Broad Band EUV/FUV Mirror Coatings for a Solar Spectrograph Mission

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ABSTRACT

As it is rich in spectral lines emitted by plasma between 10000 K and 20 MK, the vacuum ultraviolet (VUV - 17 to 200 nm) solar spectrum is extremely valuable for instruments that study the physics of the solar atmosphere.

We present multilayer coatings with simultaneous broadband reflectance in the two spectral ranges of 16.9 nm to 21.5 nm and 46.3 nm to 127.5 nm. The coatings are based on Mo/Si multilayers with a thin capping layer of boron carbide (B_4C).

Samples were produced and their reflectance measured. Their performance in terms of resistance to high temperatures and low micro-roughness was also assessed by measurement. Our study shows that a coating with the characteristics required by next generation spectrometers for studies of the solar atmosphere is feasible.

Keywords: multilayer coatings, vacuum-ultraviolet, solar spectrograph

1. INTRODUCTION

The solar atmosphere is characterized by structures whose temperatures go from the 10000 K of the solar chromosphere to the multi-million Kelvin of the corona and up to the tens of million Kelvin that can be reached during solar flares. Thus, the radiation emitted by the solar atmosphere ranges over all the electromagnetic spectrum. In particular the VUV (17 to 200 nm) spectrum contains emission lines formed all over the above temperature range. As the VUV range is entirely accessible by normal incidence optics, it is extremely valuable for instruments that want to study the physics of solar atmosphere at high spatial resolution. The major difficulty is that of bringing most of the above spectrum into the focal plane of a spectrometer simultaneously. For such a purpose, broadband coatings with high reflectivity need to be designed and validated.

Presently, there is no mirror coating that provides high reflectivity across the whole VUV range. Instead, at the short wavelengths end of it (also known as extreme ultraviolet – EUV), mirrors rely on multilayer coatings specially designed for particular spectral lines of the solar spectrum. Above 45 nm, on the other hand, a broad band reflectivity can be obtained with a single coating of boron carbide (B_4C)[1] or silicon carbide (SiC)[2]. A thin coating of approximately 10 nm thickness is sufficient to provide acceptable reflectance[0]. Below about 45 nm such a coating is substantially transparent, so it can be used as a reflective capping layer on top of a multilayer coating for the EUV range. The goal of our work is to develop a combined coating, to cover two wavelength regions of VUV that have been indicated as ideal for a next generation spectrometer for solar studies[4].Thus, we investigated a coating for the two wavelength bands from 16.9 nm to 21.5 nm and 46.0 nm to 127.5 nm based on a specially designed EUV multilayer coating with a boron carbide capping layer.

Samples were designed, produced, and tested at optiX fab and their reflectance measured at the Physikalisch Technische Bundesanstalt (PTB) Metrology Light Source (MLS) in Berlin[5]. Their performance in terms of resistance to high temperatures and low micro-roughness was also assessed by measurement.

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2. COATING REQUIREMENTS

Solar instruments operating in the EUV are usually protected by an entrance thin metallic filter that drastically reduces the thermal load on the primary mirror. Adding a second spectral band above 45 nm implies the removal of such a filter leaving the mirror to cope with the full solar irradiation.

In addition, the coating must provide adequate reflectance and must not compromise the surface micro-roughness of the mirror. This leads to the following requirements:

- The reflectance in the specific wavelength regions shall be comparable, and possibly higher, to those shown in Figure 2-1. To achieve a balanced solution of the reflective properties in the short wavelength (here after SW) and long wavelength (hereafter LW) ranges, different thicknesses of the B_4C coating on top of the Mo/Si broadband are explored.
- The coating shall be resistant to temperatures up 120 °C, ideally up to 200 °C and down to −20 °C.
- Micro-roughness of the surface should not be increased by more than 0.2 nm RMS. by the coating.

