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Study of amorphous dielectric optical coatings deposited by plasma ion assisted electron beam evaporation for gravitational wave detectors

Carlos Garcia Nuñez,[1,*] Gavin Wallace,[2] Lewis Fleming,[1] Kieran Craig,[2] Shigeng Song, [1] Sam Ahmadzadeh,[1] Caspar Clark,[3] Simon Tait,[4] Iain Martin,[4] Stuart Reid,[2] Sheila Rowan,[4] Des Gibson[1]

[1]Institute of Thin Films, Sensors and Imaging, University of the West of Scotland, SUPA, Paisley PA1 2BE, Scotland, UK
[2]University of Strathclyde, SUPA Glasgow G1 1XQ, Scotland, UK
[3]Helia Photonics Ltd, Rosebank Park, Livingston West Lothian EH54 7EJ, Scotland, UK
[4]University of Glasgow, SUPA Glasgow G12 8QQ, Scotland, UK
[*]Corresponding author: carlos.garcianunez@uws.ac.uk

Abstract: This work presents the characterisation of optical and mechanical properties of Sc_2O_3, MgF_2, and HfO_2 thin films deposited by plasma ion assisted electron beam evaporation, evaluating their potential as mirror coatings in gravitational wave detectors.

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OCIS Codes: (310.1860) Deposition and fabrication; (310.6845) Thin film devices and applications; (310.3840) Materials and process characterization

1. Introduction

Optical interference coatings for gravitational wave detection have to meet a variety of specific requirements, such as low absorption, high reflectance and low mechanical losses, as well as high resistance against damage by power laser radiation, and long-term optical stability [1]. When utilised in gravitational wave detectors (GWDs), material processing for coatings on large and massive suspended mirrors leads to additional problems arising from the requirements of low stress, low density of defects and high uniformity. To that end, the improvement of more reliable and sophisticated thin-film deposition technologies is needed, allowing next generation of GWDs to augment their sensitivity towards the detection of interacting black holes, neutron stars, and even new unpredicted phenomena [2].

High reflecting (HR) coatings utilised in GWDs such as aLIGO and AdvVirgo consist of Bragg's reflectors alternating layers of low (SiO_2, n = 1.45) and high (TiO_2:Ta_2O_5, n = 2.1) refractive-index materials, and are typically deposited by ion-beam sputtering (IBS) technique. Reflectivities as high as R > 99.9995% can be achieved with mechanical losses of 10^{-4} (Ta_2O_5) to 10^{-5} (SiO_2) and optical absorption of less than 1 ppm [3]. In the meantime, optimisation of the coating design aiming to dilute the loss contribution of the high-index material has been explored through the deposition of new materials and utilisation of deposition techniques different to IBS.

This work presents a study of optical and mechanical properties of three potential material candidates to improve the performance of next generation of GWDs, including scandium oxide (Sc_2O_3, scandia), magnesium fluoride (MgF_2) and hafnium oxide (HfO_2, hafnia). These films were deposited by plasma ion assisted electron beam evaporation which is a technique that presents some advantages with respect to standard IBS, including higher deposition control (deposition rate, thickness and doping) and uniformity, as well as less Ar-impurities and material consumption [4].

2. Experimental Details

2.1 Deposition of Thin Films

Single layer coatings consisting of 396-nm thick Sc_2O_3, 345-nm thick MgF_2 and 238-nm thick HfO_2 were deposited using plasma ion assisted electron beam evaporation (Satis 1200 vacuum deposition system with a multi-pocket Temescal electron beam gun). This work utilizes a novel plasma source [4-5] based on inductive heating inner and outer surfaces of a lanthanum hexaboride high-efficiency thermionic emitter hollow cathode [7]. The deposition conditions were as follows: I_{AC} = 35A, V_{AC} = 145V, I_{EX} = 7A, O_2 = 50sccms, $Ar_{Cathode}$ = 6sccms, and Ar_{Anode} = 6sccms, using an induction power heater of 2.7kW, and e-beam evaporation rate of 3Å/s.

