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Narrowband coatings for the 100-105 nm range

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ABSTRACT

Observations in the far ultraviolet (FUV) at wavelengths in the ~100-105 nm range, which include 102.6 nm (H Lyman β), 103.2 and 103.8 nm (O VI lines), are expected to unveil fundamental information for solar physics and astrophysics. Often the intensity of those lines is weak, and they may be masked by more intense lines, such as H Lyman α at 121.6 nm for observations of the solar corona. Narrowband multilayers peaked in the ~100-105 nm have not been available because of the absorption of materials at these wavelengths along with a strong influence of contamination in this range. When efficient narrowband coatings are not possible, an option is the use of coatings with high reflectance at the target wavelength and simultaneously low reflectance at the undesired wavelength, such as 121.6 nm. High-reflective-narrowband coatings peaked at 100-105 nm have been developed. We have designed a four-layer system (Al/LiF/SiC/LiF) that results in a high H Lyman β-to-Lyman α reflectance ratio. Three samples with slightly different film thicknesses were prepared and measured in the 50-190 nm spectral range. All samples showed a promising reflectance ratio when fresh; however, some sample ageing was observed after months of storage in a desiccator, probably due to the effect of reaction with water vapor among other contaminants at the outermost layer (LiF). All samples retained a narrowband performance over time. The reflectance at 121.6 nm, which was very low on fresh samples, typically increased over time, although keeping a high 102.6-to-121.6-nm reflectance ratio. The same system results in an efficient narrowband coating peaked in the target spectral range. We measured a reflectance as high as 63% at the peak wavelength of 100.3 nm, at near-normal incidence, the highest experimental reflectance reported in this range for a narrowband coating.

Keywords: Multilayers, Extreme Ultraviolet, Far 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) 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.

Particularly, the 100-105 nm spectral range is of high importance, since it contains the H Lyman β line (102.6 nm) and other remarkable lines for solar physics, such as O VI (103.2 and 103.7 nm). These lines are useful for the observation/ mapping of the solar chromosphere, transition region and corona, in terms of plasma parameters and flows. Nowadays, the efficiency of FUV optics limits the imaging instrument performance, and the development of novel optics, and more specifically coatings for the 100-105-nm range, are a key element for the future generation of space solar instruments.

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FUV-EUV observations for solar physics and astrophysics are limited in a chief part by the performance of coatings. Novel coatings have been developed in the last years based on the existence of materials that are transparent enough. The recent availability of these coatings has resulted in instruments that are providing valuable information for solar physics and astrophysics, in addition to the coating use in other fields such as synchrotron and free electron radiation, plasma physics, lithography, etc. In the following we summarize the multilayer coatings that have been developed in the FUV-EUV.

In the 12.5-60 nm range, there are several materials with specific low absorptions bands, which can be used as spacers in multilayers, such as Si, Al, Mg and Sc. Therefore, there has been an important development of multilayers based on these spacers, with relatively high and narrowband performance, such as Mo/Si[1], Sc/Si[2], B_4C/Si[1], Al/SiC[3], SiC/Si[1], and Mg/SiC[4].

In the 60-100 nm range, most materials have a strong absorption, which has precluded the development of multilayer coatings until recently. Exceptions to this are the lanthanide elements, who present a low absorption band in this region. Thus, some lanthanide-based multilayers with relatively narrow performance, and with a moderate high reflectance have been developed in the last years[5,6,7,8]. Recently, new Mg/SiC[9] multilayer coatings have been developed that efficiently reflect radiation in single or multiple narrow bands centered at wavelengths in the spectral region from 25 to 80 nm.

In the FUV, MgF_2 is the preferred spacer material down to its cutoff (wavelength above which the material is transparent) at ~115 nm. Narrowband multilayer coatings have been developed that alternate MgF_2 with a second material that was selected among either Al (mostly for transmittance filters) or some fluorides, like BaF_2, LaF_3 or NdF_3[10,11].

Contrarily, the spectral range between 100 and 115 nm, and particularly, the H Lyman β line (102.6 nm), has been lacking efficient narrowband coatings. Fortunately, the H Lyman β 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: its transparent-to-absorbing cutoff is at ~105 nm; 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[12]. Until now, no narrowband performance has been possible other than the natural transmittance bands of few usable materials, such as In and LiF, with a relatively modest performance[13]. Finally, above ~100 nm, wideband mirrors based on Al overcoated with LiF, are available with high reflectance at Lyman β[14,15,16].

