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ABSORBING COATING IN MAGNETRON SPUTTERING FOR PARASITIC LIGHT REDUCTION

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I. INTRODUCTION

Stray light is an important issue in optical systems and may be responsible for huge limitation of final performances. Use of black coated surfaces is known to be an efficient means to reduce such parasitic light sources and various solutions exist [1,2,3] that can be applied to mechanical surfaces such as black paints or black anodization; these coatings are relatively thick and to produce thin, black baffle edges, a thin layer technology is thus needed. In this paper, we show how thin film multilayer coatings can be a solution to answer this problematic as it is possible to design accurate spectral response that present a very low level of reflectance with a zero value of transmittance.

After a detailed description of the design steps, we will focus on the manufacture of such sophisticated metal-dielectric multilayer stacks using magnetron sputtering technique; in particular, we will show that control of refractive index of very thin metallic layers is an asset to achieve accurate performances and how in situ optical broadband monitoring allows excellent reproducibility of production processes even for few nanometers-thick layers required in metal-dielectric absorbers.

Spectral and angular measurements of different coating designs will be given on various types of substrates glass or metallic, the later either sanded or machined. In this case, since the coating is thin, the scattering behavior depends on the substrate roughness. Environmental tests and spectral characterizations led on qualification samples are also presented showing the stability of the performances in severe conditions compatible with space environment.

In particular, coatings developed in the frame of research and development activity for the French space agency (Centre National d'Etudes Spatiales) for broadband spectral and incidence range will illustrate this study. At last, experimental results will be given for coatings directly deposited on baffles vanes samples with extreme shapes of edges.

II. DETAILED DESCRIPTION OF THE DESIGN STEPS

Absorbing functions can be designed with multilayers using alternative metallic and dielectric layers. In such case, light is absorbed inside the multilayer stack with some layers that have only few nanometers.

Among the large variety of metals that can be selected to constitute the multilayer, it has already been demonstrated that low reflective metals are more appropriate as they exhibit a real part of refractive index higher than the incident medium, which makes multilayer design easier [2]. As an example we give in the following graph (Fig.1), refractive indices (real and imaginary parts) from bibliographic tables of several metals deposited in thin films [4].

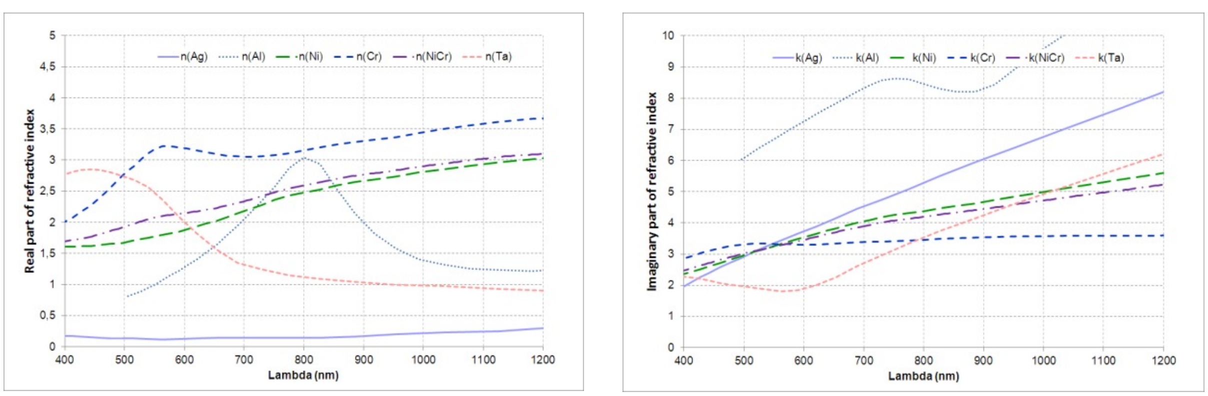

Fig.1. Refractive indices of metals as a function of wavelength

The first step of design consists in selecting the materials that can be used to reach the requirements. As we have at our disposal various options, our selection will be based on the easiness to produce them on an industrial coating chamber and to match them inside a multilayer without any impact on the others.

A description of the design steps is given below (Fig.2) where we can see that real values of refractive index produced with the chosen technique are injected in the design software in order to master the production with a high level of reliability.

