the spectral range between 15 and 40 nm, preference is often given to Mo/Si multilayers as the most technologically advanced. In principle, other material combinations would provide higher reflectance and, sometimes, a narrower bandwidth. The latter is especially important when the selection of a particular one of close emission lines is concerned. The spectral selectivity $\lambda/\Delta\lambda$, which can be attained with Mo/Si

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is about 10 in this spectral range. There were reported various Si-based multilayers showing somewhat better performance in terms of reflectivity than Mo/Si. These are the structures with high absorbing material other than Mo (B_4C, C, Mo_2C, SiC, etc.) [5–7] and three-component multilayers, for instance B_4C/Mo/Si [8]. Two other groups could be constituted of Al- and Mg-based systems, as these materials are less absorbing than Si beyond their absorption edges, which are situated at 17 and 25 nm, respectively. One of the Mg-based multilayers, Mg/SiC, was reported to have been successfully produced and tested [9–11].

In the present paper, we report on the development of EUV multilayer mirrors made with aluminum. Section 2 starts from expectations and problems related to fabrication and characterization of Al-based multilayers. We will discuss the possibility to improve their optical performance via optimization of multilayer deposition process and (in Sect. 3) introduction of a third material into the multilayer structure. We will present and discuss experimental results obtained with some three-component Al-based systems designed for three wavelengths: 17.1, 19.5 and 30.4 nm, corresponding to emission lines of Fe IX-X, Fe XI-XII and He II [12].

2 Al-based multilayers

2.1 Problem of high roughness and reflectance loss

Reflectivity and bandwidth calculations predict a very promising performance for multilayers made of alternatively deposited Al and another material (Mo, Y, Nb, Zr, SiO_2, SiC, B_4C, etc. . . .) in the range from 17 to 40 nm compared to that of Mo/Si (an example of a simulation is shown in Fig. 1). In practice, however, there is a quite limited number of published works which deal with Al-based reflecting coatings for the EUV range. It often happens that if results are not optimistic, they rarely appear in publications. In fact, from the data found in the literature on Al/Nb, Al/SiO_2, Al/Mo, Al/Y and a few others [13–17], one can deduce that there is a problem to realize multilayer mirrors having a reasonably fair performance. To our knowledge, relatively good results in terms of reflectivity and stability of optical characteristics were obtained only for the Al/Zr system [18, 19]. All other structures demonstrated significantly reduced reflectivity compared to that expected from simulations. Moreover, the multilayer reflectance degraded with time.

The most probable reason of the poor performance of the available Al-based multilayer mirrors is the formation of a rough interface between Al and adjacent material as a result of interdiffusion and/or inhomogeneous crystallization of Al. It is also known the ability of Al to form solid solutions with other materials [20]. Thus, it has to avoid using materials, which might have solid-state reactions with Al. Finally, the oxidation of surface layers may also contribute to the reflectivity loss.

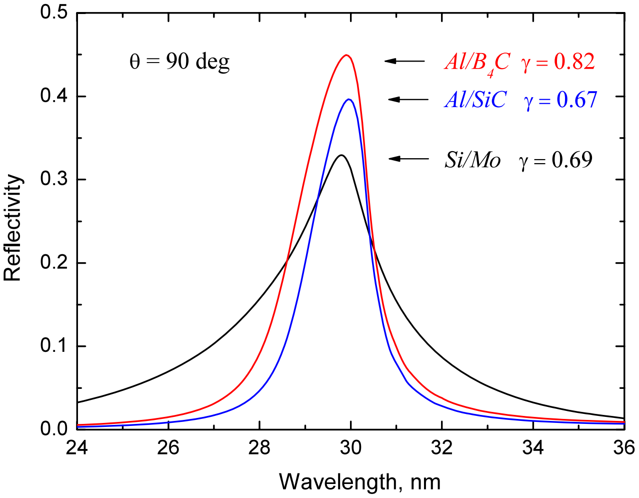

Fig. 1 Theoretical reflectivity of Al/SiC, Al/B_4C and Si/Mo multilayers at normal incidence. All spectra are calculated for the multilayer period $d = 16$ nm, number of periods $N = 50$, thickness ratio $\gamma = d_1/d$ is optimized for each multilayer, zero roughness being assumed

Variation of substrate bias during the multilayer deposition was reported to sharpen the interface and improve the reflectance of Mo/Al multilayers at 18.5 nm from 28 up to 33% [21]. The influence of post-deposition thermal treatment (annealing) on the interface quality in Mo/Al multilayers was studied in [22]. A certain improvement was observed from analysis of the hard x-ray reflectivity curves, which showed additional Bragg peaks appearing after the treatment.