Often the intensity of the H Lyman β line and of close lines is weak in practical applications, and these lines may be masked by more intense lines, such as H Lyman α at 121.6 nm, which is the strongest line of the solar spectrum[17,18]. A pioneering work on the development of coatings with high Lyman β-to-Lyman α ratio was published by Edelstein[19]. In view that narrowband coatings were not available, he developed coatings that aimed at optimizing the above ratio, regardless of the performance at other wavelengths. 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[20,21,22].

In this proceeding we address the development of narrowband coatings with high reflectance at H Lyman β based on a four-layer system (Al/LiF/SiC/LiF) that show a good performance both fresh and over time. We have made a design aimed to keep a good reflectance at H Lyman β and, at the same time, to reject H Lyman α, with a small reflectance at this line. The design shows an efficient reflection/rejection ratio for the H Lyman β-to-Lyman α.

The proceeding is organized as follows. Section 2 gives a short description of the experimental equipment used. Section 3 presents four Al/LiF/SiC/LiF filters with high reflectance in the H Lyman β and almost zero reflectance in the H Lyman α. Sample ageing is also presented.

2. EXPERIMENTAL EQUIPMENT

Present coatings for the FUV-EUV were deposited and measured in a combined reflectometer and deposition system at GOLD. 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 W 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 ~10^{-6} (Al) and ~$2x10^{-7}$ (LiF) Pa. Films of SiC were deposited by ion-beam-sputtering (IBS) by impinging energetic ions at 45° on a target placed facing the substrate. A 96.5-mm diameter target of CVD-SiC of 99.9995% purity was used. The target was 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 ~60 mA. Ar was used as a process gas, and the pressure in the chamber during the sputtering process was increased to ~$7x10^{-2}$ 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
- Reflectance measurements versus incidence angle from near normal (~3º) to near grazing incidence (~87º)
- Controlled coating exposure to gases, atomic oxygen, or UV light can be performed in situ
- The reflectometer/deposition system is placed in an ISO-8 clean room

3. RESULTS

In order to design multilayers with the largest possible reflectance at 102.6 nm and with a high Lyman β-to-Lyman α ratio, the first step was to select the most promising materials. In this work we have followed a similar approach to the one used by Edelstein[19], who developed coatings 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. We have followed a design that is also based in Al and LiF innermost layers, due to the large refractive index contrast between Al and LiF, in addition to the relatively low absorption of LiF down to this range. Yet, we selected SiC for the third material in the multilayer, due to the relatively low absorption and high reflectance in this region. However, Al/LiF/SiC multilayers resulted in a reflectance increase under ageing at wavelengths longer than the peak[12]; therefore we added a second LiF layer on top of SiC in order to avoid the limited SiC oxidation in contact with the atmosphere.

A four-layer system (Al/LiF/SiC/LiF) filter was designed in order to obtain narrowband performance peaked at ~102.6 nm. The first design used was as follows: 45 nm Al/14.4 nm LiF/8.8 nm SiC/15.1 nm LiF. In the design we used the optical constant data available in the literature for Al[23,24], LiF[25] and SiC[26,27]; due to the difference mainly in absorption between thin films and bulk materials in this spectral range, we used optical constants measured on thin films.

We prepared three samples (#1, #2 and #3), based all on the design. Fig.1 shows the reflectance measured on these coatings when fresh

