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Coatings with high 102.6-to-121.6 nm reflectance ratio

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ABSTRACT

Observations in the far ultraviolet (FUV) at wavelengths below ~125 nm, which include the H Lyman series and the spectral lines of many other important species, are expected to unveil fundamental information for solar physics and astrophysics. Among these, observations of the solar corona at 102.6 nm H Lyman β are of high interest, but they may be masked by the strong H Lyman α at 121.6 nm. This goal has been addressed here through the development of novel multilayer coatings with high reflectance at 102.6 nm and at the same time a low reflectance at 121.6 nm; the latter wavelength is mostly absorbed. An efficient reflection/rejection coating is not straightforward because of the lack of high-transmission materials in the short FUV. We have designed and prepared novel multilayers with combinations of the following materials: Al, LiF, SiC and C. Various combinations were found to display a high reflectance ratio at 102.6/121.6 nm when fresh. Some of them resulted in an undesired reflectance increase at 121.6 nm for the samples aged for a few weeks. The most promising multilayers are based on Al/LiF/SiC/LiF (starting with the innermost layer), which resulted in a good performance and a small evolution after months of storage in a desiccator. At the same time, these multilayers may the most efficient reflective narrowband coatings that have been developed with a peak wavelength in the ~100-130 nm.

Keywords: Multilayers, Far Ultraviolet, Extreme Ultraviolet, Solar physics, Filters, Space optics, Mirrors

1. INTRODUCTION

Observations in the far ultraviolet (FUV, λ~100-200 nm) and extreme ultraviolet (EUV, λ~10-100 nm) parts of the spectrum will unveil fundamental information for solar physics and astrophysics. The emission of hot environments and the electronic transitions of the primary molecules in the solar chromosphere and corona, in stars, in galaxies or in the interstellar medium can be optimally observed in many cases in the FUV-EUV ranges. These observations, which must be performed outside the terrestrial atmosphere, will help solve fundamental unknowns for solar physics and astrophysics about the plasma diagnostics of the solar atmosphere, galaxy formation, the origin and evolution of planets and stars, etc.

FUV-EUV observations for solar physics and astrophysics are limited in a chief part by the performance of coatings. For short EUV wavelengths (~12.5 nm to 50 nm), important achievements have been obtained in the last years based on novel coatings with relatively high and narrowband performance. These coatings are possible due to the availability of materials that are transparent enough. The development of these coatings has provided valuable information for solar physics and astrophysics, such as mapping the distribution of specific spectral lines in various environments.

Contrarily, the EUV/FUV spectral range between ~50 and 125 nm has been lacking efficient coatings due to the unavailability of transparent materials to develop efficient multilayers. New coating development for this range is of high importance, since it contains the ubiquitous H Lyman α and the rest of the Lyman series, along with the spectral lines of many fundamental species with various ionization states. So far, coatings for this range have been mostly limited to single layer coatings (sometimes overcoated with a protective overlayer). Recently, narrowband multilayers have been developed peaked around ~60 nm[1,2]. Our group has also developed narrowband multilayer coatings based on Yb[3] or Eu[4] peaked in the ~50-100 nm range. Other than this, In presents a natural transmittance band centered at 80-90 nm. At longer wavelengths, narrowband metal-dielectric transmittance coatings are available above 120 nm[5].

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The needs of coatings for solar physics and astrophysics include narrowband coatings for the best performance at H Lyman α (121.6 nm), coatings with rejection at this wavelength and high performance at close spectral lines of H, O, etc, polarizers or retarders at H Lyman α and shorter wavelengths, etc. The observation/ mapping of the solar chromosphere, transition region and corona relies upon strong lines such as Lyman α and β (102.6 nm) of neutral hydrogen, OVI (103.2 and 103.7 nm) and other lines below 160 nm. These lines are useful for a diagnostic of the respective regions, in terms of plasma parameters and flows. Nowadays, the efficiency of FUV optics limits the imaging instrument performance, and these optics are a key element for the future generation of space solar instruments.

