Capped Mo/Si multilayers with improved performance at 30.4 nm for future solar missions

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Abstract: Novel capping layer structures have been deposited on periodic Mo/Si multilayers to optimize reflectance at 30.4 nm. Design, deposition and characterization of such coatings are presented. Most of the structures proposed show improved performance with respect to standard Mo/Si multilayers and are stable over time. Reflectance at 121.6 nm and in the visible spectral range have been also tested to explore the applicability of such coatings to the Multi Element Telescope for Imaging and Spectroscopy (METIS) instrument, a coronagraph being developed for the ESA Solar Orbiter platform.

OCIS codes: (230.4170) Multilayers; (310.4165) Multilayer design; (260.7200) Ultraviolet, extreme; (310.1860) Deposition and fabrication; (350.1260) Astronomical optics; (350.6090) Space optics.

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1. Introduction

NASA and ESA have conceived several space missions dedicated to the observation of the solar atmosphere and surface dynamics. Imaging and spectroscopic observations combined with in situ measurements of particles and waves that are leaving the sun are needed to understand the solar activity and its influence on Earth. Among those missions, Solar Orbiter (SOLO), an ESA Sun-observing satellite candidate planned for launch in January 2017, will eventually operate at the closest distance to the Sun ever reached by a satellite, thereby providing observations characterized by unprecedented resolution. The Multi Element Telescope for Imaging and Spectroscopy (METIS) is a coronagraph being developed as part of the SOLO payload [1]. METIS will study the structure and dynamics of the solar corona with unprecedented temporal and spatial resolution at different heights over the ecliptic plane. METIS is an inverted-occultation coronagraph that images the solar corona in three different

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spectral ranges (visible light between 450 and 650 nm, and the two Lyman-α lines of hydrogen and helium, H I 121.6 nm and He II 30.4 nm) through of a combination of multilayer coatings and spectral bandpass filters. A spectroscopic channel will allow simultaneously acquisition of the H I and He II Lyman lines in a sector of the corona in order to determine the line of sight velocity distributions of protons and helium ions between 1.2 and 2 solar radii. These measurements will be fundamental for understanding solar wind particle acceleration processes. The current optical design of the METIS telescope is based on an on-axis Gregorian telescope with external inverted occulter. Since common optical coatings working near normal incidence provide negligible reflection efficiency in the Extreme Ultraviolet (EUV) spectral range, the telescope mirrors will use multilayer (ML) coatings, a technology that will also be adopted in another instrument on board of SOLO, the Extreme Ultraviolet Imager (EUI) [2]. The same coating technology will be also used in the METIS spectroscopic channel to coat the grating optical component. Since MLs operate by interference effect, these coatings are typically optimized to perform at one specific wavelength; however, in the specific case of METIS, since the different spectral channels share a common optical path in telescope, it is necessary that the ML coatings perform well in all three spectral regions of interest, without any degradation over the duration of the mission. Periodic multilayer coatings using Mo/Si bilayers have been used in previous space missions [3–5], and their long term stability have been therefore proven. However, at 30.4 nm they show an experimental peak reflectance of only ~20% near normal incidence, and so their use would limit the METIS telescope effective area, which scales quadratically with peak reflectance due to the double mirrors configuration [6]. The performances could be improved only relatively by controlling diffusion at interfaces [7] or improving the optical contrasts between Mo and Si [8]. Improved multilayer performance at this wavelength can be realized using tri-layer structures such as B4C/Mo/Si [9]; however, it is not clear if these films are stable over time, in particular considering experimental results obtained on multilayer based on Si/B_4C bilayers [10–12]; further investigations are needed to determine the long-term stability of these coatings. New multilayer structures based on other material couples, which in principle can offer higher efficiency, are also under investigation. The Mg/SiC system can achieve up to 40% reflectance at 30.4 nm, and its stability has been widely investigated [13,14]. This structure was also deposited on the mirrors used in the NASA SCORE sounding rocket, lunched in 2010. That instrument was conceived as a METIS prototype in order to test the performance of the optical configuration. A variety of prototype of Mg/SiC MLs coatings was fabricated, in which the thickness ratio between the two materials, and/or the capping layer material and thickness, were systematically varied. It was found that optimized coatings can offer superior performance [14], however some of the SCORE prototype films were found to be unstable. Co/Mg MLs for this wavelength range have also been recently reported [13], but in light of the stability problems associated with SiC/Mg MLs, the stability of these new films must be thoroughly investigated as well before they can be used in a satellite instrument such as METIS. Other new structures have been recently proposed as well. For example, Ir/Si MLs have been shown to reflect up to 25% near 30 nm, with good stability over six months; however their long-term stability must also be investigated prior to satellite use [15]. Al/Mo/SiC multilayers have been developed for the SOLO-EUI instrument, but their long term stability must be demonstrated as well [16]. As an alternative to developing all-new multilayer structures, we propose instead to improve the performance of standard Mo/Si structures near 30 nm by the use of capping layers that are designed to increase the peak reflectance at 30.4 nm [17]. While the stability of any new multilayer structures must be experimentally demonstrated, we can increase the likelihood of success by building on the stable, well-understood Mo/Si structure. In this paper we report on the design and characterization of such multilayer structures, showing experimental reflectance obtained in all three spectral ranges. We have also begun to investigate the temporal stability of these coatings, and we report here on the interim results obtained after six months duration.