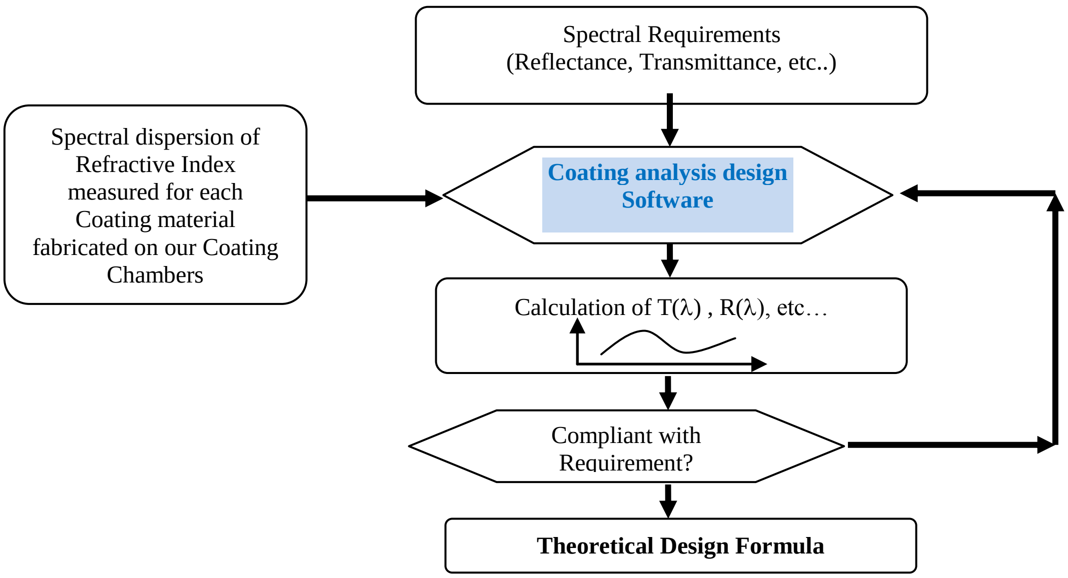

Fig.2. Design steps involving real materials data

For this purpose, it is necessary to master the refractive index of each material even for very thin metallic layers. In particular, it has been shown [2, 5] that refractive index for metallic layer is dependent on the thickness of the layer. So, we first characterize refractive index of metallic layers for several thicknesses in a representative range of the thicknesses involved in the coating.

On the following graphs, we give the theoretical response as a function of incidence of 2 different multilayer coatings adapted for different range of wavelength (visible and near infrared).

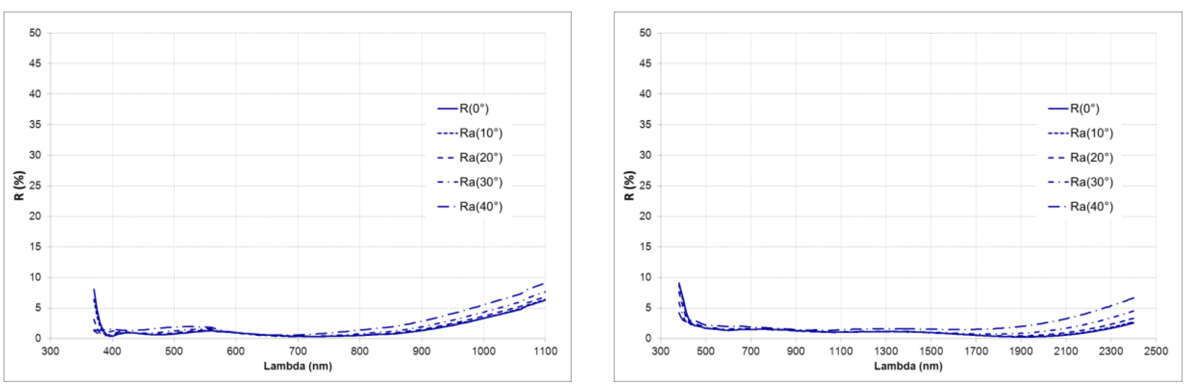

Fig.3. Metal-dielectric absorbing coatings solutions

III. REALIZATION OF METAL-DIELECTRIC MULTILAYER STACKS

A. Coating deposition technique

As presented before, the absorbers designs involve very thin layers in the multilayer and it may be an issue to monitor such coating. In fact, we use magnetron sputtering technique as it is known to be very stable and reproducible; moreover, the packing density of the layers that are deposited with this technique is very high and close to bulk material.

