Stability of extreme ultraviolet multilayer coatings to low energy proton bombardment

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Abstract: In this work we present results of an new experiment related to low energy protons bombardments on nano-structured optical coatings. Multilayer structures protected by different capping layers have been fabricated and exposed to low energy protons (1keV). The experimental parameters have been selected considering the potential application of the coatings to solar mission instrumentation. Future solar missions will investigate the Sun from very close distances and optical components are constantly exposed to low energy ion particles irradiation. The experiment was repeated fixing the proton flux while varying the total dose accumulated. Results show that physical processes occurred at the uppermost interfaces can strongly damage the structure.

OCIS codes: (230.4170) Multilayer; (260.7200) Ultraviolet, extreme; (350.4990) Particles; (350.1820) Damage; (350.6090) Space optics.

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1. Introduction

Solar Orbiter (SOLO) is a Sun-observing satellite under development by European Space Agency (ESA) whose launch is eventually foreseen in January 2017. It will operate at the closest distance to the Sun ever reached, providing observations characterized by unprecedented resolution. Reaching a minimum perihelion distance of only 0.28 AU, the spacecraft will be immersed in a very harsh environment, which effect on spacecraft and on-board instrumentations must be critically evaluated. One of the main concerns regards possible degradation of optical components induced by the constant bombardment of solar wind ion particles. In this work we present the first experiment carried on in laboratory to establish the effect of solar wind low energy proton bombardment in nanostructured optical coatings. Even though the coatings used in the experiment are specifically optimized for the Multi Element Telescope for Imaging and Spectroscopy (METIS) [1, 2], the experimental approach and results can be of interest not only for other instruments which adopt the same type of coatings, as the Extreme Ultraviolet Imager (EUI) [3], but also for any type of coatings, filters, or optical components experiencing similar environment.

The quiet solar wind plasma is mainly composed of protons and other ion species such as α particles (He2 +), O6 + and Fe10 + , with velocities typically included in the range 300-1200km/s, and with an average value around 468 km/s [4]. More severe occasional disturbances in the solar wind are represented by coronal mass ejections or solar flares, in which high energetic particles are thrown in the heliosphere; even though energies of such particles are higher (in the MeV range) than those of the quiet solar wind (in the keV range), the effect on the instrument components is considered less severe [5].

Periodic Mo/Si multilayer working in quasi normal incidence (i.e., 5°) and tuned at HeII Lyman-α line were designed using IMD software: the multilayer stack has 35 bi-layers with period of 16.84 nm and a thickness layer ratio Γ of 0.82. The structure includes also a thin Si capping-layer (which is expected to oxidize upon exposure to air). The same multilayer structure has been designed also with the addition of three different capping-layers, the firsts based on an Ir/Mo and Ir/Si couples and the second on a Ru/Mo one. The capping layer structures were optimized through an analysis of the standing wave distribution throughout the film stack, to maximize the peak reflectance at the target wavelength [6, 7]. These coatings offer superior performance with respect to standard Mo/Si multilayers; full analysis of these new structures is presented elsewhere [8], and the results are compared also with the performance of other recently developed structures [9–11]. In this context the capping layer structure investigation is interesting to verify also the protection capabilities of the underlying Mo/Si structure with respect to the protons bombardment. Ion implantation experiment has been performed using the Low Energy Implanter (LEI) facility. Samples have been characterized prior and after exposure; a non-exposed reference sample for each type of the structure was also monitored to avoid presence of misleading results. Experimental details, results and discussion are presented in the present work.

2. Materials and methods

The multilayer used in this experiment are based on a standard Mo/Si periodic multilayer (parameters in Tab.1) on top of which different capping layer materials structures have been deposited.

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Tab.1 Mo/Si multilayer parameter

Mo/Si multilayer tuned at 30.4nm	
d	16.40 nm
Γ	0.82
Si	13.45 nm
Mo	2.95 nm
N	30

Tab.2 Capping layers structure parameters

ML	Capping layer
REF	SiO2 (1.0 nm) Si (1.0 nm)
CL1	Ir (2.0 nm) Mo (2.2 nm)
CL2	Ir (2.0 nm) Si (13.5 nm)
CL 3	Ru (2.0 nm) Mo (2.0 nm) Si (14.0 nm) Mo (3.0 nm)