We believed, however, that it might be possible to obtain smoother Al layers throughout optimization of the thin film and multilayer deposition process.

2.2 Optimization of deposition process and characterization of multilayers

Films and multilayers for our study were prepared at *Laboratoire Charles Fabry de l'Institut d'Optique*. A combined rf/dc magnetron sputtering system, which can accommodate up to four targets, is described elsewhere [8, 23]. We have decided to use a silicon doped (1.5%) aluminum target. The idea behind is that the presence of Si even in small proportion would disfavor crystallization of Al and, thus, provide for a smoother film growth. We have studied the influence of various parameters of the magnetron sputtering process (dc- and rf-modes and power, gas pressure, a distance between cathode and substrate) on the quality of the deposited films and multilayers. All the samples were routinely characterized by using grazing incidence x-ray (Cu k_α, $\lambda = 0.154$ nm) reflectivity (GIXR). We have deduced their structural parameters from fits to the GIXR data:

the film thickness or the multilayer period and values of average roughness. Finally, we run the process in *rf*-mode at a working gas (Ar) pressure of 2 mTorr. The power applied was 150 W and the substrate (Si wafer) was placed at 10 cm from the target. The optimization of the sputtering process has allowed us to grow relatively smooth Al films. We found the average roughness values of a few tens-of-nanometers-thick Al films in the order of 1 nm, which is approximately a factor two smaller than we had before with pure Al target. We kept the parameters defined for Al and proceeded to the multilayers deposition. For other targets that we used to fabricate various Al-based multilayers, the deposition parameters were also revised. Eventually, the B_4C and SiC targets were operated in *rf*-mode (both at 150 W). For the multilayers reported in Sect. 3, we utilized the Mo and W targets in *dc*-mode (0.06 and 0.07 A).

At-wavelength characterization was performed by using EUV reflectometer installed at *LIXAM* [24]. The reflectometer is equipped with a laser-plasma source based on the laser beam (YAG:Nd 2ω, $\lambda = 532$ nm, $E = 400$ mJ, $\tau = 5$ ns) interaction with a target made of copper foil. The source delivers single shots to a monochromator selecting radiation in the range from 8 to 50 nm. A low repetition rate (1 shot per second) makes it difficult to obtain a precise calibration of the intensity of incident beam and, thus, the multilayer response is measured with relatively large error bars ($\pm 5\%$). Nevertheless, the EUV reflectivity measurements at normal incidence have allowed us to make a pre-selection of samples for absolute calibration with synchrotron radiation, which has been realized later on, during an experimental run at *BEAR* beamline of *Elettra Sincrotrone Trieste* [25]. The absolute uncertainty of the peak reflectance measured at *Elettra* is less than 0.5%.

2.3 Al/SiC and Al/B_4C multilayers

For an initial study, we have selected two systems: Al/SiC and Al/B_4C with the period d in the range from 9 to 16 nm, designed according to our calculations of their reflectivity at about 17, 19 and 30 nm, which were made using the *IMD* program [26]. Refraction indices of the materials were taken from a database of optical constants integrated in the *IMD* package. The simulations predict that the peak reflectance of Al/SiC and Al/B_4C is achieved in the range of $\gamma = 0.6$–0.7 (the ratio between the individual Al layer thickness and the multilayer period). The calculated reflectivity saturates with approximately 60 bi-layers in case of a short period ($d = 9 \div 10$ nm) and with about 30 bi-layers in case of multilayers having the period $d \sim 16$ nm.

Several samples of different period numbers (from 10 to 40) and various thicknesses of individual layers were fabricated in order to study an influence of interfacial roughness as a function of the number of bi-layers and the thickness ratio on the multilayers reflectivity and selectivity. Experimental results are summarized in Table 1. The best results obtained with Al/SiC multilayers are similar to those of the Mo/Si system [4] in terms of EUV reflectivity and superior in terms of spectral selectivity. In spite of the higher theoretical reflectivity of Al/B_4C, its real performance turned out to be worse. Obviously, the roughness problem for this system is more pronounced than for Al/SiC.

