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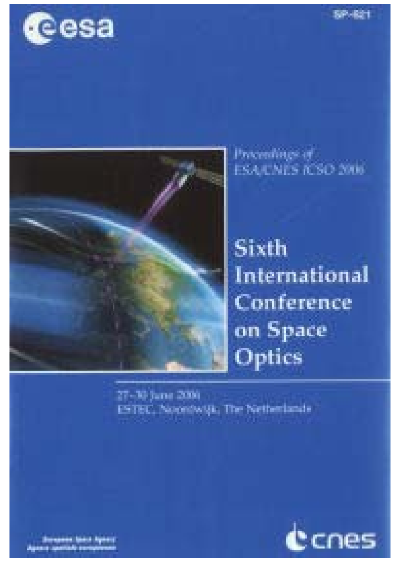

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DEVELOPMENT OF A REAL-TIME REFLECTANCE AND TRANSMITTANCE MONITORING SYSTEM FOR THE MANUFACTURING OF METAL-DIELECTRIC LIGHT ABSORBERS

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ABSTRACT

Metal-dielectric light absorbers are of great interest for suppressing stray light in optical systems. Such coatings can give an absorption level greater than 99.9 % over a broad spectral range provided that the complex refractive index of metallic films is accurately known. For this purpose we developed a new real-time monitoring system that allows to measure in situ both reflectance and transmittance of the coating during manufacturing in the deposition chamber. This paper describes the system design and its characteristics and gives some preliminary results concerning metallic thin film characterizations.

1. INTRODUCTION

The classical solution for suppressing stray light in optical systems consists in using black paints. As an alternative solution, metal-dielectric light absorbers offer two major advantages. First, the absorption level is much higher and can reach 99.9 %, even on a broad spectral range such as the visible range. Second, the total thickness of the coating is less than one micron while the painting thickness is about one hundred. However, this solution is much more expensive, which restricts its use to specific cases only. These coatings are formed with a few alternated metallic and dielectric layers, best results being obtained combining a low reflecting metal with a low index dielectric material [1]. Each layer has a precise thickness resulting in an interference pattern that forces the light to be absorbed in the metallic ones. Most often, the first metallic layer is completely opaque. As a result, the transmittance of the coating is cancelled and the nature of the substrate does not influence the optical properties of the final component. Next metallic layers are semi transparent with generally decreasing thicknesses (typically from 100 nm to 10 nm) so that the light can easily penetrate in the stack from the air side before being trapped. However, this kind of coating behaves as an antireflection coating, and as usual for this kind of coating, errors concerning the coating design result in an increase of the residual reflectance. This explains why the layer thicknesses must be precisely defined, which requires at first an accurate knowledge of the refractive indices of used materials. This point is not a difficulty for the dielectric material since its refractive index can be easily and classically extracted from the modulation of the reflectance and transmittance spectral curves of a single film. But this is not the case for metallic films since there is no spectral modulation anymore. Also notice that the characterization of a metallic material requires extracting two unknown values, its refractive index and its extinction coefficient. Moreover the coating design contains very thin metallic layers corresponding to the early beginning of the deposition process when the film structure and packing density are rapidly changing. As a result, the film optical constants must be determined as a function of the thickness and cannot be considered as homogeneous. To overcome all these difficulties, we developed an index determination method based on two measurement steps [2]. The metallic film is deposited at first and we measure both transmission and reflection. Then this film is overcoated with a known dielectric material and spectral responses are again measured. It is then possible from these four measurements to extract all the metallic film parameters. However, this method suffers from several drawbacks. First, we need to bring back the sample at the ambient atmosphere for measurements after deposition of the metallic film and this can induce a partial oxidation at the air interface that will false measurements. Second, its remains difficult to investigate the non homogeneous structure of the metallic film without manufacturing numerous samples, especially very thin metallic layers for which the oxidation phenomena is much more critical. This was not the case for our first studies on the subject since the coatings were manufactured at the time using a fluoride dielectric material. However, in order to obtain hard coatings, with climatic and mechanical characteristics that are compliant with space requirements, we had to use a high energy deposition process [3, 4] and this kind of deposition technique is not optimum for fluoride but oxide materials. In our case, hafnium is now used as metallic films, silica as

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dielectric films, and an argon/oxygen plasma is created in the deposition chamber to generate high energy assistance. Obviously, only argon is used for the deposition of metallic films but oxygen is also mandatory for silica deposition which results in a partial oxidation of Hf/SiO_2 interfaces that can with difficulty be taken into account.
At present time, these difficulties are the major limiting factors to improve the optical performances of the coatings, especially for broadband absorbers. As a consequence, we decided to develop an in situ real-time measurement system to perform during deposition a broadband reflectance and transmittance characterisation of the coating. This will prevent extra oxidation occurring during measurement at ambient atmosphere and will give information on the oxidation that takes place at the beginning of silica deposition. In addition, this measurement system can be used efficiently as a monitoring system during the manufacturing of any type of coating including light absorbers to automatically stop the deposition of a layer.
The next section will describe the system and section 3 will give some preliminary results concerning metallic films index determination.