Multilayers were deposited onto a Si (100) substrate by magnetron sputtering, using a system that has been described previously [12]. Reflectivity measurements at 5° incidence angle in the 25–37 nm spectral range were performed after deposition at the Bending Magnet for Emission Absorption and Reflectivity (BEAR) beamline at ELETTRA Synchrotron (Trieste, Italy), using a 0.9 polarized beam. The ion implantation experiment has been performed at Forschungszentrum Dresden-Rossendorf (Germany, ex Helmholtz-Zentrum Dresden-Rossendorf) using the Low Energy Implanter (LEI) facility. Experimental ion implantation parameters of the experiment have been recovered by the information available in literature related the composition and density of the quiet solar wind species, which have been experimentally measured in situ by different instruments at 1 AU [4]; such parameters are reported in Tab.3. To derive the same parameters at the innermost part of the heliosphere at a generic distance r from the Sun, such experimental values at 1AU are multiplied by the inverse square of r, assuming therefore a model based on a radial law. In Tab.4 the minimum, maximum and average orbits distance from the Sun reached by SOLO are reported together with the correspondent scaling factors, according to the ESA document [4].

Tab.3: Solar Wind parameters

	At 1AU (Earth)	Mission average
Density (ions/cm-3)	8,7	25
Speed (km/s)	468	468
Nalpha/Nproton	0,047	0,047

From the proton plasma density at 1 AU (8.7 ions/cm3), the density at mission average distance can be therefore calculated (Tab.3).

Tab. 4: Scaling factor for SOLO mission

	Radius [AU]	Scaling factor [r-2]
Min perihelion	0.23	12.70
Max aphelion	1.48	0.45
Mission average	0.59	2.87

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Given the assumption on proton plasma density (25 ions/cm^3) and solar wind velocity (468 km/s), it is possible to determine the flux at the SOLO average distance as 1.17x10^9 proton/cm^2 per second. From this data, the number of protons impinging a 1 cm^2 sample can be derived as 3.03x10^{15} in a month, 9.1x10^{15} in three months, 36.4x10^{15} in a year, and 36.4x10^{16} total of the mission (ten years). Two low energy proton implantation sessions have been carried out in order to expose the samples with a three months equivalent dose and one year equivalent dose. The experimental details of each implanting session are summarized in Tab.5. Proton fluxes used during experiments were computed in order to obtain the desired protons total dose in a reasonable period of time (32 hours for each session in our experiment).

Table 5. Ion implantation experiment details.

	Session A (3 months dose)	Session B (one year dose)
Duration	32 h	32 h
Protons energy	1 keV	1 keV
Protons flux	$0.79 \cdot 10^{11} cm^2 sec^{-1}$	$3.1 \cdot 10^{11} cm^{-2} sec^{-1}$
Total dose	9.1x10^{15} protons	36.4x10^{15} protons

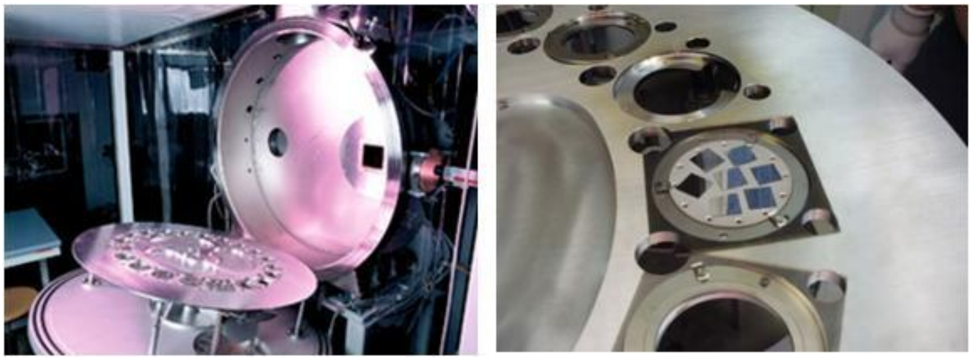

Fig. 1. The LEI facility at Forschungszentrum Dresden-Rossendorf and the samples accommodated in the support of LEI.

During the implantation sessions, the vacuum pressure was lower than 10^{-8} mbar. The samples were accommodated in a conductive support placed perpendicularly to the proton direction for ensuring a normal incidence implantation (Fig. 1). The proton current was measured and integrated over time in order to control the total dose. After implantation, the samples were measured again at BEAR beamline.

3. Results and discussion

The reflectances of the structures before and after implantation are reported in Fig. 2. A non-irradiated witness sample has also been re-measured to verify possible degradation due to natural aging of the structures.