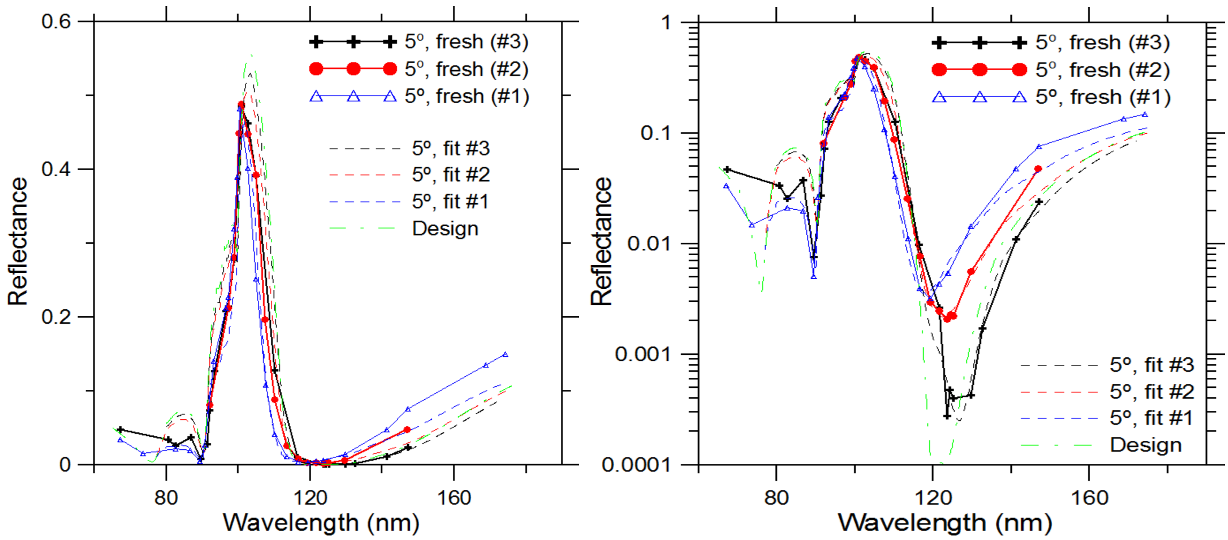


Fig. 1. The reflectance as a function of wavelength of samples #1, #2 and #3 when fresh. The design and fit are also plotted. Left and right: reflectance in linear and logarithm scale, respectively

The above measurements for fresh coatings were very promising. The Lyman β-to-Lyman α ratios for fresh samples were 177, 180 and 93 for samples #3, #2, and #1, respectively. As shown in Fig. 1, each filter approaches better to the design than the previous one.

We fitted these reflectance measurements in order to refine the thicknesses of each layer in the multilayer, and the results are shown in fig. 1. The fits were calculated using the IMD software[28]. Table 1 summarizes the layer thicknesses in the original design and the ones obtained in the fits.

Table 1. Layer thicknesses of the Al/LiF/SiC/LiF multilayer for samples #1, #2, and #3.

Sample #	Al thickness (nm)	LiF thickness (nm)	SiC thickness (nm)	LiF thickness (nm) (outermost)
Design	**45.0**	**14.4**	**8.8**	**15.1**
1	45.0	12.5	8.2	12.8
2	45.0	14.4	8.7	14.7
3	45.0	14.8	8.8	15.3

According to table 1, the LiF film thicknesses in sample #1 were shorter than in the design. It was originated in an incorrect initial thickness calibration of LiF. This film thickness was hence increased in samples #2 and #3.

All filters were measured again after some storage period in a desiccator. Figs. 2, 3 and 4 summarize the experimental reflectance measured for the samples #1, #2 and #3, when fresh and after storage in a desiccator.

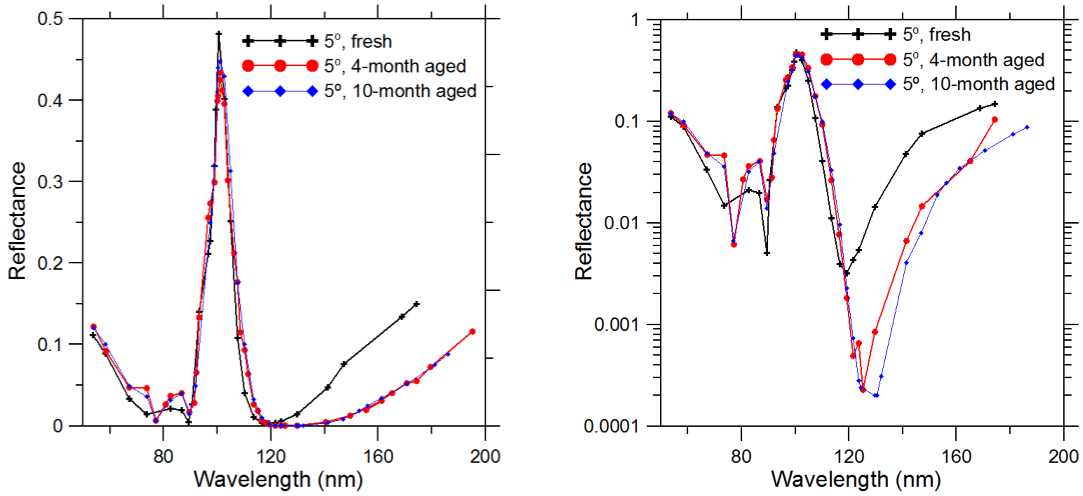


Fig. 2. The reflectance as a function of wavelength for sample #1 when fresh and after storage of 4 and 10 months in a desiccator. Left and right: reflectance and its logarithm, respectively.