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2. Multilayer design and fabrication

The selection of capping layer material candidates was based on simulations performed with the goal of improving peak reflectance at 30.4 nm, while simultaneously optimizing the reflectance at 121.6 nm and in the visible range. We focused principally on non-toxic and stable metals for the top-most layer of the cap, with iridium (Ir), ruthenium (Ru) and tungsten (W) emerging as the best choices. The final optimization of the capping layer design uses an analysis of the standing wave distribution throughout the film stack in order to maximize the peak reflectance at the target wavelength [18]; this technique was developed for EUV lithographic applications [19, 20]. The underneath Mo/Si periodic structure, optimized for peak reflectance at 30.4 nm with a 5° incidence angle, was designed using IMD software [21] and its parameters are reported in Table 1.

Table 1. Structure Of The Periodic Mo/Si ML Tuned To 30.4nm. d Is The Multilayer Period, Γ Is The Layer Thickness Ratio, And N Is The Number Of Periods

Mo/Si multilayer tuned at 30.4nm	
d	16.40 nm
Γ	0.82
Si	13.45 nm
Mo	2.95 nm
N	35

Optimized capping layer structure parameters are reported in Table 2. Ir is an attractive candidate due to its resilience to oxidation and its widely use in space applications. For this investigation, Ir top layers were coupled with either Mo (CL1) or Si (CL2) underlayers. These two alternatives have been considered in order to explore possible different behaviors at the Ir interfaces. Ru is also an attractive candidate material due to its demonstrated stability in harsh environments in EUV lithography applications, even though Ru can form an oxide. The Ru capping layer design (CL3), is coupled with a Mo layer is used in order to avoid inter-diffusions with the Si top layer of the underlying Mo/Si coating [22]. The fourth capping layer structure uses a single W layer. Periodic multilayers containing W are widely used for X-ray applications [23]. Intermixing between W and Si is expected, similar to the intermixing found in Mo/Si multilayers, with formation of tungsten silicides; W is also expected to oxidize upon exposure to air.

The optical performance of the capped multilayers are compared with the reference standard structure reported in Table 1, The reference standard is a periodic Mo/Si ML with a 2-nm-thick Si layer cap; this top Si layer is expected to oxidize after exposure to air, forming a SiO2 layer approximately 1 nm in thickness.

The calculated peak reflectance values are also shown in Table 2, computed assuming perfectly smooth, sharp interfaces. For these calculations we have used the optical constants provided by the Center for X-ray Optics for amorphous Si, Ir, Ru and W and those from Tarrio et al [24, 25] for Mo. These simulations show that all the capped structures have higher reflectance relative to the standard Mo/Si reference structure.