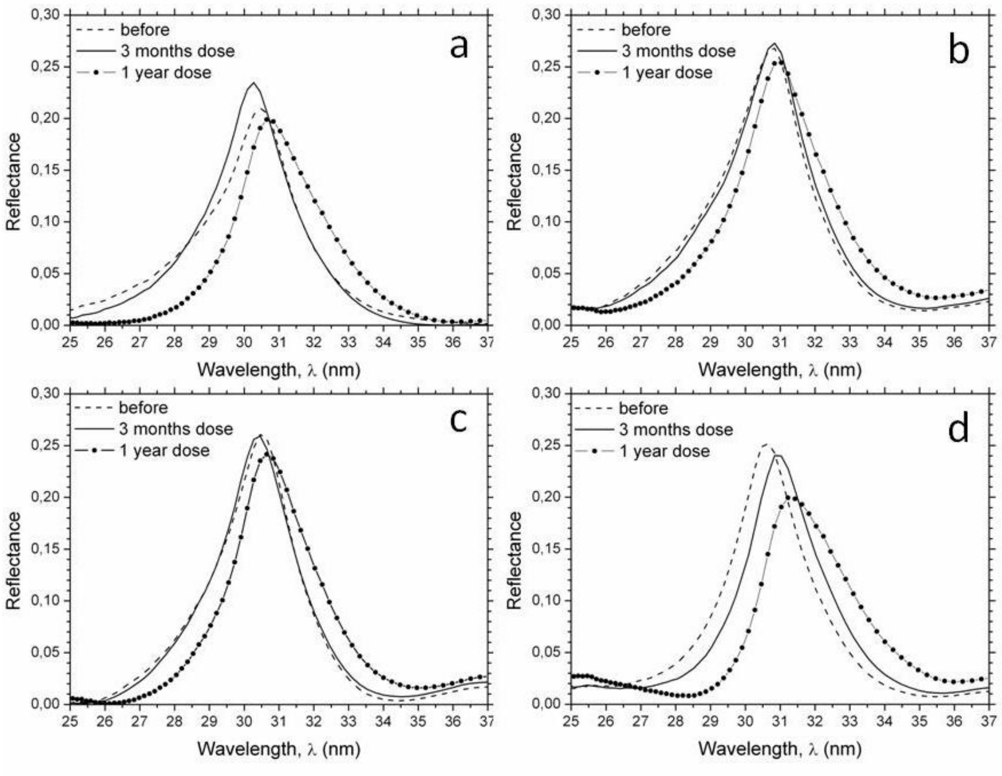

Fig. 2. Reflectance measurements of the structures REF (a), CL1 (b), CL2 (c) and CL3 (d) before and after low energy protons implantation experiment.

Tab.6 Peak reflectance of the multilayer structure before and proton implantation experiment.

ML	Before implantation	3 months dose	1 year dose
REF	0.21 @ 30.2nm	0.23 @ 30.0nm	0.20 @ 30.4nm
CL1	0.26 @ 30.2nm	0.26 @ 30.2nm	0.24 @ 30.4nm
CL2	0.27 @ 30.6nm	0.27 @ 30.6nm	0.25 @ 30.8nm
CL3	0.25 @ 30.4nm	0.24 @ 30.6nm	0.20 @ 31.0nm

The reflectance peaks prior proton bombardment of the structures overcoated by a capping-layer are higher than standard Si/Mo, being such values ~0.27 for the Ir/Mo, ~0.25 for the Ru/Mo and only ~0.21 for the Si/Mo (Tab.6). The same spectral bandpass of 2.9 nm FWHM has been found in all samples. All coatings have been verified being stable after six months. After proton the bombardment, measurements have been repeated, mapping the surface in different places. After implantation session B, the Ir/* capped multilayers dropped slightly. In contrast, the Ru/Mo capped film showed a reflectance loss after session A, with even more dramatic losses in reflectance after session B. While after session B also Mo/Si standard multilayer show a degradation, they shows an initial increase of reflectance after session A. This behavior has been verified on two identical Si/Mo samples, which were deposited by sputtering in two different deposition runs; further investigation had been necessary to understand such physical phenomena. Therefore X-ray Photoemission Spectroscopy (XPS) has been carried out for all samples at ELETTRA-BEAR beamline. XPS survey photons (energy 800eV, pass energy 20eV, binding energy step 0.152eV) on Ir/Mo capped multilayer before and after implantation (session B) is shown in Fig. 3: the data show an increase of the carbon emission after session B, which should be attributed to a deposition of a carbon layer on the top of the structure due to contamination during implantation.