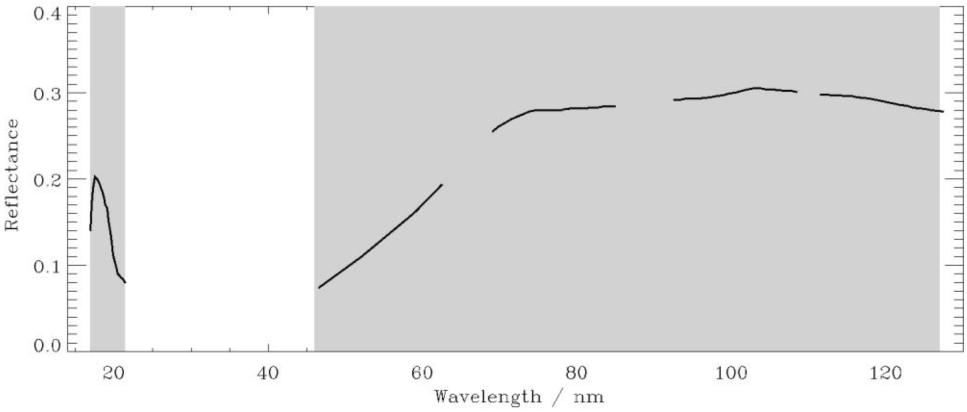

Figure 2-1: Target reflectance in the selected spectral regions (from data used by [4] and computed by combining literature values and experimental data).

3. DESIGN SOLUTIONS

To achieve a broadband response in the desired EUV range and the required overall reflectance various steps of coating design are necessary. First, an EUV broadband coating needs to be designed with respect to the requirements on thermal stability. EUV broadband coatings typically consist of a design Mo/X/Si/X, where X is material used as interdiffusion barrier, enhancing thermal stability and/or reflectance.

Studies of suitable interdiffusion barrier materials for Mo/Si coatings are abundant in the literature. As an example, carbon (C)[6], boron carbide (B_4C)[7][8] or molybdenum carbide (Mo_2C)[8] have been investigated extensively for enhancement of EUV mirrors reflectivity while being thermally stable.

Using Mo_2C does not seem to be favorable as, despite increasing the thermal resistance, it is diminishing the reflectance[9]. C barriers have been applied successfully for great thermal resistance and increased reflectivity at the same time[10]. However, if used as a barrier layer of 0.5 nm thickness at both interfaces Mo/Si and Si/Mo, it reduces the reflectivity compared to B_4C of approximately 1.5 %[8].

It is known from the literature[7] that the loss of reflectance at high temperatures is caused by widening of interdiffusion zones at the interfaces between each layer due to higher energy and mobility of atoms. Thus, as we do not expect problems below ambient temperature and down to -20° C, priority was given to testing at high temperatures.

Based on successful experience with broadband coatings realized using B_4C as interdiffusion barrier, it was chosen to start a thermal stability test with various thicknesses of B_4C, knowing that it can advantageously be used in broadband designs as interdiffusion barrier material.

With knowledge about other broadband designs done in the past and from literature values of thermal stability of Mo/B_4C/Si/B_4C coatings, a thermal design study has been prepared to determine the thickness of the B_4C that is finally used for the broadband designing process. Therefore samples with 22 periods of [Mo/B_4C/Si/B_4C] have been deposited on Si wafer test substrates. The B_4C thickness varies from 0.4 nm up to 1.4 nm, while the Mo and Si are adapted for each different B_4C thickness to keep the layer stack thickness almost constant at around 10.55 nm. With these stack layer

thicknesses we get a conventional EUV coating centered around 19.5 nm. Such a simple design has several advantages for the evaluation of the thermal tests.
Most important is that the characterization of the samples could be done in-house using X-ray reflectometry (XRR) which can detect, for such a design, changes in multilayer stack thicknesses down to 0.003 nm. Therefore, it is possible to get a fast response if the temperature has any influence on the multilayer, which gave us the opportunity to modify the test schedule or the multilayer barriers on a short-term base if necessary. Furthermore, in case of uncertainty it is still possible to make further examination at PTB, measuring at a wavelength range where the final broadband coating will also be situated.

3.1 Thickness of interdiffusion barriers and thermal stability

Thermal tests at 100 °C, 200 °C and 300 °C were performed with durations up to 100 hours. A complete overview of the test setup is given in Table **1**. These tests are performed using a vacuum annealing oven. The samples are stored inside the vacuum chamber. After achieving a vacuum pressure below 10^{-5} mbar the given thermal test is performed by heating the samples with approximately 1 K/min up to the required temperature. During the test the temperature is maintained at the given level. At the end of test time, the heater is switched off. The samples are kept under vacuum conditions until they have cooled down to 80°C or lower.