We estimated the interfacial and surface roughness from GIXR and atomic force microscopy (AFM) measurements. From analysis of the GIXR data, it was found that the interfaces SiC(B_4C)-on-Al and Al-on-SiC(B_4C) are not symmetrical and can be characterized with different values of roughness. The latter interface is more problematic than the former. For the surface roughness values determined by AFM, we obtained ~1.2 nm *rms* for one of the Al/SiC multilayers (25 periods of 15.5 nm) and nearly 2 nm *rms* for the Al/B_4C sample (25 periods of 15 nm).

As a matter of fact, the interfacial roughness increases with thickness of the individual Al layers. Moreover, it accumulates with the number of bi-layers. We observed that starting from a certain period number, which is still far from the saturation value, the multilayer reflectance does not increase any more but rather decreases at the expense of rapidly increasing interfacial roughness. Both Al/SiC and

Table 1 The parameters and optical properties of Al/SiC and Al/B_4C multilayers measured at 80 deg. Here, the first interface is SiC-on-Al (or B_4C-on-Al), while the second one is Al-on-SiC (or Al-on-B_4C)

Multilayer type	Al/SiC					Al/B_4C
Period d, nm	8.8	10.0	15.9	15.5	16.0	14.9
Period number	40	40	10	25	40	25
Al thickness ratio γ (d_{Al}/d)	0.61	0.64	0.66	0.68	0.72	0.72
Roughness at 1st interface, nm	0.6	0.6	0.4	1.0	1.1	1.1
Roughness at 2nd interface, nm	1.0	1.0	0.7	1.3	2.3	2.1
Reflectance peak position, nm	17.2	19.1	29.3	28.7	29.4	27.0
Reflectivity, %	37.8	28.0	18.6	22.8	8.2	11.2
Selectivity, $\lambda/\Delta\lambda$	57	36	12	22	29	24

Al/B$_4$C multilayers have the same feature but the latter is usually rougher. For the time being, we have decided to focus on the Al/SiC system looking for different ways to improve its optical performance.

3 Three-component Al-based structures

3.1 Reasoning and optimization

When the problem of roughness in multilayers is concerned, the addition of a thin barrier layer between two materials often helps to obtain a sharper interface as it might prevent interdiffusion and formation of intermixing zone [27–29]. Here one has the option of applying the same or even different buffer materials on both sides of one layer. The materials' selection is made according to their physico-chemical and optical properties. The influence of thin ($\sim 0.3 \div 0.5$ nm) amorphous carbon layers deposited between the metal layers on the reflectance and stability of the Al/Mo and Al/Y systems was studied more than 10 years ago [17]. To our disappointment, no significant improvement was reported on the use of barrier layers in Al-based multilayers then and later on.

On the other hand, it has been shown, both theoretically and experimentally, that a periodical structure made of three (or even four) materials can be a more efficient reflector to some extent than the classical two-material multilayer [8, 30–33]. In case of multi-component structure, the number of periods needed to attain the maximum theoretical reflectance is normally smaller than in the two-material one, because of a bigger number of interfaces contributing to the multilayer reflectivity. It is particularly important if the deposition process produces no smoothing effect. Therefore, we have preferred to introduce an additional material in the multilayer structure design. The idea is twofold since it could help to fabricate multilayers having a sharper interface and higher theoretical reflectance.

Unfortunately, the theory does not give a direct indication on how the design of such a multi-component periodic system can be realized in the wavelength range of our interest. Logically thinking, the third material must provide for a higher contrast of refraction indices; but being the most absorbing, it should not completely replace one of the other materials. Obviously, the design needs optimization in order to arrange the materials and to determine the thicknesses of individual layers and the number of periods, which would yield the multilayer peak reflectance at a given wavelength. A special code, which exploits the optimization function from the *Matlab* toolbox, was developed for this purpose. An iterative algorithm applied to calculate the reflectivity of multi-component multilayers, was described previously in [8].

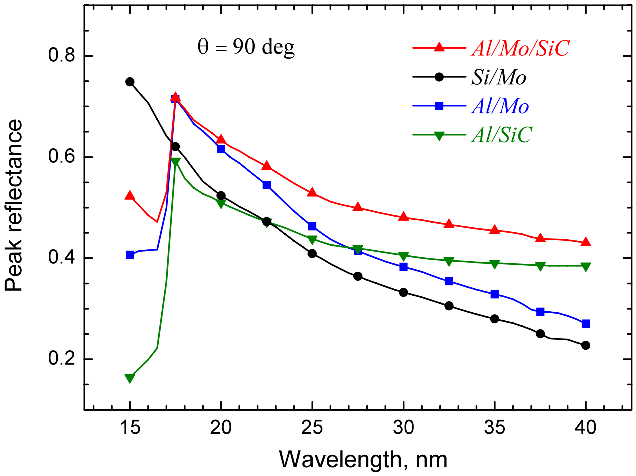

Fig. 2 Peak theoretical reflectance of Si/Mo, Al/Mo, Al/SiC and Al/Mo/SiC multilayers at normal incidence in the wavelength range from 15 to 40 nm. Calculations are performed for perfect structures (zero roughness) of 50 periods. For clarity reasons, the results are plotted with the step of 2.5 nm

