

UV optical filters based in magnesium

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Abstract. This article presents the fabrication of magnesium fluoride (MgF₂) and magnesium oxide (MgO) thin film-based ultraviolet (UV) optical filters and compares their optical transmission. The MgF₂ thin-films were deposited by electron beam (e-beam) technique and their optical properties were characterized by ellipsometry. The effects of substrate temperature on the optical properties were studied. The MgF₂ optimum refractive indices were obtained with high substrate temperatures, 200 °C and 300 °C. Optical simulations were performed to compare the performance of MgF₂ and MgO in the fabrication of near UV narrow bandpass optical filters. While MgO based optical filters result in a higher transmittance peak intensity, especially at 350 nm, the MgF₂ optical filters are narrower, *i. e.*, present lower values of full width at half maximum (FWHM), mean value of 21 nm. The MgF₂/TiO₂ UV optical filters show a peak transmittance approximately of 70 % close to 400 nm as expected. The results were discussed with a focus on applications in fluorescent optical sensors for the peaks at 350, 370, 380 and 400 nm respectively.

1. Introduction

The use of magnesium oxide (MgO) as low-refractive index material for the near ultraviolet (UV) optical filters is already reported on literature [1]. The use of magnesium fluoride (MgF₂) as low refractive index material for UV optical filters is only reported for the visible range (central wavelength of 511 nm) [2]. The MgF₂ presents a wide transparency range from 120 nm to 8 μm, good mechanical properties, low optical absorption and low optical refraction index [3, 4]. MgF₂ thin-films are widely used in antireflection coatings and in dielectric interference filters [5]. Also, the MgF₂ thin-films suffer from high tensile stress and for reasonable durability must be deposited on heated substrates [6]. Dumas *et al.* deposited MgF₂ by electron beam (e-beam) technique at substrate temperatures from the room temperature to 300 °C and at a pressure of 1×10^{-7} mbar [7]. The work of Ristau *et al.* present a deposited MgF₂ by thermal e-beam with a substrate temperature of 300 °C at a pressure of 6×10^{-6} mbar [8]. Yang *et al.* conclude that the characteristics of the MgF₂ thin-films obtained at a substrate temperature of 200 °C were the best for an antireflection film for solar cells [9]. This work presents the study of MgF₂ as low-refractive index material for UV optical filters. The MgF₂ thin-films were deposited by e-beam technique and optical characterized. Also, optical

simulations were performed on TFCalc™ software to compare the performance of MgF₂ and MgO in the fabrication of narrow bandpass UV optical filters, between 350 nm and 400 nm. Additionally, a Fabry-Perot based MgF₂/TiO₂ UV optical filter was fabricated using e-beam and a reactive Radio Frequency (RF) sputtering technique. The fabricated UV optical filter was optically characterized. The obtained results were discussed taking into account specific applications on fluorescent optical sensors.

2. Simulation and methods

2.1. MgF₂/TiO₂ UV optical filters

The performance of the MgF₂/TiO₂ UV optical filters (considering the experimental refractive indices presented on Figure 3, at 200 °C) was evaluated. Moreover, for the simulation of MgF₂/TiO₂ UV optical filters, the refractive indices of TiO₂ thin-films (deposited by a reactive RF-sputtering) were used, since they are reported by the authors in [10]. Table 1 shows the thickness of each thin-film that forms each optical filter centered at 350, 370, 380 and 400 nm. The optimization function in continuous approach and interval of 10 nm for each peak was used. By this way, the precise thickness for each layer is obtained and results in a sharper and thin peak (high intensity and low full width at half maximum (FWHM)).

Table 1. Layer thicknesses for the simulated narrow bandpass UV optical filters with MgF₂/TiO₂.

Material	Filter transmittance peak (nm)			
	350	370	380	400
	Layer thickness (nm)			
TiO ₂		30		
MgF ₂		110		
TiO ₂		10		
MgF ₂	259	287	300	330
TiO ₂		10		
MgF ₂		110		
TiO ₂		30		

Figure 1 shows the simulation results extracted from TFCalc™ software of the near UV optical filters based in MgF₂/TiO₂ thin-films.