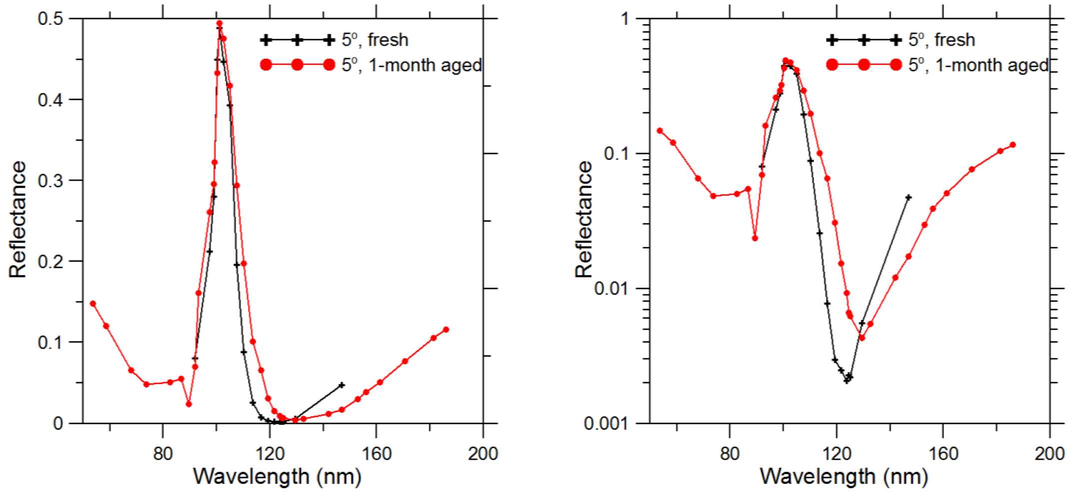
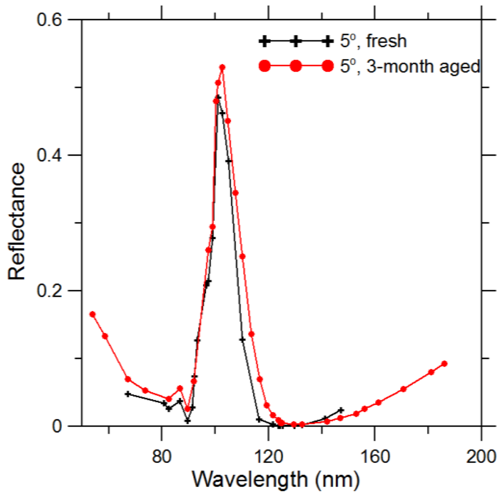


Fig. 3. The reflectance as a function of wavelength for sample #2 when fresh and after storage of 1 month in a desiccator. Left and right: reflectance and its logarithm, respectively.



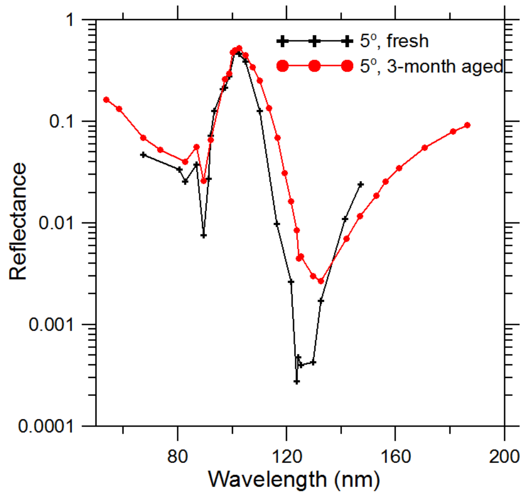


Fig. 4. The reflectance as a function of wavelength for sample #3 when fresh and after storage of 3 months in a desiccator. Left and right: reflectance and its logarithm, respectively.