Table 1: Thermal stability tests for different B4C layer thicknesses

B_4C thickness: 0.4 nm; 0.6 nm; 0.8 nm; 1.0 nm and 1.4 nm		
Thermal test 1 h	Thermal test 10 h	Thermal test 100 h
100° C	100° C	--
200° C	200° C	200° C
300° C	300° C	300° C
100°C +200°C	100°C +200°C	100°C +200°C
100°C +200°C + 300°C	100°C +200°C + 300°C	200°C + 300°C

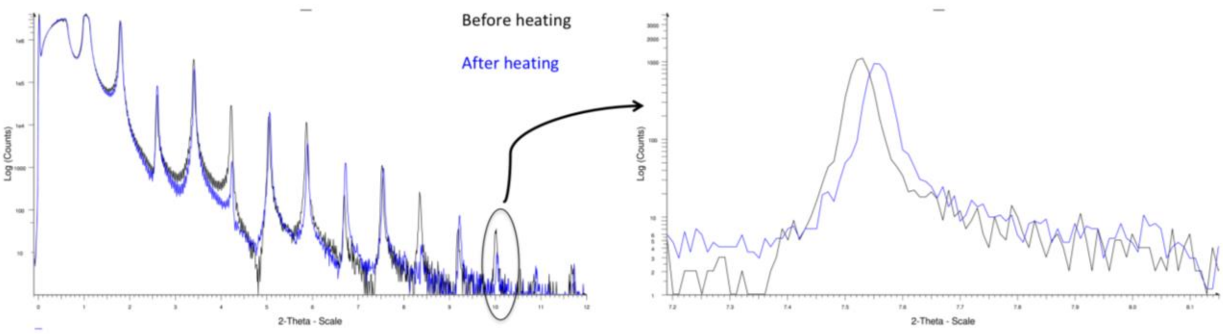

Figure 3-1: XRR measurement of a thermal test sample. The shift in the peaks enlarged on the right panel correspond to a multilayer period thickness change of 0.043 (±0,003) nm.

Determination with XRR gives a measurement curve like that shown in Figure **3-1**. A change in layer thickness can easily be detected based on a shift in peak positions when measuring the samples before and after the test.
In Figure **3-2** the dependence between B_4C layer thickness and the change in period thickness before and after a heating of the samples to the given temperatures is shown. With 1 h at 100 °C all coatings remain stable, the detected shift to a slightly higher thickness can be explained with measurement errors. At 200 °C the period thickness is, with the exception of the 0.4 nm barrier, growing slightly. The wider the barriers are, the larger the growth is. Going up to 300 °C the layer thicknesses for the barriers of 0.4 nm and 0.6 nm B4C are rapidly decreasing, while the 0.8 nm remains stable and the large ones are still slightly increasing. A similar behavior can be observed for test duration of 10 h and 100 h.
From these results it appears that the requirement of stability up to 200 °C for the broadband coating design can be met by adopting 0.8 nm thick B_4C barriers within the multilayer, as it appears being stable up to 100 h in these thermal tests.

