

Stable high-reflection Be/Mg multilayer mirrors for solar astronomy at 30.4 nm

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The He-II ($\lambda=30.4$ nm) emission line is one of the spectral channels chosen to study solar corona. This paper reports on investigations of novel Be/Mg multilayer coatings which, when incorporated beneath a protective bilayer of aluminium and beryllium, ensure particularly high reflection coefficients of up to 56%, a spectral width of $\Delta\lambda=1.6$ nm ($\lambda/\Delta\lambda\approx 20$) and high temporal stability. © 2018 Optical Society of America

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The transition layer from the solar chromosphere to the corona is characterized by a sharp temperature increase (10^4 to 10^6 K). This transition layer is characterized by intense emissions of various ions whose excitation energies belong to this temperature range. The emissions are dominated by the lines of the He-II ions that are found in the lowermost areas of the transition layer (a region with temperatures around 8×10^4 K).

Some of the most important problems in solar physics is understanding the mechanism of heating of the solar corona and how the principal temperature gradient occurs in the transition layer. Thus, most orbital solar missions are foreseen on recording radiation from the He-II ion ($\lambda=30.4$ nm). Such solar missions include, for example, SDO, STEREO, SOHO, and TESIS. It is also planned to make available 30.4 nm recording channels in future missions, such as those of the Solar Orbiter [1], ARKA [2], and KORTES [3] probes.

The optical basis of the telescopes in the observatories under consideration is normal-incidence multilayer mirrors (MLMs). Most often, a Mo/Si structure [4,5] is used for such MLMs. The

peak reflectance of these MLMs $R\approx 25\%$, at $\lambda=30.4$ nm while the spectral width (FWHM) of the reflection peak is $\Delta\lambda>3$ nm.

Such a large value of $\Delta\lambda$ leads to the issue that, along with the radiation of the target He-II ion, the detector also records signals from the spectral lines of the FeXV ($\lambda=28.4$ nm) and FeXVI ($\lambda=33.5$ nm) ions. FeXV and FeXVI ions are formed in the outer regions of the solar corona at temperatures of around 2.5×10^6 K and, therefore, represent processes occurring in that region, rather than in the transition layer.

Another significant disadvantage of Mo/Si MLMs is their high (up to 50%) second-order reflectivity that falls to the neighborhood of the 17 nm wavelength. This area of the solar spectrum is dominated by the emission line of the FeIX ($\lambda=17.1$ nm) and FeX ($\lambda=17.5$ nm) ions; radiation that is formed at the boundary between the corona and the transition layer at temperatures of around 10^6 K.

The paper [6] reports on multilayer Al/Mo/B₄C MLMs. The peak reflectance of this MLMs at wavelength 30.4 nm was 42% with a FWHM comparable with Mo/Si ($\Delta\lambda> 3$ nm). The second reflection order of such mirrors exceeds 20%. There is also a long-term stability of the reflection characteristics of these MLMs. However, due to a large value of $\Delta\lambda$ this structure does not effectively suppress the emission of the FeXV and FeXVI ions lines.

Magnesium-containing MLMs have been examined as alternative structures. Magnesium, the absorption edge of which is at $\lambda_c=25$ nm, is one of the most transparent materials at wavelengths greater than 25 nm. Therefore, multilayer mirrors based on Mg can potentially combine both a high reflectivity and spectral selectivity. The problem of second-order reflection is also solved due to the fact that in the spectral range of $\lambda<25$ nm Mg has high absorption and magnesium-containing MLMs have low reflectance.

Theoretically, SiC/Mg, B₄C/Mg, and Si/Mg MLMs have reflectivities $\sim 60\%$. Such mirrors have been studied

experimentally, e.g. in [4,7-9]. In each case, two points were evident. First: there were significant differences between the experimental data and the theoretical calculations. Second: there was the poor temporal stability of the reflective characteristics. In particular, as reported in [7], it was not even possible to determine the actual value of the reflectivity R immediately after the mirrors deposition. At the time of measurement, the reflectance of the Si/Mg MLMs had decreased to $R=5.6\%$ at $\lambda/\Delta\lambda=22$, and for B_4C/Mg to $R=0.2\%$ at $\lambda/\Delta\lambda=16$. By contrast, SiC/Mg MLMs are more stable; with an initial reflectance $R=42-44\%$ ($\lambda/\Delta\lambda\sim 20$), it only decreased to 30% over the five years of recording reported in [9].

The problem of the difference between theoretical predictions and experimental data is most frequently attributed to the effect of intermixing of materials at the interfaces. Barrier layers have been used successfully to counter such intermixing [4, 10, 11]. To date, the best combination of peak reflectivity and spectral bandwidth has been provided by the four-component Si/ B_4C /Mg/Cr structure proposed in [4], and further studied in [12]. According to [12], immediately after deposition, this MLM had values of $R=46\%$ and $\Delta\lambda=1$ nm. However, due to oxidation, during the first year, the value of R fell to 30%. It then remained at this level over several years of successive monitoring.