Prototype multilayer structures were deposited at Reflective X-ray Optics LLC (New York, USA) by DC magnetron sputtering onto polished Si(100) substrates measuring 16 mm x 16 mm, using a system that has been described previously [26].

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Table 2. The Capping-Layers Structure And Their Theoretical Reflectance Peak At 30.4nm

ML	Capping layer	R @30.4nm
REF	SiO_2 (1.0 nm) Si (1.0 nm)	0.24
CL1	Ir (2.0 nm) Mo (2.2 nm)	0.29
CL2	Ir (2.0 nm) Si (15.4 nm) Mo (2.95 nm)	0.27
CL 3	Ru (2.0 nm) Mo (2.0 nm) Si (14.0 nm) Mo (3.0 nm)	0.29
CL 4	W (2.0 nm)	0.25

3. Reflectance test

3.1 EUV reflectance measuremen

The EUV reflectance of CL1, CL2 and CL4 along with the reference Mo/Si multilayer (REF) were measured approximately two weeks after deposition at BEAR beamline at ELETTRA Synchrotron (Trieste, Italy) [27]. Reflectance measurements were made over the 26 – 34 nm spectral range with a 5° beam incidence angle. Due to scheduling difficulties, multilayer CL3 was measured at beamline 6.3.2 at ALS (Berkeley, California USA) three weeks later using the same measurement parameters as used at BEAR. In order to compare the measurements performed at the two beamlines, CL1 was also re-measured at ALS and we find excellent agreement with the BEAR results. In Fig. 1 we show the experimental reflectance data for CL1, CL2, CL3 and REF, along with fits to these curves. Reflectance data for CL4 are shown in Fig. 3.

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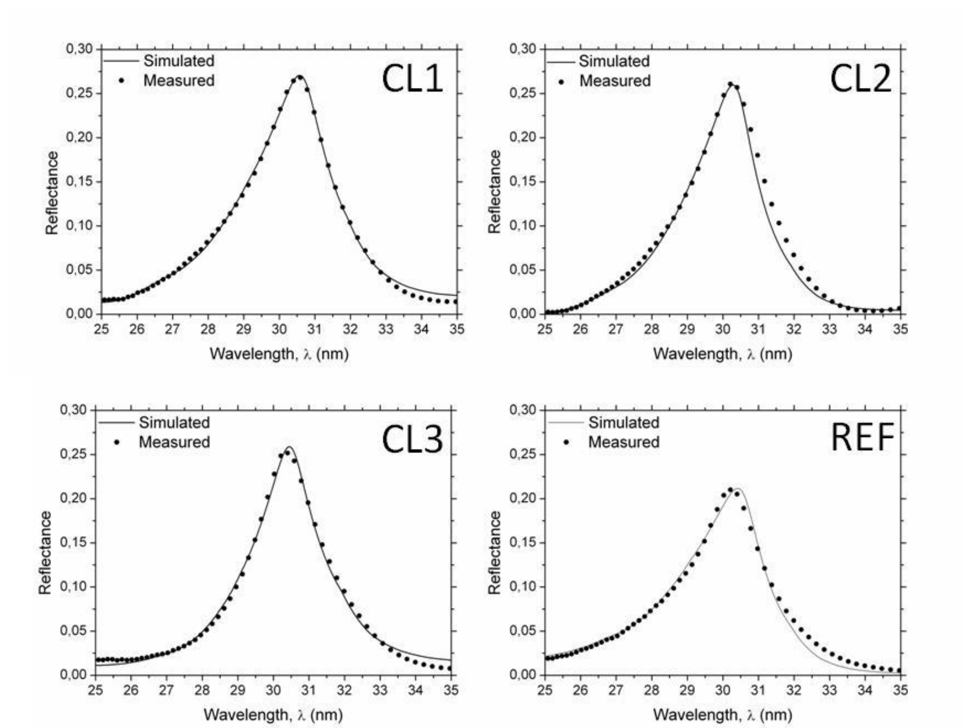

Fig. 1. Reflectance measurements performed at BEAR beamline and at ALS 6.3.2 beamline of sample CL1, CL2, CL3 and REF.