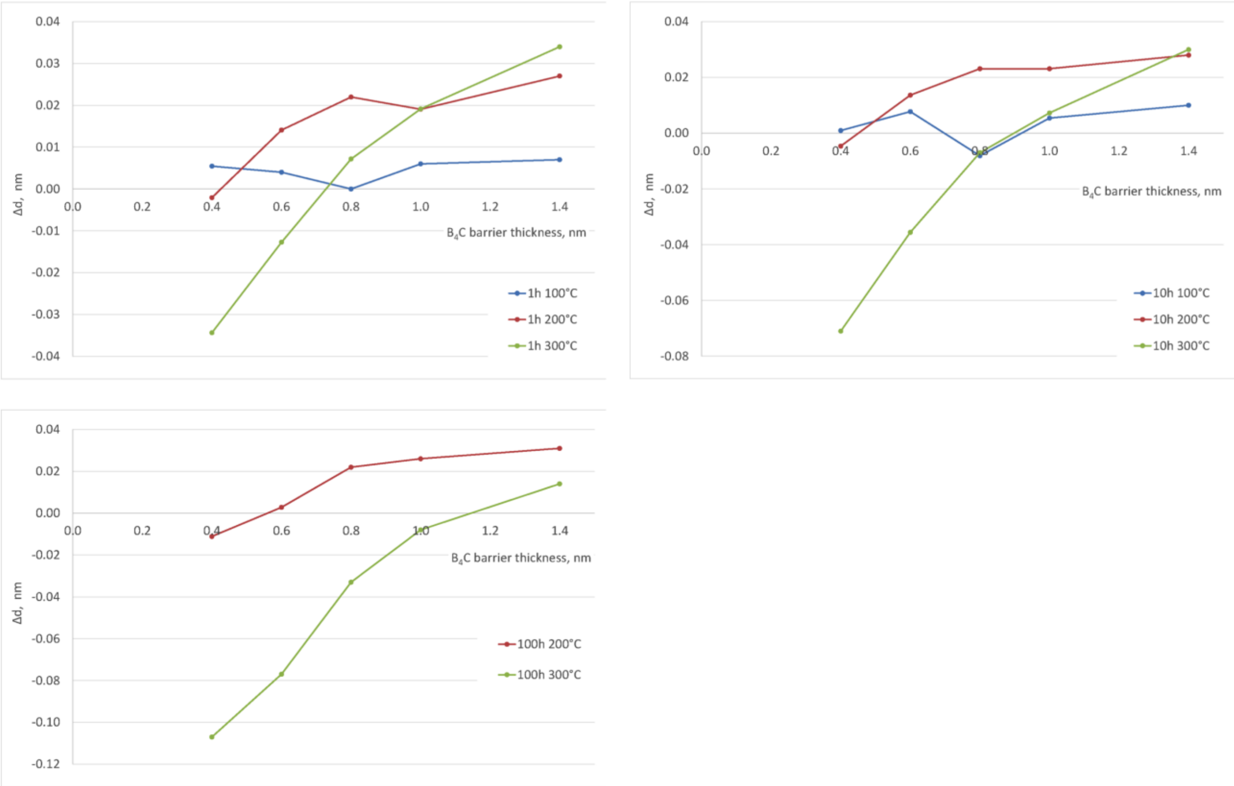

Figure 3-2: Change in period layer thickness depending on B_4C interdiffusion barrier thickness, temperature and test duration.

3.2 EUV broadband finalization

To get an initial value for the EUV broadband we started with a simple $Mo/B_4C/Si/B_4C$ multilayer with constant period thickness for the whole stack. The initial bandwidth can be easily increased, at the expense of the peak reflectance, by just reducing the number of multilayer pairs used. With such an approach it is not possible to achieve the required bandwidth and reflectance.

More sophisticated multilayer coatings consist of different stacks with various different period thicknesses or being made with completely stochastic layer thickness distribution. With this approach a substantially better result was obtained. However, there was still lack of reflectance at the long wavelength side of the EUV band.

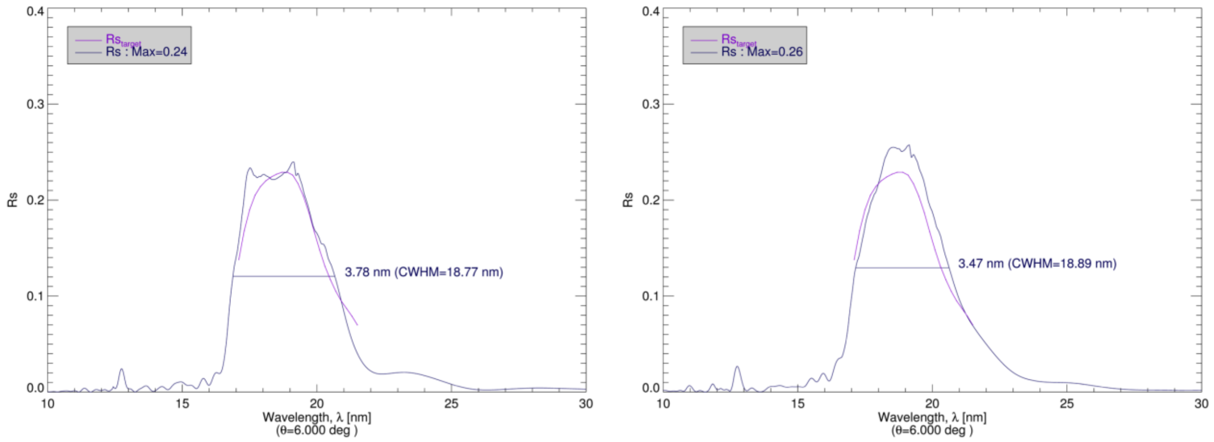

Figure 3-3: Stochastic design approaches (Design 025 / Design 026)

To overcome this issue, stochastic designs have been applied. Two examples are shown in Figure **3-3**.