2.2 Characterisation of Thin Films

Morphological properties of the coatings, including thickness and surface roughness, were analysed by scanning electron microscopy (SEM) at 20 kV (S4100 cold FEG from Hitachi). The amorphous structure of the films was also analysed by X-ray diffraction analysis (XRD). Optical properties of coatings, including refractive index (n) and extinction coefficient (k) as well as, a confirmation of films thickness (d) have been obtained by spectrophotometry (UV-VIS-IR spectrophotometer from Aquila Instruments). For this, films were deposited on glass slides. Prior to the deposition, substrates were cleaned in an ultrasound bath (5 min acetone, followed by 5 min IPA) rinsed in DI water and dried under nitrogen flow. Optical density (OD) was measured as a function of the wavelength (λ) at experimental conditions, comprising incident angle of 0º, P polarisation, and tolerance of 0.5%. Then, MacLeod software was utilised to calculate *n*, *k* and *d* of each coating by fitting the experimental data to a single layer model. Stress measurements were carried out in a Wafer Geometry Gauge system (MX 203-6-33 from E+H Metrology GmbH). 4-inch Si(100) wafers were used as substrates. MX-NT and Wafer Studio software were used to carry out the measurements and for the visualization and data analysis, respectively.

Q-factor measurements were carried out to obtain the mechanical loss angle (ϕ) of each coating. For that, coatings were deposited on fused silica substrates (SiO_2, DSP, with flat, Corning 7980 from University Wafers) with a diameter of 76.2 mm, thickness of 511±1μm and a flat of 25 mm. Prior to the deposition, substrates were pre-annealed at 1000ºC for 4 hours. The ϕ was measured using a gentle nodal suspension (GeNS) placed inside a vacuum chamber [8], recording the decay of the excited resonant mode amplitude of the sample (ringdown) exhibiting damped harmonic motion, consisting of carrier and envelop signals, where the amplitude of the envelope is:

$$V(t) = V_0 \exp[-\pi f_0 \phi(f_0) t] \tag{1}$$

From (1), one can understand that the fitting of the ringdown measurement, will allow to directly calculate the mechanical loss angle as a function of the resonant mode frequency. The coating loss ($\phi_{coating}$ coating) is

$$\phi(\omega_0)_{coating} = \frac{E_s}{E_c}[\phi(\omega_0)_{coated} - \phi(\omega_0)_{substrate}] \tag{2}$$

Where E_s/E_c is the substrate/coating energy ratio estimated using finite elements analysis ANSYS package (Workbench 2021 Development R2) and utilised here to calculate $\phi_{coating}$ by Eq (2) for each of the resonant modes.

3. Results and Discussion

3.1 Stress Measurements

The stress value obtained in Sc_2O_3 films around 32.19 MPa, indicates a tensile stress which is orders of magnitudes lower than those reported for IBS Sc_2O_3 films (range of GPa) the latter caused by the high density of oxygen interstitials produced by that Ar based IBS method [9]. MgF_2 films evaporated in this work exhibit 150 MPa, being lower tensile stress values than those reported in the literature (550 MPa) [10] mainly due to the benefits of plasma ion assisted method to reduce the density of isolated crystalline aggregates drastically contribution to the partial disorder of the resulting lattice. Tensile stress values of 118 MPa obtained for HfO_2 are in good agreement with the 115 MPa obtained for 260-nm thick HfO_2 e-beam evaporated films reported in the literature, indicating that the layer could have reached a low level of porosity of around 9.4% [11].

3.2 Coating Mechanical Loss

The resulting mechanical loss angles measured in two different GeNS systems (UWS and UoS), including substrate ($\phi_{substrate}$), coated (ϕ_{coated}) and coating ($\phi_{coating}$) loss for all materials are summarized in Table 1. The table also includes the substrate/coating energy ratio (E_s/E_c) and the percentage difference ($\delta\phi_{coating}$) obtained between both systems.

Table 1. $\phi_{substrate}$, ϕ_{coated} and $\phi_{coating}$ of single layer coatings for various resonant modes.

Sample (*thickness*)	**Frequency (Hz)**	$\phi_{substrate}$ **($\times10^{-6}$)**	ϕ_{coated} **($\times10^{-6}$)**		E_s/E_c	$\phi_{coating}$ **($\times10^{-4}$)**		$\delta\phi_{coating}$ **(%)**
			UWS	**UoS**		**UWS**	**UoS**	
Sc_2O_3 (*396 nm*)	556	1.82 ± 0.02	4.26 ± 0.03	4.96 ± 0.06	169.41	4.1 ± 0.1	5.0 ± 0.06	20
	1277	2.15 ± 0.04	6.39 ± 0.06	6.52 ± 0.02	170.39	7.22 ± 0.06	6.27 ± 0.06	14
	3387	1.83 ± 0.05	6.89 ± 0.04	7.23 ± 0.06	163.60	8.3 ± 0.1	7.7 ± 0.1	8
MgF_2 (*345 nm*)	549	1.73 ± 0.03	8.23 ± 0.03	7.5 ± 0.4	275.65	17.92 ± 0.07	16 ± 1	11
	1266	1.41 ± 0.03	9.35 ± 0.03	9.3 ± 0.7	280.79	22.29 ± 0.06	22 ± 2	1
HfO_2 (*238 nm*)	513	1.01 ± 0.05	7.13 ± 0.04	4.2 ± 0.2	314.29	19.2 ± 0.3	10.3 ± 0.7	60
	1189	1.95 ± 0.04	4.25 ± 0.01	5.2 ± 0.2	308.68	7.1 ± 0.2	9.92 ± 0.8	33
	2076	2.72 ± 0.03	6.25 ± 0.05	7.0 ± 0.6	304.85	10.8 ± 0.1	13 ± 2	18