The fits shown in Fig. 1 were computed using IMD software, assuming a polarization factor of 0.9 of the beamline experimental beam. Fit parameters (i.e. layer thicknesses and interfacial roughness values are reported in Table 3, along with the measured peak reflectance values. The samples are found to have a period that in some cases is slightly different from the theoretical values reported in Table 1, fact that explains the small shift in the peak wavelengths in Fig. 1; on the contrary, such small period variation does not affect peak reflectance value. The capping layer thicknesses were found to be within a few angstroms of the target values in all cases. From the fitting, interface roughness value have been estimated.

The reflectance measurements were performed again six months after deposition; all samples have been measured at BEAR. Samples CL1, CL3 and CL3 were found to be stable, with no measurable change in reflectance. From the results shown in Fig. 1 we find that all three capped multilayers (i.e., CL1, CL2 and CL3) perform better than the standard Mo/Si ML in term of peak reflectance; the spectral bandpass was found to be ~1.8 nm FWHM for all samples. Both Ir capped samples have a peak reflectance of 0.26, showing a 20% relative reflectance with respect to the reference sample. While a Mo layer was used with CL1 in order to avoid the creation of an Ir-Si interface that might have been found to be unstable, we see from the reflectance results for CL2, which does have an Ir-Si interface, that this coating is so far as stable as CL1. In Fig. 2 we show an HR-TEM image of a 2.3 nm top Ir layer deposited onto a 13.45 nm Si layer over an Ir/Si ML structure obtained after 6 month from deposition is shown; in the image a top 2.3 nm Ir layer and an underneath Si layer on top of which there is a formation of a mixed composition Si-Ir interlayer of about 2.5 nm can be identified. In Fig. 3 the experimental data related to CL4 are presented. In this case the sample results unstable over time. This multilayer has therefore considered not suitable for space applications. The reason of such instability has been investigated and described later on. Here we anticipate that the initial fitting was obtained assuming that, due a large intermixing at the W/Si interface, a WSi_2 compound is formed, as reported in Table 3.

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Tab.3 Experimental data fitting parameters and peak reflectances.

Coating	Fit parameters	Peak R	Coating	Fit parameters	Peak R
REF	d=16.52 nm Γ=0.81 Roughness; 0.75nm Top oxide SiO_2: 1 nm	0.20	CL3	d=16.43 nm Γ=0.83 Ru: 2.0 nm Mo: 2.5 nm Si: 14.3 nm Mo: 2.97 nm Roughness: 0.7 nm Top oxide RuO_2: 1 nm	0.25
CL1	d=16.58 nm Γ=0.80 Ir: 2.2nm Mo: 2.3nm Roughness: 0.65nm	0.26	CL4	d=16.52 nm Γ=0.76 W: 1.28 nm WSi_2: 0.52 nm Roughness: 0.7nm Top oxide WO_3: 1 nm	0.21
CL2	d=16.40 nm Γ=0.81 Ir: 2.0 nm Si: 15.4 nm Mo: 2.95 nm Roughness: 0.65 nm Roughness Ir/Si: 1 nm	0.26			

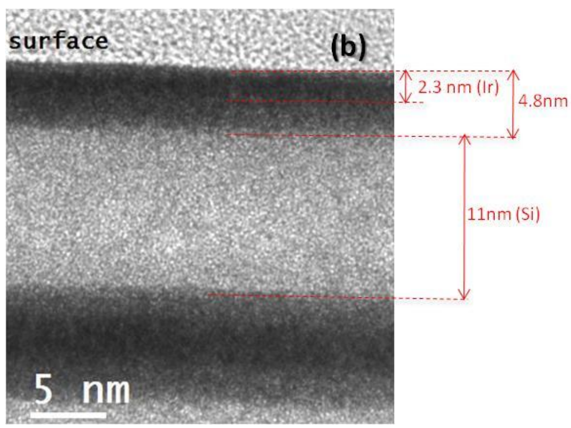

Fig. 2. HR-TEM image of top layers in a Ir/Si ML; the top layer structure is equivalent to CL2.