We have selected refractory metals W or Mo as the high absorbing material to introduce in the multilayer structure along with Al and SiC. The calculations have been made for various combinations of Al, W and SiC in the spectral range from 15 to 40 nm with a step of 0.5 nm. The same procedure was performed for Al, Mo and SiC. The best reflectivity is expected to yield with the Al/Mo/SiC system (the order of deposition is given from left to right). Results for a perfect (zero roughness) Al/Mo/SiC multilayer of 50 periods are presented in Fig. 2 along with those calculated for few two-component systems for comparison: Al/SiC, Al/Mo and Si/Mo. The maximum reflectance of Al/W/SiC (not shown here) was found to be a few percent lower than that of Al/Mo/SiC in the whole range. It is also inferior to Al/Mo at $\lambda < 22.8$ nm.

The spectral selectivity of both Al/Mo/SiC and Al/W/SiC is not as good as one can achieve with the Al/SiC system. Nevertheless, the new three-component multilayers are expected to have about 20 to 30% more narrow spectral bandpass than Mo/Si multilayers in the range of our interest.

3.2 Experimental results and discussion

Based on the above reasoning, we have prepared a set of Al/W/SiC and Al/Mo/SiC multilayers. The GIXR results revealed spectacularly different behavior of angular-dependent x-ray reflectivity of three-component multilayers compare to the Al/SiC samples of similar periods. The distinctive features of the GIXR curves are the superior intensity and the bigger number of Bragg's peaks of higher orders. A typical example of a comparison, Al/Mo/SiC versus Al/SiC, is shown in Fig. 3. Structural parameters of the multilayers deduced from fits to the measured θ–2θ curves are given in the caption to Fig. 3 and in Tables 1, 2. Their analysis supports the conclusion on the reduced average roughness.

As in case of two-component multilayers, we have selected a number of three-component samples with the periods of 9, 10 and 16 nm for *at-wavelength* characterization at normal incidence. Dedicated EUV reflectivity measurements were performed at the same *LIXAM* facility and at *BEAR* beamline of *Elettra*. The results are summarized in Table 2. Among the short-period multilayers, the Al/Mo/SiC samples exhibit reasonably good reflectance, which attains nearly 50% at 17.3 nm and more than 45% at 19.2 nm. At the long wavelength end of the region of our interest, around 30 nm, the better reflectance of ∼30% is measured with one of the Al/W/SiC structures. The experimentally determined spectral selectivity is consistent with the calculated one.

Representative examples of measured and simulated EUV reflectivity spectra of Al/Mo/SiC and Al/W/SiC multilayers are shown in Figs. 4 and 5. A lack of good agreement between theoretical and experimental data was observed, especially for the short-period multilayers. In the first approximation, the theoretical reflectivity of the multilayers was calculated with values of the layers thickness and roughness issued from analysis of the GIXR data, while the roughness impact on the normal incidence reflectivity may vary depending on the morphology of imperfections at the interfaces. Moreover, there are other important aspects to account for while simulating the performance of reflecting multilayer coating designed for various EUV applications.

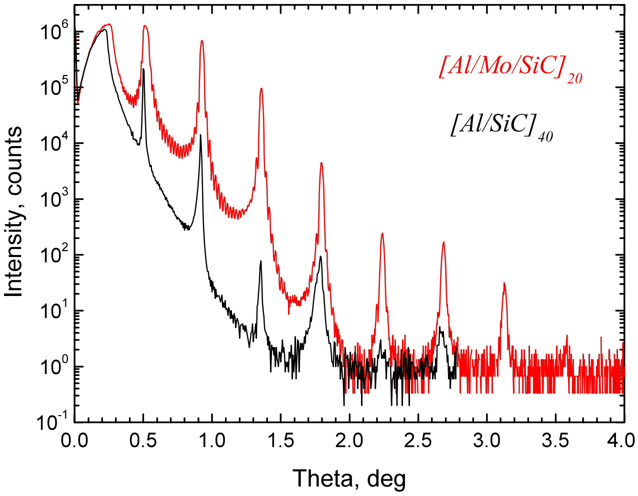

Fig. 3 Grazing incidence x-ray reflectivity curves of two Al-based multilayers: Al/SiC (40 periods of 10.0 nm) and Al/Mo/SiC (20 periods of 9.9 nm)

Foremost, accurate knowledge of the optical constants of the materials is needed. In the EUV range, their experimental determination is quite a difficult task because of strong absorption of the radiation by nearly all materials [34]. This is why the data reliability and accuracy varies from source to source.