All these samples had a large Lyman β-to-Lyman α reflectance ratio when fresh; however, after some storage in a desiccator, we found that reflectance increased at 121.6 nm upon ageing, except for sample #1, where there was some reflectance evolution over time but there was no significant change at 121.6 nm. The reflectance increment at 121.6 nm and close range for the samples seems to consist in a small shift at the minimun towards longer wavelengths. In sample #1, the minimum reflectance for the fresh sample was at a wavelength shorter than 121.6 nm (119 nm), and therefore the minimum shift did not result in a significant change. Therefore, even though fresh samples #2 and #3 reproduced more accurately the nominal design, the evolution over time turned sample #1 as the one that kept lowest reflectance at 121.6 nm. In spite of this change at 121.6 nm, the reflectance of all samples slightly increased over time at 102.6 nm, which is attributed to some band widening.

The Lyman β-to-Lyman α ratio for aged samples #3, #2 and #1 was 33, 32, and 614, respectively. Table 2 summaries the main parameters of each sample.

Table 2. Summary of the main parameters of samples #1, #2, and #3: reflectance at Lyman β, at Lyman α and their ratio, bandwidth (FWHM), peak wavelength and peak reflectance for both fresh and aged coatings.

FRESH						
Sample #	R @ L β	R @ L α	FWHM (nm)	Ratio Lβ/Lα	Peak (nm)	R @ Peak
1	0.40	0.0043	7.6	**93**	100.3	0.48
2	0.45	0.0025	8.6	**180**	100.9	0.49
3	0.46	0.0026	9.5	**177**	100.9	0.49
AGED						
Sample #	R @ L β	R @ L α	FWHM (nm)	Ratio Lβ/Lα	Peak (nm)	R @ Peak
1	0.43	0.0007	10.1	**614**	100.9	0.45
2	0.48	0.015	11.9	**32**	100.9	0.49
3	0.53	0.016	12.2	**33**	102.6	0.53

For comparison purposes, Edelstein`s aged Al_2O_3-based coatings had a reflectance at 121.6 nm and near normal incidence of ~0.014, and the reflectance at 102.6 nm was ~0.30[19], which gives a Lyman β-to-Lyman α reflectance ratio of ~21.

The narrowband coatings displayed above have a high Lyman β-to-Lyman α reflectance ratio. These are large ratios, although often the intensity of Lyman α line may be considerably larger than Lyman β: according to data from OSO-8, which were analyzed by Lemaire *et al.*[18], on the solar disk (radiance), the average quiet Sun Lyman α-to-Lyman β line energy ratio is in the 76-90 range. A further larger rejection of Lyman α line can be obtained upon reflection on two successive mirrors coated with the present multilayer design; the throughput at 102.6 nm would still be ~18% and a large enough Lyman β-to-Lyman α throughput ratio would be obtained as to imaging Lyman β in a background of Lyman α as the one reported by Lemaire *et al.*[18]

Although the shape of the previous filters was narrow, another four-layer system (Al/LiF/SiC/LiF) filter was designed with the target that the band and the reflectance at 102.6 nm were as narrow and as high as possible. The new design was: 45 nm Al/12.0 nm LiF/10.2 nm SiC/14.8 nm LiF.

Fig. 5 shows the reflectance measured for this sample, both fresh and after seven weeks of storage in a desiccator.