In this proceeding we address the development of coatings with high reflectance at H Lyman β (102.6 nm) and at the same time are able to reject Lyman α (121.6 nm) with a small reflectance at this line, which is the strongest line of the solar spectrum[6]. Other close fundamental spectral lines for solar physics close to H Lyman β, such as OVI 103.2 and 103.7 nm, might also benefit of such novel coatings. A pioneering work on this subject was published by Edelstein[7]. Other than this, a relatively similar problem has been addressed in the literature, which is the development of coatings with a good 83.4-to-121.6 nm reflectance ratio, 83.4 nm being an O II line[8,9,10].

The main difficulty to meet the present requirement is the scant materials that are transparent enough in this range in order to develop efficient multilayers. In this respect, 102.6 nm is an easier target than the above mentioned 83.4 nm, since the former is still on the edge of the relatively transparent side of LiF, the material in nature that keeps transparency down to the shortest possible wavelength. For LiF, the transparent-to-absorbing cutoff is at ~105 nm (the exact wavelength depends on cutoff definition and on the specific set of optical constants used); LiF absorption increases fast with decreasing wavelength, but 102.6 nm is still close enough to the cutoff as to make LiF the best choice of transparent material for multilayer coatings peaked at 102.6 nm.

The proceeding is organized as follows. Section 2 gives a short description of the experimental equipment used. Section 3 presents the various designs that have been followed and the reflectance measurements on various fresh and aged multilayers that have been prepared.

2. EXPERIMENTAL EQUIPMENT

Present coatings for the FUV-EUV were deposited and measured in a combined reflectometer and deposition system at GOLD. For the preparation of high quality coatings, we have developed a new deposition system able to coat large optics in clean-room conditions. We explain sample preparation and describe these two pieces of equipment in the following sub-sections.

2.1 Sample preparation

Multilayer coatings were deposited in ultra high vacuum (UHV) conditions in the reflectometer-deposition system that is described in the next subsection. Films of Al and LiF were deposited by evaporation using tungsten multi-stranded filaments (Al), and Mo boats (LiF); the evaporant material was 99.999% pure Al and VUV-grade LiF; the deposition rate was 0.5-0.9 (Al) and 0.2-0.5 (LiF) nm/s; pressure during deposition was $\sim 10^{-6}$ (Al) and $\sim 2\times10^{-7}$ (LiF) Pa. Films of SiC and C were deposited by ion-beam-sputtering (IBS) by impinging energetic ions at 45° on a target placed facing the substrate. We used 96.5-mm diameter targets of CVD-SiC and of C, with 99.9995% (SiC) and 99.999% (C) purity. The targets were placed in a rotatable target holder that hosts up to four targets, which are cooled down with water. Ions were produced by means of a 3-cm hollow cathode ion gun working with a hollow cathode neutralizer; this gun and neutralizer contain no filament, which minimizes contamination. Typical deposition conditions were ion energy of 1200 eV and a total ion current of ~50 mA. Ar was used as a process gas, and the pressure in the chamber during the sputtering process was $\sim 7\times10^{-2}$ Pa. The films were deposited onto non-intentionally heated or cooled polished float glass substrates.

2.2 GOLD reflectometer and deposition system

Present samples were prepared and their reflectance measured in this system. It consists in a deposition system connected to an FUV-EUV reflectometer, both working under UHV. In this reflectometer-deposition system, multilayer coatings can be prepared using two different deposition techniques: IBS and evaporation, and both were used in this research. Since the two techniques are in different UHV chambers that are connected in vacuum, the samples travelled without breaking vacuum from the evaporation to the IBS chamber and back in order to alternate evaporation and sputtering in the multilayer. Base pressure in the evaporation and IBS chambers were ~$2x10^{-8}$ and 7×10^{-8} Pa, respectively. When the multilayer was ready, we transferred it to the reflectometer without breaking vacuum, so that reflectance could be measured for freshly-deposited coatings; the reflectometer enables transmittance measurements too.