On the other hand, the optical properties of the materials in thin film or multilayer form depend on their microstructure. For instance, the state of a metal layer may change from amorphous to crystalline with the layer thickness [35]. It is known that the density of thin amorphous Mo films (<2 nm) can be as low as just $80 \div 90\%$ with respect to the bulk Mo density [36]. Therefore, the refractive index of such a reduced density Mo film differs from that of the bulk crystalline material, which is usually available for reflectivity calculations.

In both Al/W/SiC and Al/Mo/SiC multilayers, the high absorbing layers, Mo or W, are rather thin (in the order of 2 nm). While in case of the W layer this value is already beyond the crystallization threshold, the Mo layers are most probably in the amorphous state. This can explain the overall reduction of the reflectivity of Al/Mo/SiC multilayers with respect to calculated values and the better reflectivity of Al/W/SiC at about 30 nm.

Another aspect to consider is the possible formation of oxide layer on top of the stack upon exposure to air. In case of the top SiC layer, its native oxide is SiO_2, though there is experimental evidence that oxidation of SiC could result in mixed oxide products containing C species [37]. The oxygen might also penetrate into underlying layers (Mo, Al) throughout the SiC layer, which is very thin (∼0.8 nm) in the short-period multilayers.

Table 2 Structural parameters and optical properties of Al/Mo/SiC and Al/W/SiC multilayers measured at near-normal incidence ($\theta = 80$ deg). Here, the individual layer thicknesses are given in the order of deposition, and the order of interfaces is Mo (or W)-on-Al/SiC-on-Mo (or W)/Al-on-SiC

Multilayer type	Period, nm	Period number	Layers thickness, nm	RMS roughness, nm	Peak position, nm	Selectivity, $\lambda/\Delta\lambda$	Reflectivity, %
Al/Mo/SiC	16.4	15	11.2/1.7/3.5	0.9/1.2/1.0	30.2	13	28.8
	9.9	20	7.4/1.7/0.8	0.6/0.8/0.6	19.2	16	45.7
	8.9	20	6.3/1.8/0.8	0.6/0.8/0.6	17.3	21	49.2
Al/W/SiC	16.1	15	10.1/2.0/4.0	0.9/1.1/0.9	29.9	15	30.0
	9.7	25	5.9/2.0/1.8	0.9/1.2/1.0	18.6	16	25.9

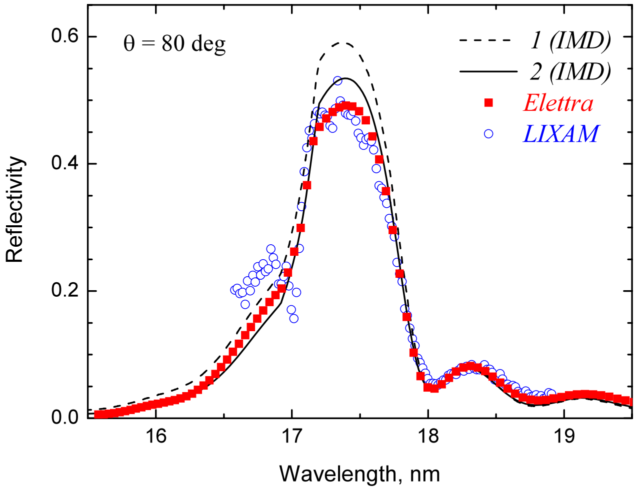

Fig. 4 Reflectivity of Al/Mo/SiC multilayer (20 periods of 8.9 nm) measured with EUV laser-plasma source at *LIXAM* (*open circles*) and with synchrotron radiation at *Elettra* (*squares*). The theoretical reflectivity is calculated using the *IMD* program: for the multilayer parameters determined from GIXR measurements (*dashed line*—1); under the assumption of a 10% reduced density of the Mo layers and replacement of the outermost SiC layer by a 1.5 nm thin SiO_2 layer (*solid line*—2)