Thus, the use of barrier layers only partially resolves the problems associated with degradation of the reflectance characteristics over time. In this case, the determining factor is the high chemical reactivity of magnesium, particularly its susceptibility to oxidation.

In [9, 13], the problem of the temporal stability of the reflective characteristics of magnesium-containing MLMs is solved by deposition a capping layer that prevents oxidation of structures. The authors investigated the coating of Al, Si and SiC. The deposition of capping layers initially leads to a slight decrease in the reflection coefficient. Nevertheless, the reflectance of protected MLM after long storage is significantly higher than that of uncoated structure.

However, the problem of obtaining desired reflective characteristics, combining high reflection and spectral selectivity, makes us look for new solutions.

As an attempt to fully or partially solve the above-mentioned issues of MLMs for solar missions at the He-II emission lines, this paper is devoted to studying the reflective properties of Be/Mg MLMs optimized for the wavelength of 30.4 nm. Special attention is paid to the temporal stability of the reflection characteristics.

Beryllium-based mirrors have long attracted attention, primarily due to the theoretical possibility of providing a reflectivity above 70% near the absorption edge of this element ($\lambda = 11.1$ nm) [14-16]. As a result of the studies, a peak reflection coefficient was reached at this wavelength of 70.2% with a theoretical limit of 75.6%. The possibility of reaching 72% at a wavelength of 13.5 nm was demonstrated in [17] for Mo/Be/Si mirrors.

The prospects of using beryllium as the scattering material in the range $\lambda > 17$ nm were first stated in [18]. Fig. 1 shows the spectral dependencies of the real (δ) and imaginary (β) additions to the index of refraction ($n=1-\delta+i\beta$) of the most frequently used scattering materials for magnesium-containing mirrors, compared to beryllium [19].

From these dependencies, it follows that beryllium, with its absorption close to that of silicon, is superior in terms of optical contrast to magnesium $\delta_{\text{scatter}}-\delta_{\text{spacer}}$. Although silicon carbide

features better scattering ability, its absorption is significantly higher than that of beryllium. Thus, based on this brief analysis of optical properties, beryllium appears to be the most attractive scattering material for use in magnesium-containing MLMs.

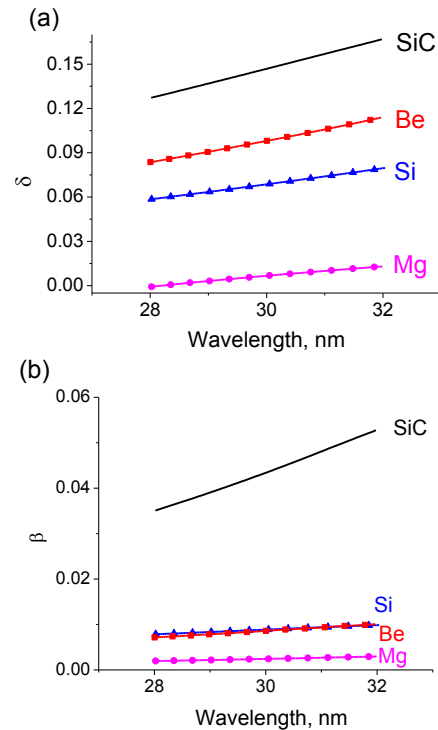


Fig. 1. Spectral dependencies of the index of refraction ($n=1-\delta+i\beta$) for real (a) and imaginary (b) additions.

The reflectivity curves for the wavelength range of 15–35 nm are calculated for the simulated ideal periodic structures Mo/Si, Al/Mo/ B_4C , Si/Mg, SiC/Mg и Be/Mg (Fig. 2). The layer thicknesses are optimized for the maximum reflectance at 30.4 nm (1st reflection order). The number of periods $N=60$; the incidence is normal. In addition, the figure shows the emission lines positions for some ions of the solar corona.

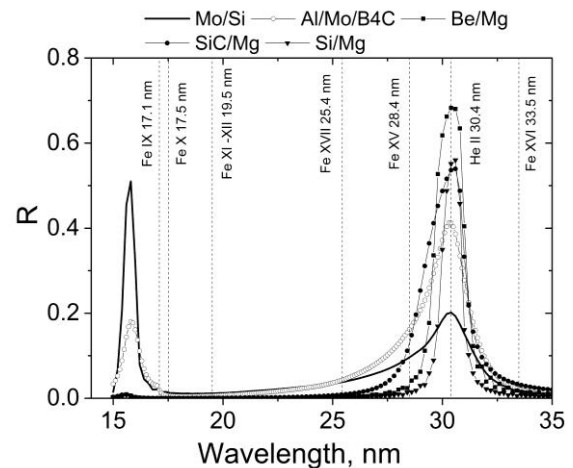


Fig. 2. Calculated spectral dependencies of the reflection coefficients of ideal Mo/Si, Al/Mo/ B_4C , Si/Mg, SiC/Mg and Be/Mg MLMs optimized for maximum reflectance at $\lambda=30.4$ nm. The periods number is $N=60$.