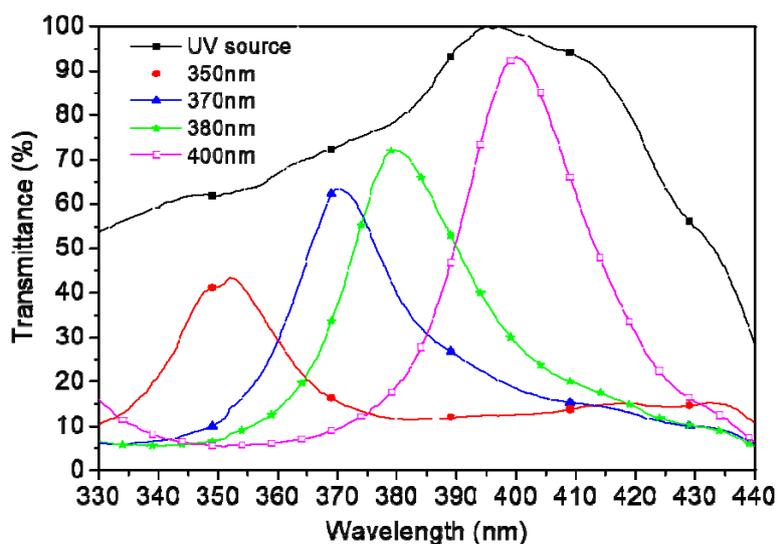


Figure 1. Transmittance of the MgF₂/TiO₂ simulated UV optical filters.

2.2. MgO/TiO₂ UV optical filters

Replacing the MgF₂ by MgO as the low-refractive index layer of the optical filter for near UV filters applied to optical sensors, the authors already reported the fabrication of narrow band pass optical filters between 350 nm and 400 nm using the MgO material [1]. The work of Pimenta *et al.* reported using MgO as low-refractive index material and TiO₂ as high-refractive index material. Both MgO and TiO₂ thin-films were deposited by Ion Beam Deposition (IBD) technique and their refractive indices were also obtained by ellipsometry [1]. Considering the MgO and TiO₂ refractive indices reported in [1], the performance of four optical filters, centered at 350, 370, 380 and 400 nm, was also simulated on TFCalcTM software. Table 2 shows the thickness of each thin-film that forms each UV optical filter.

Table 2. Layer thicknesses for the simulated narrow bandpass UV optical filters with MgO/TiO₂.

Material	Filter transmittance peak (nm)			
	350	370	380	400
	Layer thickness (nm)			
TiO ₂		30		
MgO		80		
TiO ₂		10		
MgO	215	237	248	275
TiO ₂		10		
MgO		80		
TiO ₂		30		

Figure 2 shows the simulation results extracted from TFCalcTM software of the near UV optical filters based in MgO/TiO₂ thin-films.

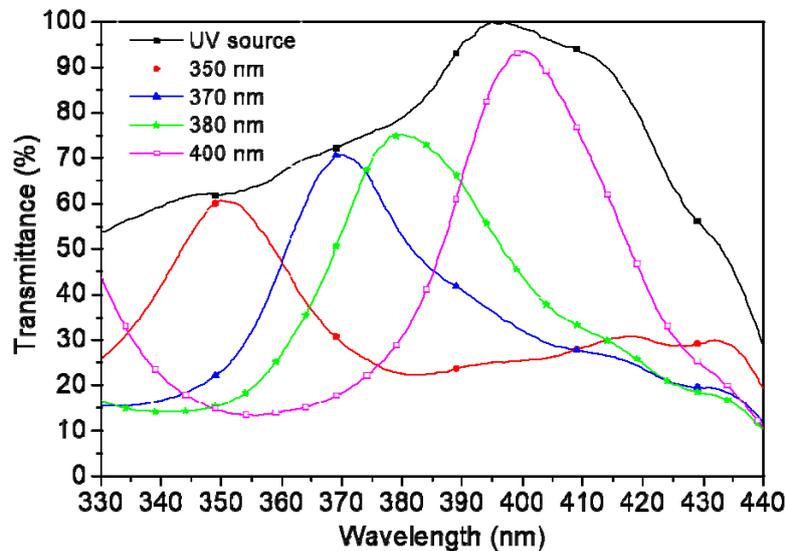


Figure 2. Transmittance of the MgO/TiO₂ simulated UV optical filters.