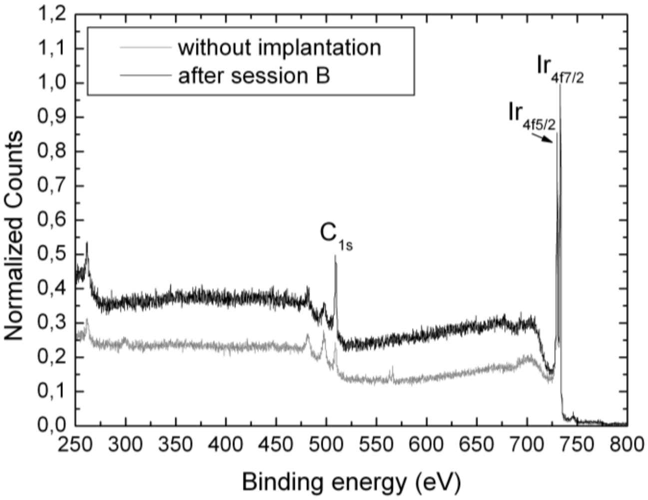

Fig. 3. XPS survey for the Ir/Mo overcoated structure before and after implantation.

An estimation of the carbon layer thickness of ~2nm has been determined from a fit of the Si/Mo reflectance measurements. This overcoating causes an increase in the interference effect of the last two layers. This same layer does not have any effect if deposited on top of a heavy metal capping layer, as in case of ruthenium and iridium. This overcoat cannot explain the wavelength shift observed in all samples, neither the dramatic loss of reflectivity of the Ru/Mo capped multilayer. The roughness of the film surfaces has been verified by AFM before and after implantation; no increase in roughness was found in any of the samples. TEM analysis was carried out on the most damaged sample, which is Ru/Mo capped multilayer after session B. Bright-Field cross-sectional images of the multilayer prior and after bombardment are shown in Fig. 4. The apparent mechanism of ML damage is the formation of a vacancy layer beneath the second Si layer from surface. Clearly, the first top layers of the structure are peeled off from the underneath structures, and this determined a definitive degradation of the multilayer.

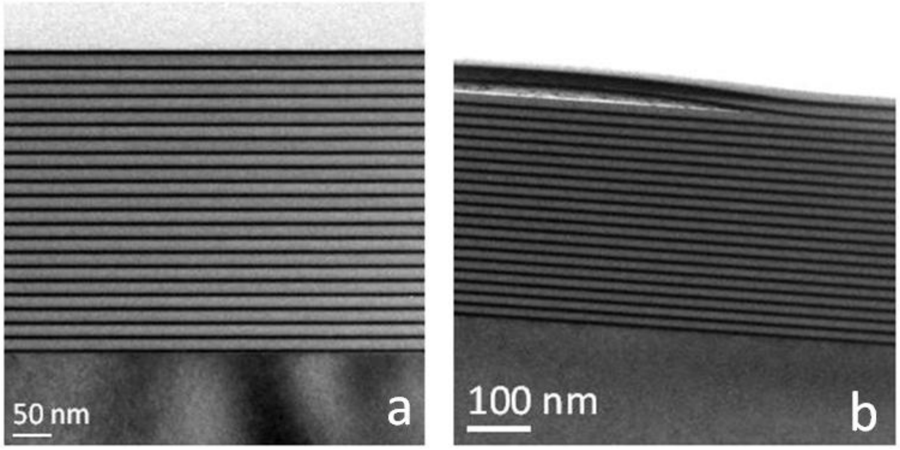

Fig. 4. Bright-Field cross-sectional TEM image of the Multilayer, prior (a) and after (b) proton bombardment.

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4. Conclusion

For the first time multilayer coatings for high EUV reflectance have been irradiated by low energy (1 keV) protons to verify their stability over time in Sun close heliosphere. Optical characterization of the sample prior and after the experiment shows that proton bombardment can strongly damage the nanostructures. All samples show a change in reflectivity (peak and wavelength shift), which has been demonstrated to be more dramatic in case of a Ru/Mo capping layer, and lesser sever in case of an Ir/Mo or Ir/Si capping layer. The physical processes happening on top surface has been investigated by TEM analysis, which shows that the damage consists in the formation of a vacancy layer underneath the first top layers. From the work completed thus far, we can conclude that Ir/Mo and Ir/Si capped multilayer provides the highest efficiency at 30.4 nm and a better stability to proton bombardment, and therefore are up to now considered as best candidate for SOLO-METIS instrument. Nevertheless, other tests are needed to fully validate these coatings, in particular using other ion species as alpha-particles.

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