The $\phi_{coating}$ results are in good agreement, exhibiting a low discrepancy percent between GeNS systems (Table 1). The low $\phi_{coating}$ values of 4.1-5.0×10^{-4} obtained in Sc_2O_3 films make the evaporation method used here to be promising a technique for h-index Sc_2O_3 based coatings. This $\phi_{coating}$ as well as those obtained for the other two materials could be further reduced by the application of thermal annealing below the crystallisation temperature [1], and the optimisation of the evaporation conditions (mainly through the increase of the ion energy).

3.3 Optical Properties

T and R spectra (Figure 1 (a)) have been used to calculate refractive index (*n*) of coatings as a function of the wavelength using the Sellmeier expression (Figure 1 (b)). Results obtained at λ = 1064 nm show *n* of 1.75 (Sc_2O_3), 1.33 (MgF_2) and 1.89 (HfO_2). The extinction coefficient in the evaporated films is below the detection limit of the spectrophotometer instrument over the wavelength range of inspection. Further optimisation of *n* needs to be performed through the increase acceleration voltage (V_{AC}) and ion energy during the evaporation process [4, 12].

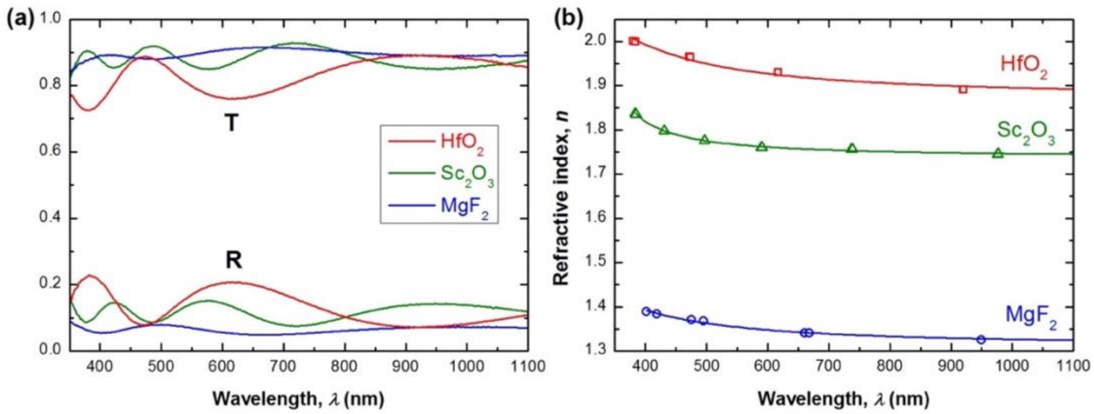

Figure 1. (a) T and R, and (b) *n* of Sc_2O_3, MgF_2 and HfO_2 thin films vs wavelength.

4. Conclusions

Sc_2O_3, MgF_2 and HfO_2 films were deposited by ion plasma assisted electron beam evaporation, showing tensile stress of 32, 115 and 150 MPa, being drastically lower to those coatings deposited by IBS. SEM confirmed the lack of crystalline aggregates and other defects which are the main responsible in IBS coatings, increasing the film stress. Optical characterisation of the films demonstrates high *n* of 1.75 and 1.89 for Sc_2O_3 and HfO_2 and low *n* of 1.33 for MgF_2 comparable to those reported in the literature for similar coatings deposited by IBS. The characterisation of ϕ_{coating} in those films resulted in values in the range of 10^{-4} (Sc_2O_3) and 10^{-3} (MgF_2 and HfO_2) without the application of any thermal treatment. These results are promising and the investigation of the ϕ_{coating} after annealing treatments are expected to reduce further the coating loss down to the range of 10^{-4} - 10^{-5} as well as the coating stress values. A further optimisation of the optical and mechanical properties will be possible through the enhancement of the ion energy by increasing the acceleration voltage in the plasma ion assisted electron beam evaporation.

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