2.3. MgF₂/TiO₂ UV optical filters fabrication

The MgF₂ thin-films were deposited by e-beam technique at several substrate temperatures (100 °C, 200 °C and 300 °C). The distance between the MgF₂ pellets to substrate is approximately 150 mm. The different substrate temperatures were obtained by irradiance with 150 W halogen heating lamps. The heating lamps are connected to a controller and to a temperature sensor installed near the substrates. This simple and reliable technique insures a controlled temperature of the substrates. For each temperature step, an MgF₂ thin film with thickness of 300 nm was deposited, on a polished silicon (100) substrate for optical characterization by ellipsometry. Initially, the deposition chamber was evacuated to 7×10^{-6} mbar. Typically, the increase of temperature inside of the deposition chamber

induces the increase of the total pressure. Therefore, the optimum conditions to deposit the MgF₂ thin-films were archive with the e-beam power of 7 kV set at 10 mA and a deposition rate of 45 Å/s was obtained. A shutter was activated to ensure the precise thickness of 300 nm.

The TiO₂ thin-films were deposited by a reactive RF sputtering using a Titanium sputtering target. The optimum conditions were obtained at a deposition pressure of 2×10^{-3} mbar, 10 sccm of Ar, 2 sccm of O₂ and a power of 200 W with a deposition rate of 0.3 Å/s. The UV optical filter was deposited on borosilicate glass substrates and on polished silicon (100) substrates.

3. Characterization

3.1. MgF₂ thin-films ellipsometry

The refractive indices were obtained using an ellipsometer (J. A. Woollam). Figure 3 shows the refractive indices from 300 nm to 500 nm light spectrum of the 300 nm thick deposited MgF₂ thin-films. Also, is presented the refractive indices for MgF₂ thin-films reference reported by E. Palik [3]. The obtained refractive indices showed good agreement with the literature results, especially at high substrate temperatures (200 °C and 300 °C).

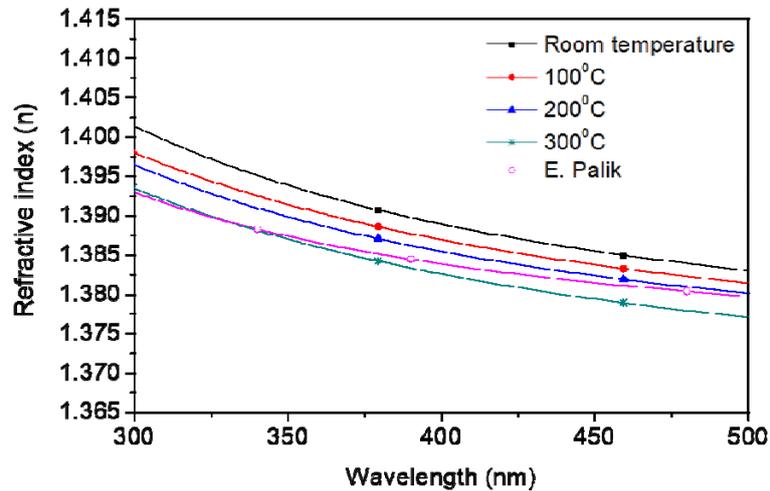


Figure 3. Refractive indices of MgF₂ thin-films.

4. Results and discussion

The MgF₂/TiO₂ UV optical filter programmed to be centered at 400 nm was fabricated, as described on section 2.1 and according to the thin-films thicknesses presented on Table 1. Figure 4 shows the optical transmittance obtained for the borosilicate glass substrates.

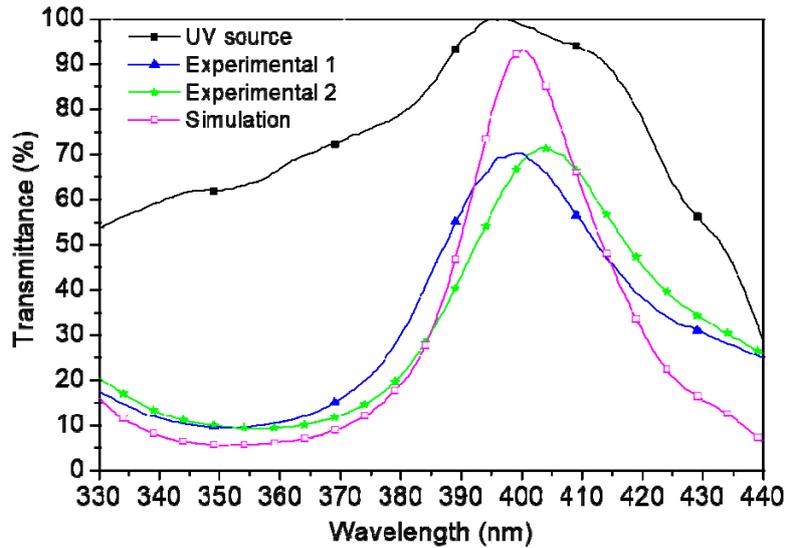


Figure 4. Experimental transmittance of the $\text{MgF}_2/\text{TiO}_2$ fabricated UV optical filter.