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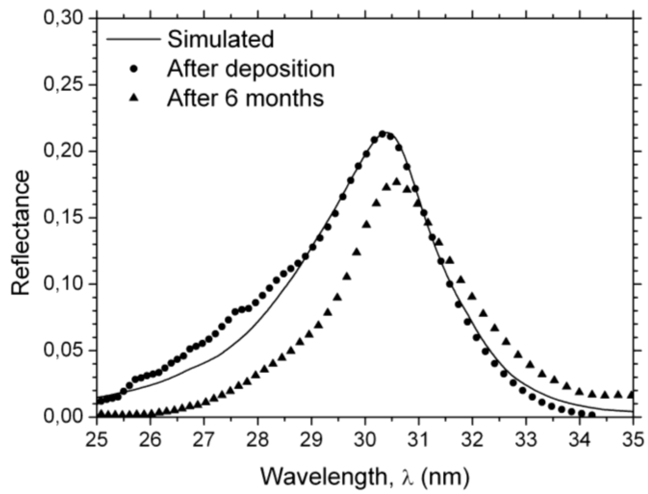

Fig. 3. Reflectance measurements of CL4 a few weeks after deposition and after six months performed at BEAR beamline.

3.2 Ly-alpha reflectance measurements

The reflectance at 121.6 nm has been measured using a normal incidence reflectometer at CNR – IFN LUXOR (Padova, Italy) three weeks after deposition. During this time, samples were kept in plastic boxes stored in regular atmosphere. A Hamamatsu deuterium lamp coupled with an EUV-FUV monochromator mounted in Johnson–Onaka configuration was used to generate monochromatic radiation. Preliminary spectral calibration of the monochromator was performed using emission lines generated by an hollow cathode lamp filled with different gases, as well as a Hg lamp. A toroidal mirror focuses the beam exiting from monochromator in the test chamber equipped by a θ – 2θ plane system. The measurements were made at 10° of incidence in two different orientations of the test chamber rotated 90° to each other; results were then averaged to obtain the reflectance for un-polarized light. The incident and reflected beam intensities were measured by a channel electron multiplier (CEM) detector in photon counting mode, and the reflectance curves as function of the incidence angle at 121.6 nm wavelength were computed from the ratio of the two signals. The results are reported in Fig. 4 along with the theoretical values calculated assuming an un-oxidized capping layer.

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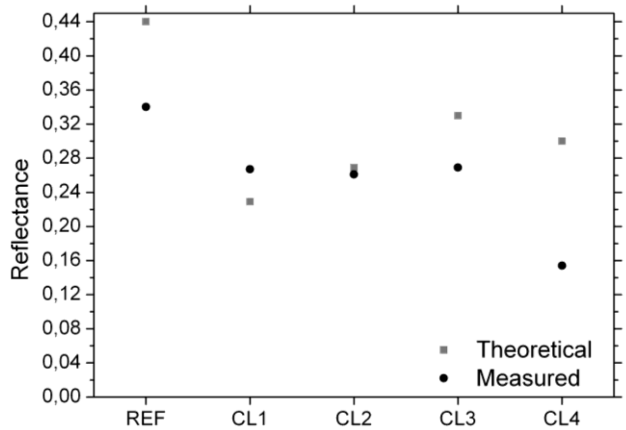

Fig. 4. Reflectance measurements at 121.6 nm performed after deposition at 10° incidence angle; the data are compared with the simulations performed with IMD program.