2. DESCRIPTION OF THE OPTICAL MONITORING SYSTEM

Optical monitoring systems are of great interest to determine the best moment to stop the deposition of a layer. In particular they can offer some self error compensation capabilities leading systematically to the right final spectral profile, while quartz crystal monitoring cannot [5]. As a consequence, all deposition chambers in the laboratory are equiped with such a system, measuring transmission for a single wavelength. Coming from a white light source located on the top of the machine, a collimated beam is transmitted through the sample before being analysed with a monochromator. It is therefore possible to follow the evolution of the transmittance during deposition, at selected wavelength. In order to avoid uniformity difficulties, the sample holder is rotating as usual and the optical monitoring is not performed on the rotation axis but on a given diameter. By this way all the samples located on this diameter present the same spectral profile since there are naturally identically coated [6]. This requires that the monitoring system be synchronised with the substrate holder rotation. This kind of system is not of great help for the manufacturing of light absorbers for two main reasons. Concerning metallic film characterization, the transmittance measurement alone is not enough. Indeed, we need to measure the reflectance also in order to estimate the absorption level that will allow us to evaluate the thickness and extinction coefficient of the film. Concerning now the manufacturing of light absorbers, the first layer of these coatings is completely opaque which excludes the use of a transmittance monitoring system. To improve these two points, we decided to develop a system measuring simultaneously both transmission and reflection. Moreover, in order to immediately obtain the spectral response, the monochromator has been replaced by array spectrometers, one for each channel. Fig. 1 gives a schematic representation of the whole system.

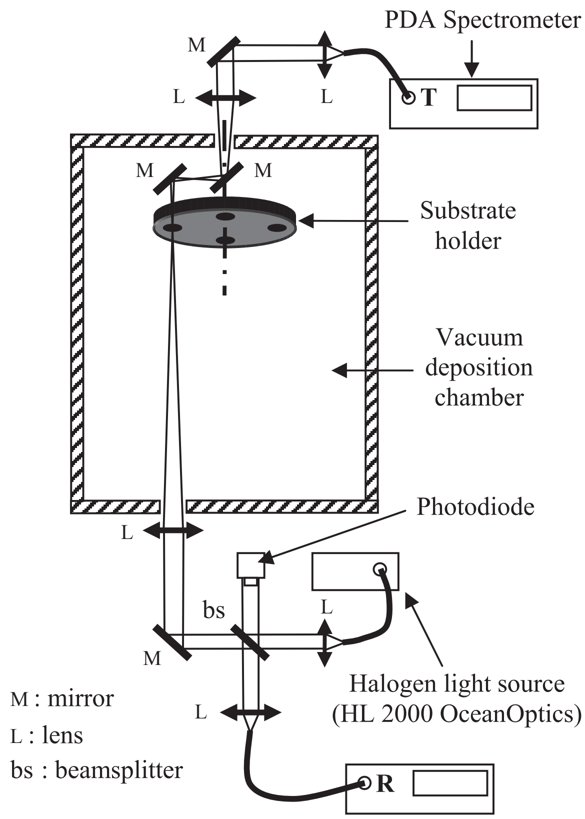

Fig. 1. Optical broadband monitoring system

For more flexibility, the halogen light source and the spectrometers are connected to the system by mean of optical fibres. Below the deposition chamber, the entrance fibre, which diameter is 200 µm, is imaged on the sample with a magnification ratio equal to 40, corresponding to an analysis beam diameter of 8 mm. A broadband beamsplitter in the 400-1000 nm range permits to collect the reflected light on the sample through an optical fibre connected to one of the spectrometers. Notice that the beamsplitter also permits to collect a reference beam and enables to balance fluctuations of the light source. This reference beam is

measured by a silicon photodiode without any spectral analysis.
Similarly, the beam transmitted by the sample is collected above the deposition chamber and focused on an optical fibre connected to the second spectrometer. Notice that since the window transmitting this beam out of the chamber is centred on the rotation axis of the substrate holder we had to use a two-mirror periscopic system located above the substrate holder.
At last, all the optical images are performed with two-lens telecentric systems so that the angular distribution of the light is preserved and allows an optimum efficiency between the entrance and exit fibres.