The reflectometer has a grazing-incidence, toroidal-grating monochromator, in which the entrance and exit arms are 146º apart. The monochromator covers the 12.5-200-nm spectral range with two Pt-coated diffraction gratings that operate in the long (250 l/mm) or in the short (950 l/mm) spectral range. A windowless discharge lamp was used in this work. The lamp is fed with various pure gases or gas mixtures with which it can generate many spectral lines to cover the spectral range of interest. The beam divergence was ~5 mrad and angle accuracy is estimated as ±0.1º. The sample holder can fit samples up to 50.8x50.8 mm^2. A channel electron multiplier with a CsI-coated photocathode was used as the detector. Reflectance was obtained by measuring alternately the incident intensity and the intensity reflected by the sample. Reflectance measurements were performed at 5º from the normal.

Here are the main characteristics of GOLD's reflectometer-deposition system:

- Deposition by evaporation in UHV with resistive sources (such as filaments) and electron beam
- Deposition by IBS in UHV
- Deposition of multilayers with up to 7 different materials
- In situ reflectance and transmittance measurements in the 12.5-200 nm range
- Reflectance / transmittance can be measured again after sample ageing outside vacuum
- Reflectance measurements versus incidence angle from near normal (~3º) to near grazing incidence (~87º)
- Controlled coating exposure to gases, atomic oxygen or UV light, which can be performed in situ
- The reflectometer/ deposition system is placed in an ISO-8 clean room

2.3 New deposition system in clean-room conditions

Other than the above system in which the present samples were prepared, a new deposition system has been developed to fabricate coatings meeting demanding requirements. It consists in a UHV evaporation system which is placed in an ISO-6 clean room. The deposition chamber has a 75-cm diameter and a height of 100 cm. The system is pumped with a cryopump. The first goal of this new deposition system is to deposit coatings by evaporation. A goal for the future is to combine evaporation and sputtering techniques. This new facility has been developed to be able to prepare coatings for the most demanding requirements, as are coatings for the FUV-EUV for future space instruments.

3. RESULTS

In order to design multilayers with the largest possible reflectance at 102.6 nm and the largest 102.6/ 121.6 nm reflectance ratio, the first step was to select the most promising materials. In his previous work, Edelstein[7] tried multilayers based on an innermost Al layer, a second layer of LiF and three choices for the outermost layer: SiO_2, Al_2O_3, and Au. All of his trial multilayers consisted on a single layer per material. He obtained best results for the multilayer with Al_2O_3, which resulted in aged coatings with a reflectance at 121.6 nm and near normal incidence of ~1.4%, with a further decrease away from normal incidence. The reflectance at 102.6 nm was ~30%.

In this work we have used a similar approach to the one used by Edelstein. Al and LiF innermost layers were again used, due to the large refractive index contrast between Al and LiF, and due to the relatively low absorption of LiF down to 102.6 nm, although absorption of LiF already starts to increase below ~105 nm. The coincidence finishes here, and we explored new combinations for outermost materials involving carbides. This choice was based on a heritage of multilayers with high reflectance in the long EUV to short FUV, based on Al, MgF_2 and SiC or B_4C[11]. These multilayers had a peak reflectance at wavelengths longer than the MgF_2 transparency cutoff at 115 nm; in the present case a high reflectance was required down to a shorter wavelength such as 102.6 nm, so that MgF_2 needed to be replaced with LiF. SiC, B_4C, and C were considered for the third material in the multilayer. The purpose of this third-layer material is to add the required destructive interference in combination with the inner layers in order to reject reflectance at 121.6 nm, and provide at the same time a high reflectance at 102.6 nm. One difficulty with the present multilayers is that they have to be stable enough as to keep a good performance over time. In this respect, IBS-deposited SiC and B_4C films undergo a limited oxidization in contact with air, mainly after the first contact, and this oxidization slows down and eventually stops after a few weeks or months. Since the stability of the Al/LiF/SiC and Al/LiF/B_4C multilayers was somewhat unknown a priori, two possible protective single-layer coatings were considered. One option was a very thin film of C, which was fixed at a thickness of 2.0 nm. A second option was to deposit a further LiF layer on top of SiC. The table displays the layer thicknesses of some possible multilayer designs for the different material combinations that were considered. We used the optical constants available in the literature: Al[12,13], LiF[14], SiC (either for unexposed SiC characterized in situ[15,16] or for SiC after a short contact to atmosphere[17]), B_4C[18,19], and C[20].