Mo/Si MLM have the smallest reflectance (about 20%) and the worst spectral selectivity ($\lambda/\Delta\lambda < 10$) at a wavelength of 30.4 nm. Better spectral selectivity is characteristic of the Si/Mg MLM (reflectivity at wavelengths of 28.4 nm and 33.5 nm are 1.3% and 0.5%, respectively). However, its peak reflection is considerably inferior to that of Be/Mg (56.8% vs. 68.8%). The reflectances of the Be/Mg MLM at wavelengths of 28.4 nm and 33.5 nm are 2.3% and 1.6%, respectively, which are also acceptable for practical use.

Obviously, the second reflection order of magnesium-free MLMs is high, while for magnesium-containing MLMs the second reflection order does not exceed 1%. Thus, the use of Mg makes it possible not to take into account the lines of the corresponding ions and not to develop special absorption filters.

Be-containing MLMs were deposited in a specially certified laboratory, since beryllium is a highly toxic material. Beryllium dust constitutes a threat to human health, and its effects have an accumulative nature. As far as deposited MLMs do not produce small Be particles that can be breathed in, according to health and safety standards, the storage, research and long-term operation of Be-containing MLMs are not harmful to human health, and these activities do not require special precautions [20].

Following the above theoretical analysis, multilayer mirrors were deposited onto super smooth (roughness RMS value of 0.1–0.2 nm) silicon substrates by means of magnetron sputtering. The sputtering was performed in argon environment with 99.998% purity at a pressure of 0.1 Pa. The residual gas pressure did not exceed 10^{-4} Pa.

The parameters of the structures (period, individual layer thickness, material density, and interlayer roughness) were determined by model to the measurements obtained at Philips X'Pert Pro diffractometer at a wavelength of $\lambda = 0.154$ nm. For fitting the reflectivity curves, IMD [21] software and an extended model were used [22].

Measurements in the vicinity of the 30.4 nm wavelength were performed both in laboratory and at the BESSY-II synchrotron radiation centre. A laboratory reflectometer with an LHT-30 grating spectrometer-monochromator (range: 25–200 nm; spectral resolution 0.1 nm) [23] and a gas-discharge radiation source were used. Synchrotron measurements in the EUV range were performed at the Optics Beamline of BESSY-II with an 11-axis reflectometer [24]. The angular and spectral reflectivity dependencies for different MLMs were studied with fixed photon energy and with a fixed angle of the incident radiation.

Great attention was paid to issues of temporal stability of the reflective characteristics of the Be/Mg MLM. To assess this, between measurements some samples were stored in vacuum (pressure of the residual atmosphere about 100 Pa), while other samples were stored in room conditions.

Initially, two-component Be/Mg structures were studied. The parameters of the MLMs, obtained from reflectometry fits (thicknesses of individual layers and roughness) are: $d_{\text{Be}} = 6.51$ nm, $d_{\text{Mg}} = 9.57$ nm, $\sigma(\text{Be-on-Mg}) = 0.35$ nm, $\sigma(\text{Mg-on-Be}) = 0.85$ nm.

The reflectance of as-deposited structure at a wavelength of 30.4 nm was 49% (angle of incidence of the radiation used was 5°). The significant difference between theoretically predicted and experimentally obtained reflection values may be explained by the presence of oxides in the structure. The temporal stability of the reflection characteristics of the $[\text{Be}/\text{Mg}] \times 60 / \text{Be}_{6.5\text{nm}}$ MLM is illustrated in Fig. 3, which shows the temporal dependence of the

peak reflectance at a $\lambda = 30.4$ nm for two MLMs; one sample was stored at room conditions, and one stored in vacuum.

As in the previously studied case of magnesium-containing structures, a degradation of the reflectivity over time was observed. After 9 months it decreased from 49% to 30%. Based on the higher stability of the sample stored under vacuum, it can be concluded that the key factor resulting in degradation of the reflection coefficient is oxidation.

We studied several materials as the protective coatings. Initially, a top beryllium layer that was increased to 10 nm was tried. However, such a film did not perform the required protective functions: the reflectance of $[\text{Be}/\text{Mg}] \times 60 / \text{Be}_{10\text{nm}}$ MLM decreased by 2% during the month of observations.