Concerning the choice of the spectrometers, the spectral range should at least extend from 400 to 1000 nm with a spectral resolution of about 3 nm. However, the most selective parameter for us was the Signal to Noise Ratio (SNR), at least 1000, for a maximum integration time equal to 50 ms corresponding to the rotation of the substrate across the monitoring beam. These specifications led us to select a Photodiode Array (PDA) array instead of a CCD array and we finally selected a ZEISS MCS 501 UV-NIR spectrometer [7]. After a set of measurement tests we can estimate the long term stability of the whole system at 0.1% in reflectance and 0.04% in transmittance for a typical duration of 2 hours corresponding to classic deposition runs. The short time stability (about ten minutes) is characterized by a SNR of 3000 concerning the transmittance channel.
Due to the rotation of the monitoring sample, measurements are performed every two seconds. In order to calculate the transmittance and reflectance of the coating during its manufacturing, all measurements are normalized with the corresponding signals measured on a bare substrate before starting the deposition.
Comparative measurements were performed with a Perkin-Elmer Lambda18 spectrophotometer. Results were in good agreement, the discrepancy being less than 0.5% regarding transmittance at any wavelength.
The software we developed to perform data acquisition permits to store all measured spectra for post processing analysis, index characterisation for example. It also permits real-time signal processing to determine the moment to stop layer deposition, based on any monochromatic, polychromatic or broadband reflectance or transmittance criteria.
This monitoring system has been first tested through the manufacturing of beamsplitters and edge filters. As expected, the monitoring capabilities are improved compared to those of the previous system [8].

3. APPLICATION TO METALLIC FILM INDEX CHARACTERISATION

The index determination method we developed for metallic films requires the deposition of a dielectric layer over the metallic one, spectral profiles being measured after the deposition of each layer. As a general rule, the index determination is more easy and accurate if a high index dielectric material is used.
In our case, since we use hafnium as metallic material, it is very convenient to use hafnium dioxide for the dielectric one since this only requires to introduce oxygen into the chamber during deposition. As the index of the dielectric material must be known to extract the optical constants of the metallic film, a single HfO_2 layer has been previously characterized alone, with an average refractive index over the visible range of about 2.11. At present time, we have only characterized an opaque metallic film and Fig. 2 gives the reflectance profiles measured in situ after the deposition of each material. As one can see, the metallic film induces no modulation of the reflectance, contrarily to the dielectric one. Notice the existance of intersection points between these two curves. Half of these points corresponds to wavelengths for which the dielectric layer optical thickness is multiple of the half wavelength, and thus do not modify the reflectance of the metallic film.

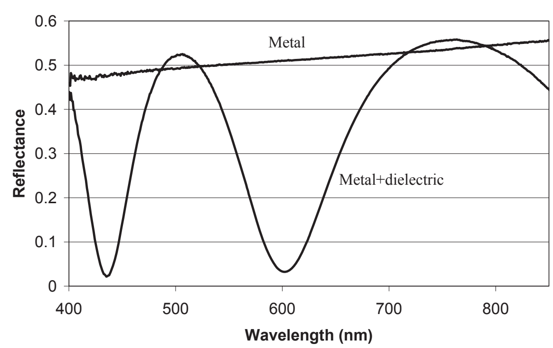

Fig. 2. Reflectance curves measured in situ for an opaque Hf layer over coated with a HfO_2 dielectric layer

From these curves, assuming that the index of the dielectric layer is known accurately, and its thickness approximately, we can extract the refractive index and extinction coefficient of the metallic film, as shown in Fig. 3. For this result, the metallic layer is assumed to be homogeneous. Considering that the film inhomogeneity only concerns the early beginning of the deposition, this assumption is correct in the case of an opaque film, relatively thick, since the material crossed by the light can be overall assumed homogeneous.