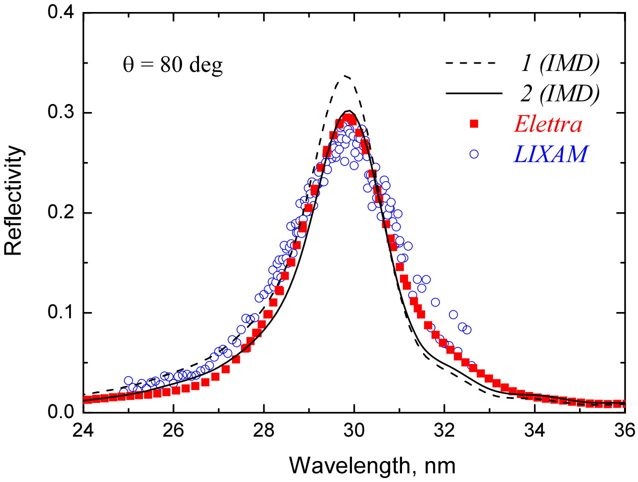

Fig. 5 Reflectivity of Al/W/SiC multilayer (15 periods of 16.1 nm) measured with EUV laser-plasma source at *LIXAM* (*open circles*) and with synchrotron radiation at *Elettra* (*squares*). The theoretical reflectivity is calculated using the *IMD* program: no oxidation of the outermost SiC layer (*dashed line*—1) and its partial oxidation with formation of a 2 nm thin SiO_2 layer on top of the stack (*solid line*—2)

To support the hypotheses mentioned above, we have simulated the multilayer reflectivity with reduced (by about 10%) density of Mo and/or with totally or partially oxidized outermost SiC layer. We considered the formation of the SiO_2 layer of various thicknesses (up to 2 nm according to the density ratio between SiC and SiO_2) on top of the multilayer stack. The resulting simulated spectra that now match better with the experimental data are shown in Figs. 4 and 5.

In the future design of three-component Al-based multilayers we plan to introduce and optimize a capping layer by taking into account the material oxidation as reported, for instance, in [23].

The possible formation of chemical compounds can be detected by x-ray emission spectroscopy (XES). This technique was employed to determine the chemical state of the Al and Si atoms presented within the multilayer structures. No change of the state was observed from XES analysis of the shape of Al K_β and Si K_β emission bands [38] and further study is in progress.

4 Conclusion

We have reported on the development of Al-based two- and three-component multilayers for the EUV range. The particular problem is the large roughness of Al layers associated with their polycrystalline structure. Therefore, we have started from optimization of the deposition process, which has allowed us to grow relatively smooth Al films. However, we have not succeeded to fabricate two-component multilayers Al/SiC and Al/B_4C of decently good optical quality, because the problem of high interfacial roughness was still significant. In order to improve the EUV performance of Al-based coatings, we have proposed to introduce an additional material (W or Mo) in the basic structure (Al/SiC) design. The multilayer parameters providing maximum theoretical reflectance of three-component multilayers in the range from 15 to 40 nm were determined prior to experiment. The order of deposition, layer thicknesses and the number of periods were selected in accordance with the results of optimization. For the first time, Al/W/SiC and Al/Mo/SiC multilayer mirrors for three wavelengths: 17, 19 and 30 nm, have been produced and characterized.

The conclusion on reduced average roughness of the new three-component Al/W/SiC and Al/Mo/SiC multilayers is drawn from analysis of the GIXR data. The EUV reflectance measurements have revealed significantly improved performance of the three-component system compare to the two-component one of the same period. The measured peak reflectance as high as 45.7 and 49.2% at near-to-normal incidence is achieved with Al/Mo/SiC multilayers designed for 19 and 17 nm. For both Al/Mo/SiC and Al/W/SiC we find the peak reflectance values of about 30% at 30 nm.

After the deposition, all samples were stocked in air during a few months before being measured with synchrotron radiation. During this period, no evolution of the multilayer parameters was observed by GIXR measurements. Nevertheless, we agree that a longer period of observation is needed to check the stability of the optical properties of these new Al-based systems.

According to simulations of the EUV reflectivity, the replacement of Mo (or W) and SiC with some other materials can be interesting from a practical point of view. We would also suggest to apply the three-component approach to the design of Mg-based multilayers for the wavelength range $\lambda > 25$ nm.

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