Table 1. The layer thicknesses, starting with the innermost layer, of various multilayer designs and the calculated normal reflectance at 102.6 nm[a]

Thickness (nm)								Refl. at 102.6 nm
Al	50	LiF	11.3	SiC	4.0			0.737 (n,k of non-exposed SiC)
Al	50	LiF	10.9	SiC	5.0			0.730 (n,k of shortly exposed SiC)
Al	50	LiF	8.6	B_4C	3.2			0.587
Al	50	LiF	4.3	C	6.1			0.204
Al	50	LiF	9.2	SiC	2.7	C	2.0	0.644
Al	50	LiF	6.7	B_4C	2.0	C	2.0	0.400
Al	50	LiF	14.5	SiC	8.8	LiF	15.1	0.549

[a] The calculated normal reflectance at 121.6 nm is either zero or several orders of magnitude lower than at 102.6 nm. For multilayers with SiC as the outermost layer, the upper and the lower design use SiC optical constants measured in situ[15,16] or after a short contact to atmosphere[17]. The other designs use SiC optical constants measured in situ.

Three multilayer designs were selected: Al/LiF/SiC, Al/LiF/SiC/C, and Al/LiF/SiC/LiF. Figs. 1, 2 and 3 summarize the reflectance measured for samples of the 3 designs, both fresh (non-exposed to the atmosphere) and after a contact to the atmosphere and some storage period in a desiccator.

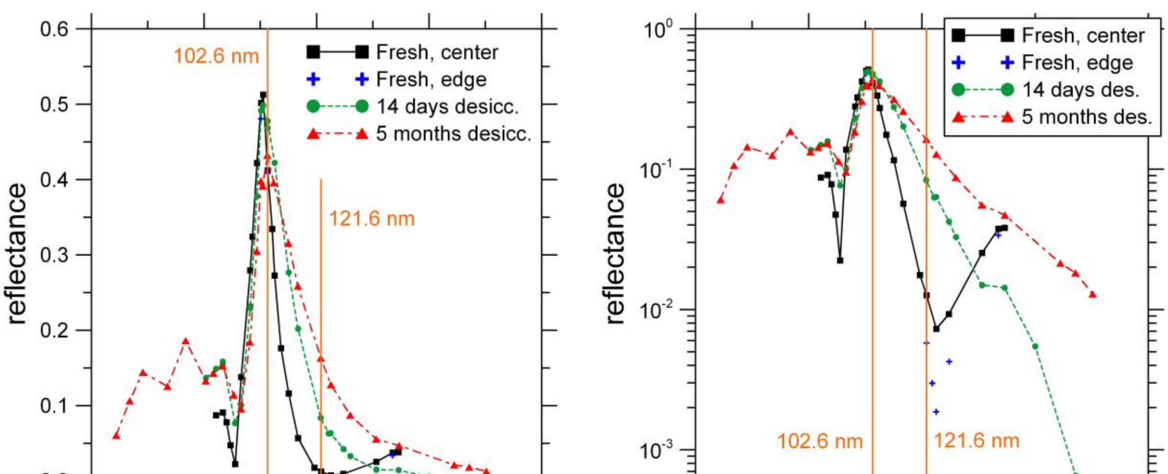

Fig. 1. The reflectance of an Al/LiF/SiC multilayer when fresh and after a storage of 14 days and of 5 months in a desiccator. Measurements that are plotted for the fresh multilayer were performed both in the center and on one edge of the sample where the reflectance at 121.6 nm was minimum. Left: linear reflectance. Right: the logarithm of reflectance for the same data plotted in the left picture

The three designs resulted in multilayers with a reflectance lower than 1% at 121.6 nm and a reflectance at 102.6 nm of 0.41 for the Al/LiF/SiC multilayer, 0.24 for the Al/LiF/SiC/C, and 0.40 for the Al/LiF/SiC/LiF multilayer. The reflectance peak was at 101.0 nm for the Al/LiF/SiC multilayer, at 100.1 nm for the Al/LiF/SiC/C multilayer, and at 100.4 nm for the Al/LiF/SiC/LiF multilayer.