The peak transmittance of the UV optical filters is approximately 70 % and the peaks are slightly deviated from the 400 nm, 399 nm (for experimental 1) and 404 nm (for experimental 2). The difference between the two samples is due to the different position of the substrates in the deposition chamber. Moreover, the FWHM increase slightly, comparing with the simulation result. However, this increase could not be critical for some applications, for example, the extraction of fluorescent emission from Fluoresbrite[®] BB Carboxylate Microspheres, since the filter transmittance at 360 nm is very low when comparing to the transmittance at 407 nm. The total thickness is 619.5 nm (measured by ellipsometer), which is close to the expected result (630 nm). Table 4 shows the peak intensity and the FWHM for each optical filter.

Table 4. Comparison between the MgO/TiO_2 and $\text{MgF}_2/\text{TiO}_2$ UV optical filters.

Materials	Filter transmittance peak (nm)							
	350		370		380		400	
	Intensity (%)	FWHM (nm)	Intensity (%)	FWHM (nm)	Intensity (%)	FWHM (nm)	Intensity (%)	FWHM (nm)
MgO/TiO_2	60.75	32	70.74	28	75.23	30	93.72	28
$\text{MgF}_2/\text{TiO}_2$	41.29	20	63.53	22	72.17	20	93.07	22

With the combination MgO/TiO_2 is possible to obtain UV optical filters with a higher transmittance peak intensity, especially at 350 nm. However, combining $\text{MgF}_2/\text{TiO}_2$ the UV optical filters are narrower. This could be especially important for fluorescent optical sensors when part of the fluorescent molecule emission spectrum overlaps the molecule excitation spectrum. The FluoSpheres[™] Carboxylate-Modified Microspheres blue fluorescent [11] and the Fluoresbrite[®] BB Carboxylate Microspheres [12] are such examples, considering the spectral range of interest (near UV). When there is a spectrum overlapping, narrow optical filters are crucial for: (1) a correct excitation of the molecule, narrow optical filter centered at its excitation peak; (2) a correct extraction of the molecule emission with low interferences from the excitation light, narrow optical filter centered at its emission peak. As an example, for the Fluoresbrite[®] BB Carboxylate Microspheres, the optical filter to extract its fluorescent emission should have a high transmittance at 407 nm (peak emission) and the minimum transmittance at 360 nm (peak excitation). According to the simulations results, this compromise is better achieved with a $\text{MgF}_2/\text{TiO}_2$ based UV optical filter.

5. Conclusions

The fabrication and characterization of MgF₂ thin-films deposited on silicon substrates at different temperatures were presented. The MgF₂ thin-films were deposited by e-beam technique and optical characterized by ellipsometry. The measured refractive indices showed good agreement with the literature results, especially for higher substrates temperatures (200 °C and 300 °C). Also, the optical simulations compared the performance of near UV optical filters when using the materials combinations MgO/TiO₂ and MgF₂/TiO₂. With the first combination, optical filters with higher peak transmittance were obtained, especially at 350 nm. Finally, with the second combination, narrower optical filters were achieved, which can be important to specific applications on fluorescent optical sensors. Those specific applications include using narrow optical filters for extraction of the fluorescent emission of a molecule without interferences from the excitation light, when the excitation and emission spectra of the molecule overlap. Additionally, an MgF₂/TiO₂ UV optical filter was fabricated based on e-beam and reactive RF-sputtering. The optical filter transmittance was measured, with a peak transmittance of approximately 70 % and close to 400 nm. The optical filter total thickness was also measured by ellipsometer, with a value of 619.5 nm, which is close to the expected value (630 nm).

Acknowledgements

This work is supported by: FCT Strategic Project UID/EEA/04436/2019; Infrastructures Micro&NanoFabs@PT, reference NORTE-01-0145-FEDER-022090, POR Norte, Portugal 2020; SARSPEC Lda, V.N. Gaia-Portugal; project OCT-RAMAN, PTDC/FIS-OTI/28296/2017, operation code NORTE-01-0145-FEDER-028296; and project OpticalBrain, PTDC/CTM-REF/28406/2017, operation code NORTE-01-0145-FEDER-028406.

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