Both designs shown in Figure **3-3** have been derived with the same aim, to match the given target reflectance as close as possible. The reason for the deviation between both calculated designs are different optimization algorithms that have been used. The design shown on the right side of Figure **3-3** appears to be particularly good, having an overall higher reflection with a higher emphasis for wavelengths above 18 nm, a spectral region with important but generally weaker emission lines.

In fact, the design can be tuned to maximize reflectivity in a specific spectral region (e.g., the 19 to 21 nm range where containing some relevant emission lines from highly ionized Ca and Fe), while still keeping acceptable reflectance in the rest of the spectral range down to 16.9 nm. One such solution is shown in Figure **3-4**, where the target reflectance is also shown. With this concept an absolute reflectivity enhancement of up to 3 % for the desired emission lines can be reached, while losing the same amount in the 16.9 nm to 18 nm range.

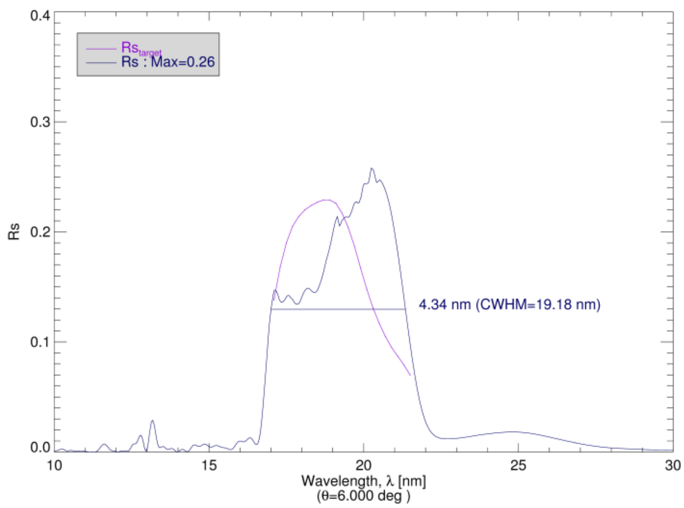

Figure 3-4: Stochastic design with higher emphasis on reflectance in the 19 to 21 nm range. The target specification is also shown.

The spectral design shown in Figure **3-4** has a steep rise of reflectivity over the spectral range. Like already stated, it is merely a question of balancing the optimization targets, so also other solutions are possible.

4. EXTENDING THE SPECTRAL RANGE TO LONGER WAVELENGTHS

To cover also the range from 45 nm to 127 nm, the stochastic Mo/B_4C/Si/B_4C coating need to be combined with a capping layer of B_4C. Seen from the design point of view, the applied B_4C thickness is a crucial issue as it changes the reflectance in the EUV bandpass. We made several attempts and iterations for optimizing the EUV broadband design for different thicknesses of B_4C. For a B_4C capped multilayer the stochastic design needs be slightly modified with respect to the one shown in Figure 3-4, that was optimized to be used without B_4C capping. A detailed finalization of the design can be done, judging against scientific requirements when necessary.

4.1 Manufacturing and measurements - reflectance

Keeping the multilayer design unchanged, samples with 7 nm, 11 nm and 15 nm B_4C have been deposited using a DC magnetron sputtering system. The optical properties for the EUV and the VUV range have been measured at PTB Berlin. The samples have been measured with s-polarized light at an angle of incidence (AOI) of 6 degrees in the EUV range and 2 degrees AOI in the VUV range, respectively. Measurements in both regions have been made on exactly the same sample.