All samples were stored in a desiccator for ageing purposes. What made a big difference among the 3 attempted designs was the reflectance stability at 121.6 nm upon ageing. Regardless of the reflectance change at 121.6 nm, the reflectance decay over time at 102.6 nm was small or there was even an increase for the Al/LiF/SiC/LiF multilayer; in fact the latter increase at 102.6 nm seems to consist in a small peak shift over time towards longer wavelengths. The total contact to normal atmosphere may have totaled ~2 h. The multilayer with a SiC outermost layer and even more the multilayer with a C outermost layer resulted in a dramatic band broadening at wavelengths longer than the peak and a large reflectance increase at 121.6 nm so that the minimum reflectance vanished. Yet, the multilayer ending in SiC keeps a narrowband performance which might still be useful in an application in which a band in the ~100-120 nm range, but not a zero reflectance at 121.6 nm, is required.

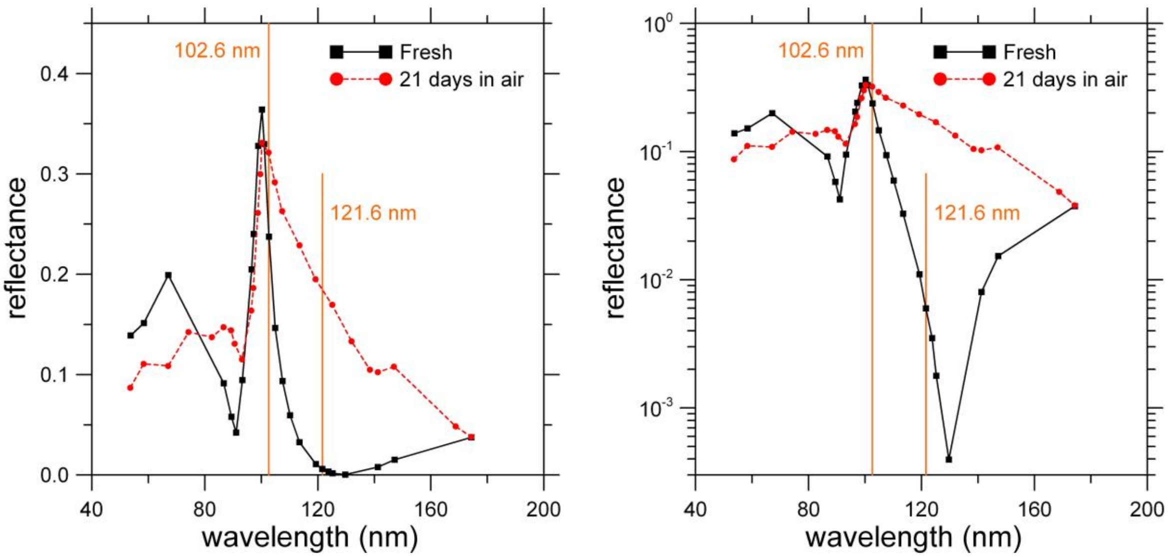

Fig. 2. The reflectance of an Al/LiF/SiC/C multilayer when fresh and after a storage of 21 days in a desiccator. Left: linear reflectance. Right: the logarithm of reflectance for the same data plotted in the left picture