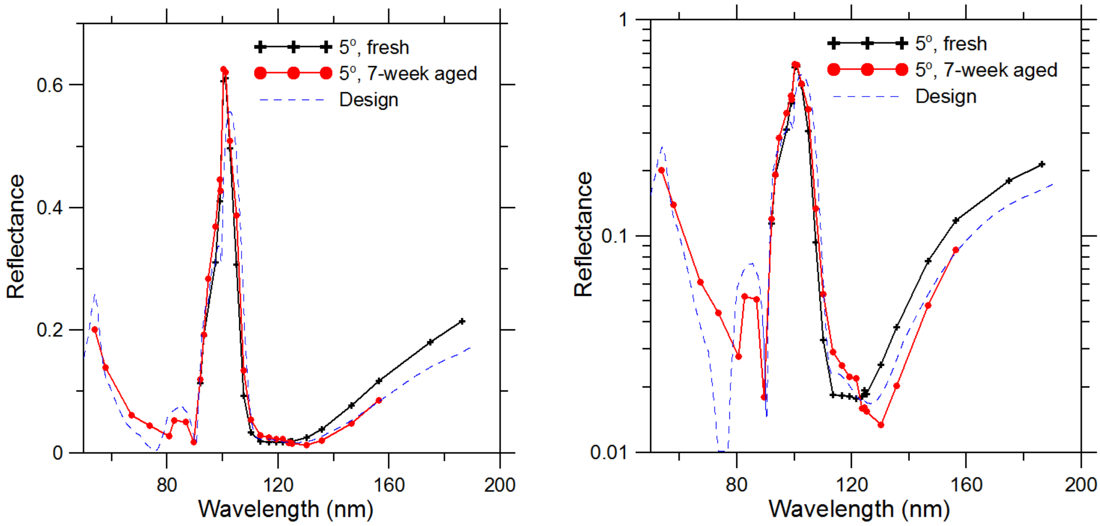


Fig. 5. The reflectance as a function of wavelength for sample #4 when fresh and after storage of 7 weeks in a desiccator. The design is also plotted. Left and right: reflectance and its logarithm, respectively.

This multilayer has a reflectance at 102.6 nm of 0.50. The reflectance peak was 0.61 at 100.9 nm and the reflectance at 121.6 nm was 0.018. Although all coatings were designed to reach the maximum reflectance at Lyman β, all of them were peaked at around 100.3-100.9 nm. Then, we attribute this slight peak shift to some uncertainty mainly in the set of LiF optical constants used in the 100-102.6 nm range. This sample was stored for 7 weeks in a desiccator for ageing purposes, and after that time, the reflectance change at 102.6 nm was negligible, from 0.50 to 0.51; the reflectance at the peak changed from 0.61 at 100.89 nm to 0.63 at 100.3 nm.

Even though the reflectance change around the peak was negligible, the reflectance at 121.6 nm increased in a more significant value, from 0.018 to 0.022. Regarding the behavior of the FWHM of the filter over time, it was 7.7 nm when fresh and 9.9 nm after 49 days in a desiccator. Table 3 summarizes the main parameters of sample #4.

Table 3. The main parameters of sample #4: reflectance at Lyman β, at Lyman α and their ratio, bandwidth, peak wavelength and peak reflectance for both the fresh and the aged coating.

FRESH						
Sample #	R @ L β	R @ L α	FWHM (nm)	Ratio Lβ/Lα	Peak (nm)	R @ Peak
4	0.50	0.0177	**7.7**	28	100.9	**0.61**
AGED						
Sample #	R @ L β	R @ L α	FWHM (nm)	Ratio Lβ/Lα	Peak (nm)	R @ Peak
4	0.51	0.022	**9.9**	23	100.3	**0.63**

In the following we compare the present narrowband coating with the literature. Carruthers[13] prepared filters based on In and LiF, and the best performance was a transmittance of 0.038 at 102.6nm, with a FWHM of ~6 nm. The best result obtained by Edelstein[19] was for the multilayer with Al_2O_3 as the third material, which resulted in aged coatings with a ~0.30 reflectance at 102.6 nm; FWHM bandwidth could not be obtained for the aged sample from his plots.

Our filter was shown to reflect 0.63 at a wavelength of 100.3 nm at near-normal incidence, the highest experimental reflectance reported at this region for a narrowband coating.

CONCLUSIONS

We have designed a four-layer system (Al/LiF/SiC/LiF) optimized to obtain a large Lyman β-to-Lyman α reflectance ratio. Three samples were prepared with slight film thickness differences, and all of them resulted in a large reflectance ratio at the target wavelengths when fresh. All samples retained a narrowband performance over time. The minimum reflectance close to 121.6 nm resulted to be rather critical and in most cases it increased over time. The reflectance at 102.6 nm did not decay over time, but it slightly increased due to some peak widening and/or shift. Whilst all samples retained a large Lyman β-to-Lyman α reflectance ratio over time, one of them kept a value as large as the one measured when fresh; this different behavior over the samples is attributed to small film thickness differences.

The four-layer system (Al/LiF/SiC/LiF) was again optimized to obtain the narrowest possible coating with a reflectance as high as possible that is peaked in the 100-105 nm range. This sample was measured both fresh and after 7 weeks of storage in a desiccator, and no significant changes were found. The aged coating had a peak reflectance of 0.63 at a wavelength of 100.3 nm at near-normal incidence, and its FWHM was 7.7 nm when fresh and 9.9 nm after 7 weeks in a desiccator. This is the highest experimental reflectance reported at this wavelength for a narrowband coating.

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