Figure **4-1** shows the reflective properties for the target spectral ranges. Increasing the thickness of the B_4C layer, the reflectance above 45 nm range is increasing. Even for the thinnest applied B_4C coating, the reflectance is surpassing the values set as target. On the other hand, in the short wavelength region the reflectance is generally dropping with increasing B_4C layer thickness. Although, comparing the results of, e.g., 7 nm and 11 nm B_4C thickness, the reflectance decreases mainly below 18 nm, while an increase between 18.5 nm and 20.5 nm can be observed. This is due to the B_4C capping that, within a certain range of thickness, can actually be used to tune the reflectance in the EUV range.

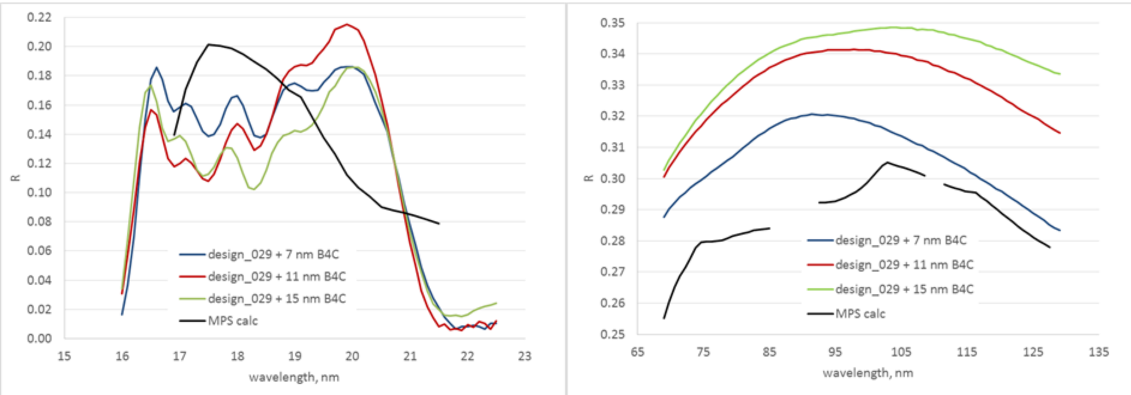

Figure 4-1: Measured reflectance in the EUV and VUV range for 7 nm, 11 nm and 15 nm of B_4C on top of multilayer structure.

4.2 Manufacturing and measurements – micro-roughness

The micro-roughness of the coating has been measured with atomic force microscopy (AFM) with 10×10 μm^2 and 1×1 μm^2 scan field size for the different B_4C capping layers. The results are shown in Figure **4-2**. The roughness in the larger area scans is below 0.2 nm RMS and it is still smaller than 0.25 nm RMS for the 1×1 μm^2 scans with higher resolution. All coatings have been deposited on Si wafer test samples. To determine the additional roughening due to the coating, an uncoated Si wafer has also been measured and found to be of 0.12 nm RMS. Thus, the overall roughness increase due to the coating is for all measurements smaller than 0.2 nm (about 0.15 nm RMS).