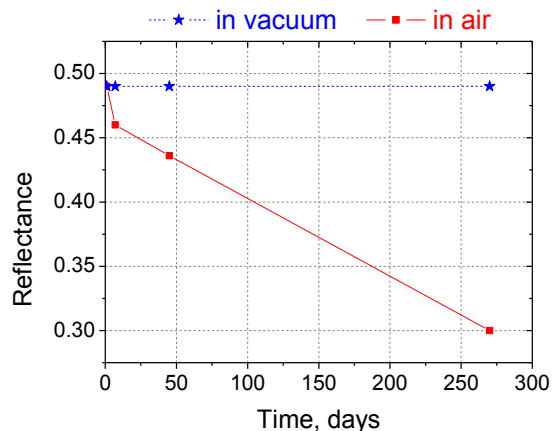


Fig. 3. Time dependence of uncoated $[\text{Be}/\text{Mg}] \times 60 / \text{Be}_{6.5\text{nm}}$ MLM reflectance at a wavelength of 30.4 nm for samples stored at room conditions (squares) and under vacuum (asterisks).

Neither did a MoSi₂ film provide sufficient protection. Tests with films 3 nm and 5 nm thick were carried out. In the first case, the reflectivity loss over the month reached to 4%, while, in the second case, the loss was 1%. However, after nine months of observation, the loss exceeded 15% in both cases.

As a protective layer, the best results showed a relatively thick (13 nm) Al film, deposited on the top beryllium layer of standard thickness (6.5 nm). If the top layer was Mg, deposition of Al film on the top did not give the desired effect. Al and Mg do not form a sharp boundary, which serves as a stop layer to penetrate deep of oxygen. Our research shows that the process of mixing aluminum and magnesium at the border continues for a long time (days and weeks).

As shown in [19], due to the crystallization of beryllium and aluminum layers in Be/Al MLMs it formed a rough (σ to 1.3 nm), but stable in time (the result of two-year observations of experimental samples) boundary. We believe that it turns out to be impervious to oxygen.

Studying the temporal stability of the reflection characteristics of $[\text{Be}/\text{Mg}] \times 60 / \text{Be}_{6.5\text{nm}} / \text{Al}_{13\text{nm}}$ MLMs revealed their stability (the difference did not exceed 0.005, and was comparable to the variation of the reflection coefficient across the sample surface) over 9 months both for the sample stored under vacuum and for the one that remained in a normal atmosphere. Furthermore, this structure also had the highest reflectivity, $R = 56\%$, and a moderate passband, $\Delta\lambda = 1.6$ nm ($\lambda/\Delta\lambda \approx 20$).

Fig. 4. shows the spectral dependency of the [Be/Mg]×60/Be/Al MLM reflectance for both the sample stored in the atmosphere (symbols) and the sample kept under vacuum (solid line). The incidence angle of probing radiation was 2°. These curves were measured 9 months after MLMs sputtering.

Along with the FWHM value, it is important to note the measured reflectance at $\lambda=28.4$ nm and $\lambda=33.5$ nm was 1.2% and 2.3%, respectively.

Thus, the main result of our investigation is as follows. A new Be/Mg pair of materials has been proposed for use in the spectral region around 30 nm. The Be/Mg structure has a spectral reflection band of $\Delta\lambda=1.6$ nm ($\lambda/\Delta\lambda\approx 20$), which is sufficient for suppressing nearby "satellite" lines. The use of a 13 nm thick Al film as a capping protective layer deposited on top Be layer made it possible to obtain the particularly high reflection coefficient of $R=56\%$, while also stabilizing the structure. Observation of these mirrors for 9 months did not reveal any degradation of their reflectivity. This indicates the great potential for the use of this pair of materials for future missions to study the sun. Despite the high reflective characteristics obtained and their temporal stability, in order to finally determine the prospects for using Be/Mg MLMs in space missions, we will continue observing samples stored in air and in the vacuum for a few more years.

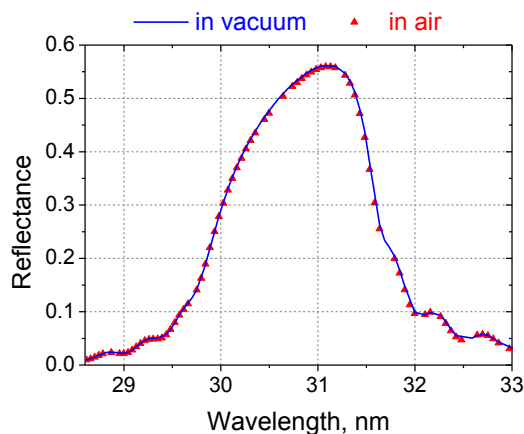


Fig. 4. Spectral dependency of [Be/Mg]×60/Be_{6.5nm}/Al_{13nm} MLM reflectance for a sample stored in the atmosphere (symbols) and a corresponding sample kept under vacuum (solid line).

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