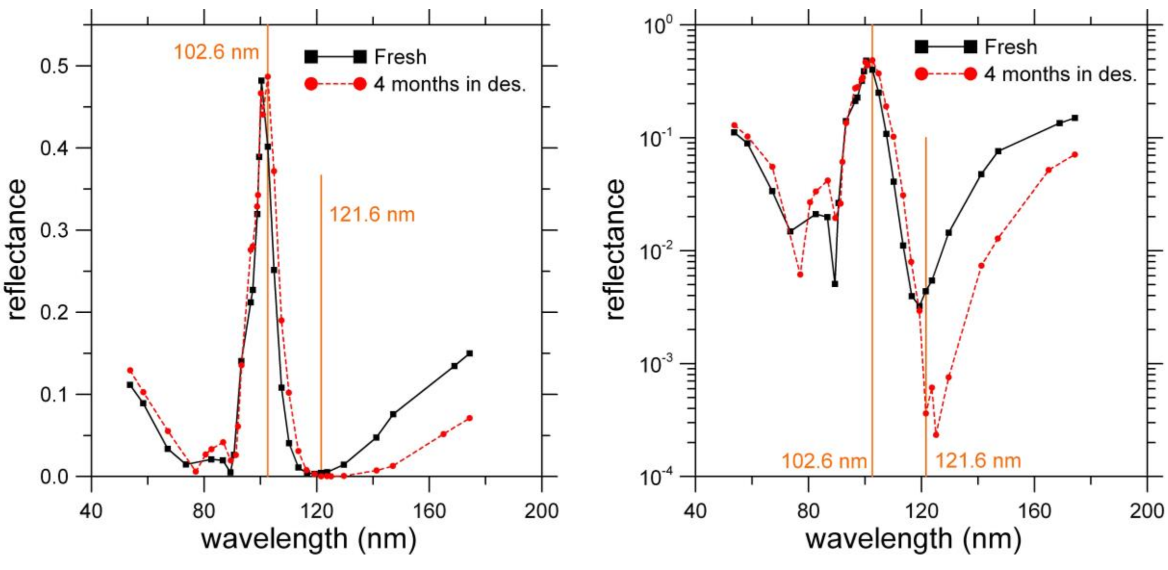

Fig. 3. The reflectance of an Al/LiF/SiC/LiF multilayer when fresh and after a storage of 4 months in a desiccator. Left: linear reflectance. Right: the logarithm of reflectance for the same data plotted in the left picture

The Al/LiF/SiC/LiF multilayer had by far the best performance upon ageing, where the reflectance at 102.6 nm has even increased to 0.487, as commented above, and the reflectance at 121.6 nm has even decreased, with the reflectance minimum turning deeper and shifting to longer wavelengths. Furthermore, reflectance at longer FUV wavelengths up to 174.4 nm decreased upon ageing with respect to the fresh coating; the latter results in that the Al/LiF/SiC/LiF multilayer can be considered not only a good 102.6/121.6 nm reflectance ratio coating, but also a high performance narrowband coating peaked at 102.6 nm, which may be the narrowband coating with the largest peak reflectance ever measured in the FUV range below ~130 nm. Some of these good results obtained for the Al/LiF/SiC/LiF multilayer may have been obtained under lucky circumstances in which ageing modified the performance in the best possible direction, and here comes a weak point of the 3 designs investigated in this research: their small tolerance on film thickness errors and on coating contamination due to the critical minimum reflectance at 121.6 nm. In fact the reflectance at 121.6 nm and the minimum wavelength were found to depend on the exact spot selected within the sample, whereas the reflectance away from the minimum was not observed to vary significantly across the sample. For both fresh and aged multilayers, reflectance measurements were performed on the sample area where reflectance at 121.6 nm was minimum, and this area somewhat shifted over time for each multilayer.

CONCLUSIONS

Novel multilayer coatings with high reflectance at 102.6 nm and at the same time a high rejection at 121.6 nm have been developed. Three multilayer designs were investigated: Al/LiF/SiC, Al/LiF/SiC/C, and Al/LiF/SiC/LiF. The three multilayer designs resulted in a high reflectance at 102.6 nm and in a high reflectance ratio at 102.6/121.6 nm when fresh. Additionally, multilayer realizations following the 3 designs, had a narrowband performance when fresh that was peaked at a wavelength close to 102.6 nm. Al/LiF/SiC and Al/LiF/SiC/C multilayers resulted in an undesired reflectance increase at 121.6 nm for the samples aged a few weeks or months, which makes them not useful for the present application. In spite of this, the Al/LiF/SiC multilayer kept a narrowband performance after ageing which might still be useful in an application in which a band in the ~100-120 nm range, but not a zero reflectance at 121.6 nm, is required.

The most promising multilayers were those consisting in Al/LiF/SiC/LiF, which resulted in an excellent 102.6/121.6 nm reflectance ratio, and in a favorable evolution of reflectance both at 102.6 nm and at the more critical 121.6 nm after 4-month of ageing in a desiccator. Furthermore, this multilayer resulted in a high performance narrowband coating, which may be the narrowband coating with the largest peak reflectance ever measured in the FUV range below ~130 nm.

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