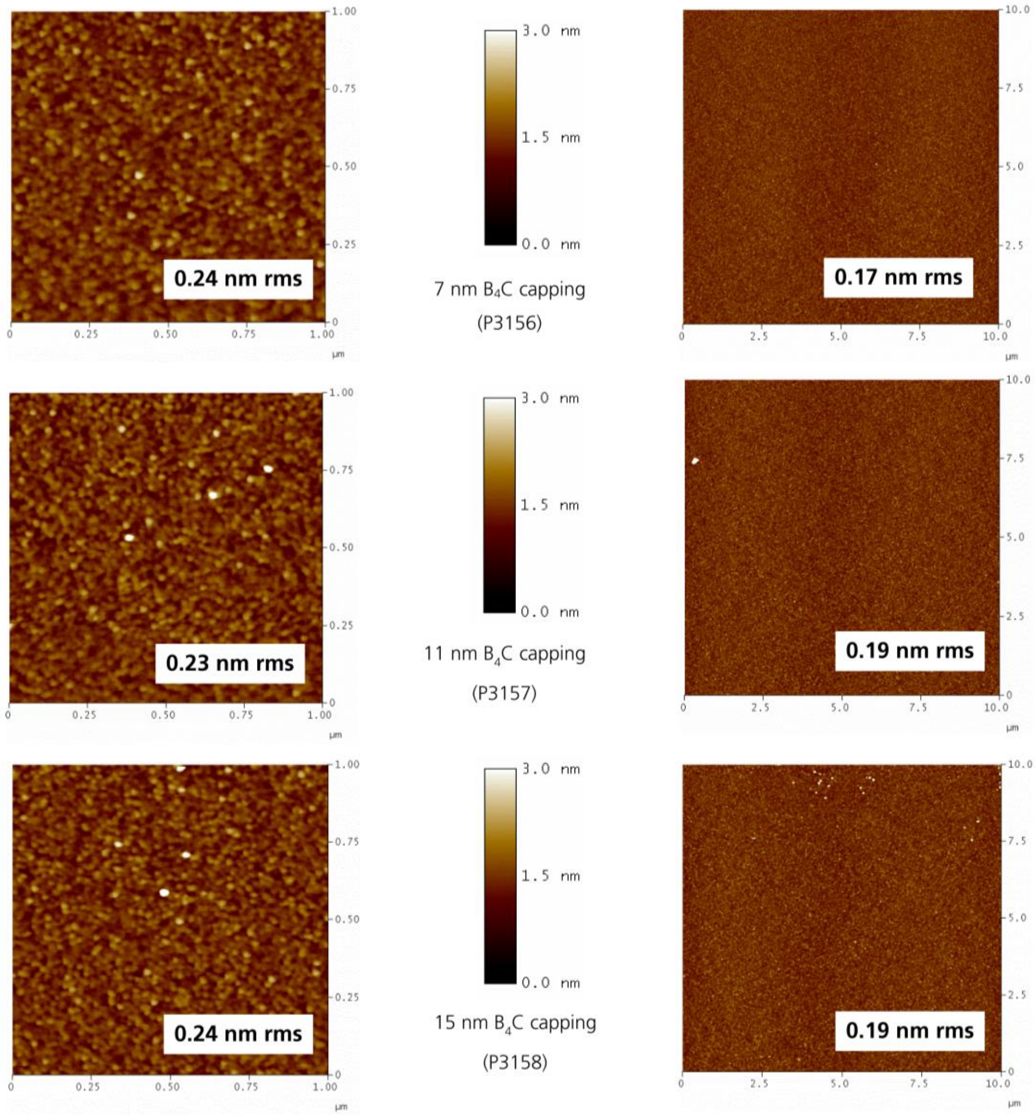

Figure 4-2: Roughness of multilayer coating with different B_4C cap layer thicknesses.

4.3 Manufacturing and measurements – thermal stability

In Section 3.1, it was already shown that the samples resist to temperatures of 200 °C for prolonged periods of time without change in reflectance. However, to prove that this is valid also for the finalized coating, a sample coated with the multilayer design and 15 nm B_4C capping has been measured at PTB before and after a thermal test (10 h at 200 °C). The results are shown in Figure **4**-**3**. In the EUV range there is no loss of reflectivity at all. The whole reflectance curve is shifted 0.1 nm towards longer wavelength. This effect is possibly due to the increase of the multilayer period thickness, which has already been shown in Figure **3**-**2**. However, most likely there are also other effects involved that come from the B_4C capping. In fact, a change of this magnitude was not noticed when the thermal test was applied to a similar multilayer without B_4C capping.

In the VUV, the reflectivity drops between 0.5 % and 1.0 % over the whole spectral range. Possible reasons for this may be a decrease in layer thickness, some kind of oxidation of the B_4C, an interdiffusion of the B_4C capping and the uppermost layer(s) of the broadband coating, or a combination thereof. To determine the root cause for this behavior further investigations will be to be necessary, particularly with respect to the fact that almost no reflectivity loss can be observed on the same B_4C coating without the multilayer underneath (i.e., directly deposited on the substrate).