The reflectance at 121.6 nm depends on the top most layers of the ML. In particular, in Fig. 5 the standing wave distribution inside the structure CL1 at this wavelength shows that the reflectance mainly depends on the capping layer and the first Si layer. The experimental results are lower than the theoretical values for all samples, except for CL1; we hypothesize that this unexpected result may be due to inaccurate optical constants for Mo. On the other hand, the experimental value for CL2 is well in agreement with simulations since both Ir and Si optical constants are well known and Ir does not oxidize. The reflectance measured for the reference sample is consistent with a Si oxide thickness of 0.7 nm, assuming the SiO_2 optical constants in IMD database. The CL3 discrepancy between theoretical and experimental reflectance values can be attributed to ruthenium oxidation; in this case simulation cannot be performed as Ru-oxide optical constants are unavailable. In the case of CL4, a large discrepancy has been observed between experimental data and theoretical simulation; in this case tungsten oxidation can play an important role.

All sample were re-measured after six months; again, all sample results to preserve the reflectance, except CL4, which value drops to 0.04.

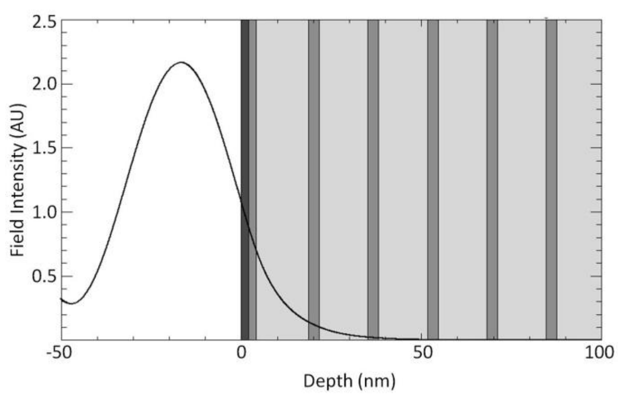

Fig. 5. Standing wave intensity distribution in CL1 at 121.6 nm.

3.3 Visible reflectance measurements

Reflectance at visible wavelengths was measured by a commercial Varian UV-Vis-NIR spectrophotometer (model Cary 5000) at normal incidence angle. Results are reported in Table 4. The diffuse reflectance was found to be negligible and, in this spectral range,

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oxidation does not strongly affect the reflectance of the materials in question. Samples were re-measured after six months and no appreciable variation was found.

Table 4. Reflectances In The Visible Spectral Range

Sample	@ 500 nm	@ 550 nm	@ 600 nm	@ 650 nm
REF	0.45	0.43	0.41	0.40
CL1	0.58	0.56	0.55	0.54
CL2	0.52	0.51	0.49	0.48
CL3	0.59	0.58	0.57	0.56
CL4	0.48	0.45	0.44	0.43

The experimental reflectance in the three spectral ranges of interest for METIS, as discussed in previous sections, are all summarized in Table 5 for readers convenience.

Table 5. Summary Of Reflectance Measurement Data

Sample	30.4 nm		121.6 nm		500 nm		600 nm	
	after two weeks	after six months	after 3 weeks	after six months	after 3 weeks	after six months	after 3 weeks	after six months
REF	0.20	0.20	0.34	0.33	0.45	0.45	0.41	0.41
CL1	0.26	0.26	0.27	0.27	0.58	0.58	0.55	0.55
CL2	0.26	0.26	0.26	0.26	0.52	0.52	0.49	0.49
CL3	0.25	0.25	0.27	0.26	0.59	0.59	0.57	0.57
CL4	0.21	0.18	0.15	0.04	0.48	0.48	0.44	0.44