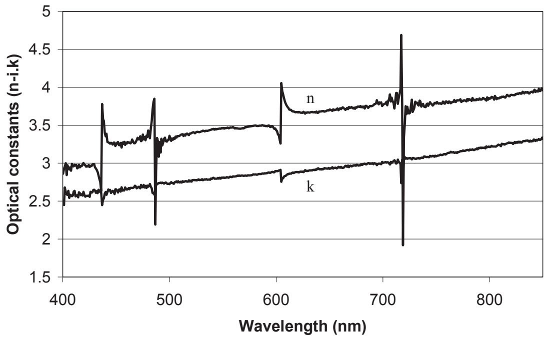

Fig. 3. Refractive index and extinction coefficient extracted from spectral curves given in Fig. 2

As one can see, the accuracy of the index determination is not constant over the whole spectrum. For example, the problem cannot be solved for all wavelengths for which the dielectric layer is half wave. This explains the sharp peaks that appear in these curves. Using these optical constants to calculate the reflectance curves, we obtain a perfect agreement with measured spectra.
Obviously these results have to be corrected with regular functions based on spectral regions where the determination is the most accurate.
However, the monitoring system not only gives us the spectral profiles at the end of the deposition but during the whole deposition time. As an example, Fig. 4 gives the evolution of the reflectance and transmittance of the sample measured during the deposition of the metallic layer at wavelength 600 nm.

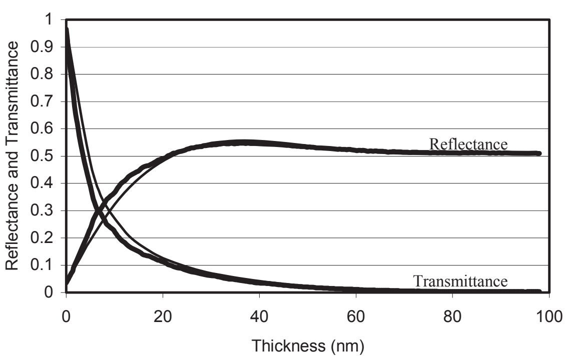

Fig. 4. Reflectance and transmittance evolution during the deposition of a Hf opaque layer @ 600 nm
- thick lines : in situ measurements
- thin lines : calculated after index determination

The corresponding calculated curves are plotted on the same graph using the optical constants determined for this wavelength. As one can see, the agreement is correct except at the beginning of the deposition, which corresponds to the non homogeneous part of the film.
Due to this non homogeneity, the index determination is more difficult for semi transparent films. Moreover, a modification of optical properties can occurs at the entrance of oxygen inside the deposition chamber, just after deposition of a thin metallic layer and before deposition of a dielectric layer. To illustrate this point, Fig. 5 shows the evolution of measured optical properties of a light absorber under manufacturing due to the entrance of oxygen after the deposition of a thin hafnium layer.

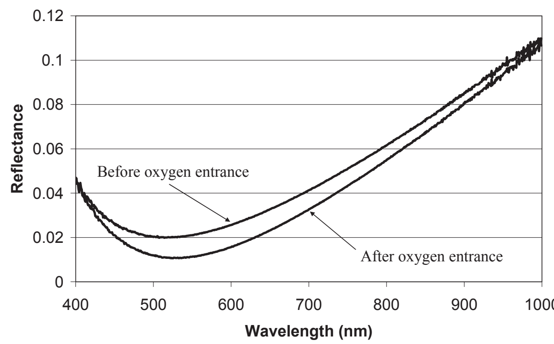

Fig. 5. Typical evolution of the reflectance due to the entrance of oxygen after the deposition of a 8 nm thick Hf layer

Such a phenomenon was suspected. Indeed, as in the case of the opaque metallic layer, the spectral profiles before and after the deposition of the dielectric layer should theoretically exhibit intersection points, but this was no more the case for very thin semi-transparent metallic films, due to the production of a transition layer between the two materials. With this new monitoring system, we are now able to measure this phenomenon and taking it into account for stack modelling.

4. CONCLUSION

Among solutions dedicated to suppress stray light, metal-dielectric light absorbers are one of the most efficient regarding optical properties. However, in order to reach the highest performances, a precise index characterization is mandatory for thin metallic films. It requires to take into account both the film thickness and its partial oxidation, especially when coatings are manufactured using an oxide material. For this purpose, we developed an in situ reflectance and transmittance measurement system. Thanks to the spectral data system collected during deposition, we are now able to perform more accurately index characterizations, especially for semi-transparent films that are not completely homogeneous. In addition, metallic partial oxidation phenomenon at the beginning of the dielectric material deposition can be measured and considered for the coating design. At present time, we still need to improve the index characterization software we developed to take advantage of these new data given by real-time measurements. At last, the final goal is to manufacture a broadband absorber that covers the whole visible range with an absorption level higher that 99.9 %.

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