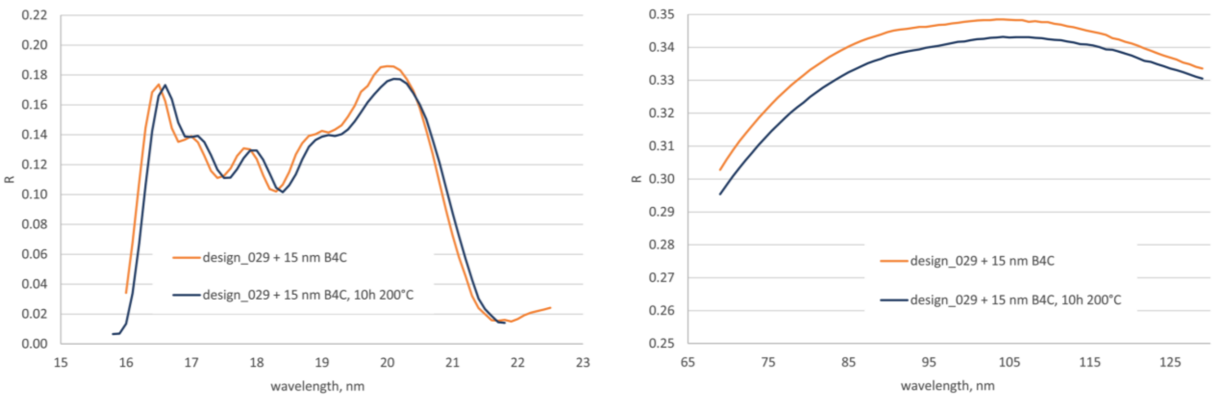

Figure 4-3: EUV and VUV reflectance before and after testing the final broadband coating for 10 h at 200°C.

5. CONCLUSIONS

We have demonstrated that a coating with the characteristics of reflectance, thermal stability, and micro-roughness required by next generation spectrographs for the study of the solar magnetized atmosphere is feasible. The design is based on a stochastic design of a Mo/B_4C/Si/B_4C based multilayer with a B_4C capping. The spectral response, particularly in the EUV, can be fine-tuned against specific scientific requirements.

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REFERENCES

[1] Blumenstock, G. M. and Keski-Kuha, R. A. M., "Ion-beam deposited boron carbide coatings for the extreme ultraviolet," Appl. Opt., 33, 5962 (1994).
[2] Larruquert, J. I. and Keski-Kuha, R. A. M., "Reflectance measurements and optical constants in the extreme ultraviolet for thin films of ion-beam-deposited SiC, Mo, Mg_2Si, and InSb and of evaporated Cr," Appl. Opt., 39, 2772 (2000).
[3] Schühle, U., Uhlig, H., Curdt, W., Feigl, T., Theissen, A. and Teriaca, L.,"Thin Silicon Carbide Coating of the Primary Mirror of VUV Imaging Instruments for Solar Orbiter," [The Second Solar Orbiter Workshop], E. Marsch, K. Tsinganos, R. Marsden, and L. Conroy, Eds., *ESA*, *SP-641*, ESA Publ. Div., Noordwijk, (2007).
[4] Teriaca, L., Andretta, V., Auchère, F. and other 39 co-authors, "LEMUR: Large European Module for solar Ultraviolet Research," Experimental Astronomy, 34, 273 (2012).
[5] Reichel, T., et al., "Developments in calibration of EUV and VUV detectors for Solar Orbiter instrumentation using synchrotron radiation," Proc SPIE, 9905, (2016).
[6] Feigl, T., Lauth, H., Yulin, S. and Kaiser, N., "Heat resistance of EUV multilayers mirrors for long-time applications," Microelectron. Eng., 57–58, 3-8 (2001).
[7] Böttger, T., Meyer, D. C., Paufler, P., Braun, S., Moss, M., Mai, H. and Beyer, E., "Thermal stability of Mo/Si multilayers with boron carbide interlayers," Thin Solid Films, 444, Issues 1–2, 165 (2003).
[8] Braun, S., Mai, H., Moss, M., Scholz, R. and Leson, A., "Mo/Si Multilayers with Different Barrier Layers for Applications as Extreme Ultraviolet Mirrors," Jpn. J. Appl. Phys., 41, 4074 (2002).
[9] Feigl, T., Yulin, S. A., Kaiser, N., Thielsch, R., "Magnetron sputtered EUV mirrors with high-thermal stability," Proc. SPIE, 3997, (2000)
[10] Yulin, S. A. et al. "High-temperature $MoSi_2$/Si and Mo/C/Si/C multilayer mirrors," [Sematec EUVL Symposium], (2004).
[11] Bruijn, S., van de Kruijs, R. W. E., Yakshin, A. E., Zoethout, E. and Bijkerk, F., "Thermally induced decomposition of B_4C barrier layers in Mo/Si multilayer structures," Surf. Coat. Technol., 205, 2469 (2010).