4. AFM analysis

Atomic Force Microscopy (AFM, Park System XE – 70) analysis has been performed operating in non – contact mode on all samples. Sample REF, CL1, CL2 and CL3 appear as a smooth, continuous films with no islanding and ~0.3 nm rms roughness. The analysis was repeated six months after deposition and no changes were found for these samples. However, CL4 has a different topography, with larger roughness after six months (Fig. 6). The AFM images indicates a re-organization of the film compatible with what already observed to be the formation of W crystallite; intermixing of W and Si in the top surface of a depth graded ML was also observed with TEM [23]: while the inner bilayers (minimum period 3.33 nm) appear as an amorphous film of Si and W somehow with well define interfaces, the top W/Si bilayer, characterized by a period of around 26 nm, appeared as fully inter-diffused, indicating the possible formation of a W_xSi_y compound. The same intermixing process was also observed in 4 nm period W/Si multilayer [28]. Crystallization of the top layer as well as intermixing between top layers materials could explain the degradation of the optical performances. Nevertheless, further investigations are required for fully understanding the physical process that affects the W surface.

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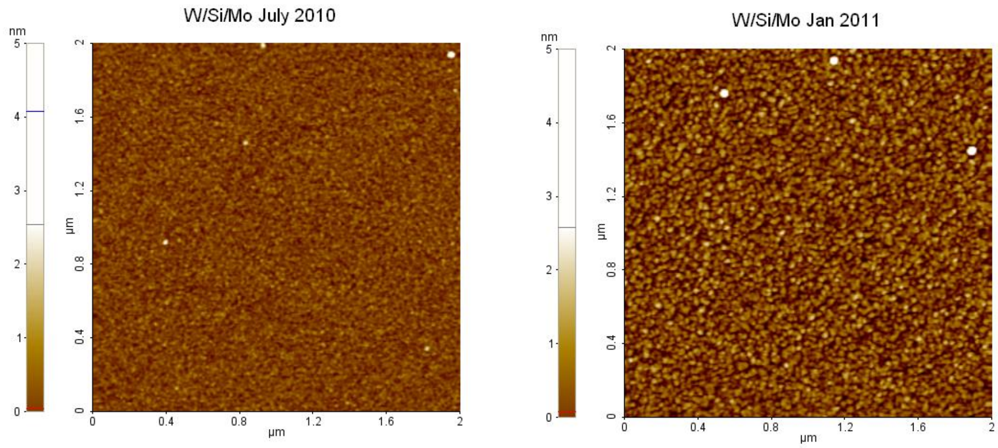

Fig. 6. AFM analysis of CL4 after deposition and six months later

5. Conclusion

A variety of capped Mo/Si multilayers have been designed for improved performance at 30.4 nm wavelength.. The choice of considering MLs based on Mo/Si bilayers is related to the proven high reliability of these films on the long term. Different capping layers are used to enhance reflectance at 30.4 nm. Multilayer based on other material couples could in principle provide higher reflectance at 30.4 nm, but their stability validation is still required for space instruments application. Reflectance measurements of prototype films were made at 30.4 nm (He II) and121.6 nm (H Ly alpha) wavelengths, and in the visible spectral range as well, in order to qualify these coatings as possible candidate for the METIS coronagraph on board of SOLar Orbiter. Reflectance measurements made over six months show excellent stability for 3 of the 4 prototypes investigated; temporal stability measurements over longer time scales are still needed, however. We find that Ru- and Ir-capped MLs show higher reflectance at 30.4 nm with respect to a standard periodic Mo/Si, while the W-capped ML is unstable over time. The reflectance values measured at 121.6 nm are lower in the capped MLs case than in the Mo/Si standard reference, but the performance sum still represents a good compromise considering that effective area at 30.4 nm is the main concern; in fact, due to the abundance of hydrogen in the solar corona, the HI Lyman-α line (121.6nm) is much brighter than the HeII Lyman-α line (30.4nm). On the other hand, we find an improvement of reflectance in the visible range. In particular, while from a structural point of view the use of a Mo interlayer in the Ir capped structures does not seem to have any impact in the performance at 30.4 nm and at 121.6 nm, its use allows to improve the performance in the visible spectral range; further analysis is required to verify possible effects of this inter-layer on long term stability. We propose these new coatings as useful for space application, for which superior performance in the EUV spectral range are required along with long-term temporal stability.

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