For more than 20 years, CILAS has specialized in thin film optical coatings and masters various coating technologies, such as electron gun evaporation with ion assisted deposition, dual ion beam sputtering or magnetron sputtering. Such technologies are particularly well suited to severe environment applications as they allow producing dense layers with packing density close to bulk material. In particular, we are equipped with a very large magnetron sputtering machine able to coat components up to 2 meters by 2 meters surfaces with improved mechanical performances and a high level of uniformity [6, 7]. Such coating machine is described in a paper given in the same session of the present conference [8].

Thickness monitoring is a key to obtaining spectral predefined template. Among the different methods that are commonly used for thin film monitoring, optical monitoring based on the direct in-situ measurement of the optical properties is particularly interesting as it allows determining optical thickness of the sample being coated. With the help of the measurements of in-situ transmittance and/or reflectance, we can extract in real time useful characteristics of the layer under construction such as thickness and complex refractive index including extinction coefficient. Such optical monitoring coupled with process automation is the pledge of the compliance of coating fabrication.

In particular, we have developed a broadband optical monitoring which allows recording a wide spectral range in real time and characterizing in-situ material parameters [9]. With a re-optimization of the design that can be made in real-time taking into account the previous layers, we can thus achieve optical performances with a good agreement with theory.

B. *Broadband optical monitoring*

Our system is coupled to the coating machine with optical fibers more than 20-m long and makes it possible to measure in-situ optical properties with a 10^4 S/N ratio over [280 nm; 2 200 nm] spectral range with a 3 nm spectral resolution in visible and 16 nm in infrared. With an interface inside the machine hard automation, the layer deposition can be stopped automatically when the following numerical criterion between spectral measurement (T_{ex}) and theoretical end-of-layer performances (T_{th}) is minimal:

$$\chi^2(e) = \sum_{n=1}^{N} \frac{\left[T_{ex}(e,\lambda_n) - T_{th}(\lambda_n)\right]^2}{N}$$

Fig.4. In-situ broadband optical monitoring connected to 14 m^3 PACA2M coating chamber

As an example, we give on the following graph (Fig.5) spectral profiles of transmittance for different layers of the coating under construction measured in-situ over a broadband range at the end of the layer. From these measurements, we can extract the thickness of the layers that has been deposited.

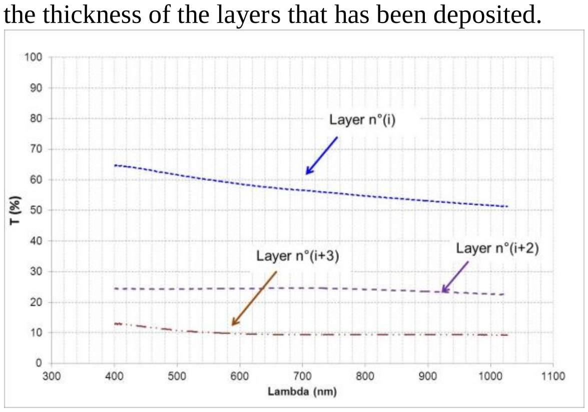

Fig.5. In-situ measurements of transmittance under vacuum over a broad spectral range

With such optical monitoring, it is thus possible to accurately monitor such coating with high reproducibility.

IV. ENVIRONMENTAL QUALIFICATION TESTS

Several samples have been coated simultaneously with absorbing function, polished glass samples and aluminum 6061 T6 samples with two types of surface finishing. First type Al samples was classical machining with a 0.5-0.7µm roughness, whereas type of second surface finishing of Al samples have been polished to reach a roughness of about 50nm.

The following tests have been done on qualification samples in order to check the stability of the performances in severe conditions compatible with space environment:

- Thermal cycling under atmospheric pressure, 20 cycles between -40°C and 110°C, 1°C/min slopes, and 15 min in min and max stages (Fig. 6), followed by adhesion test according ISO 9211-4 and severity level 2.
- Cleaning test on the coated Al 6061 T6 samples using a wash bottle which allows rinsing all the surface with a narrow stream of acetone, followed by dry nitrogen blowing and by adhesion test according ISO 9211-4 and severity level 2

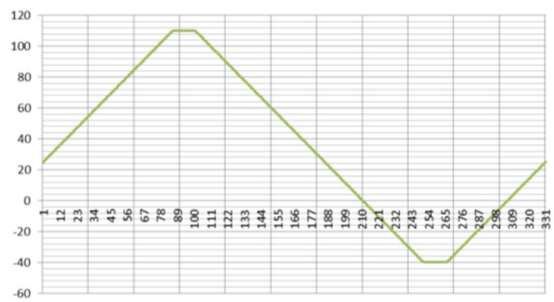

Fig. 6: Thermal cycling at atmospheric pressure

Cosmetic inspection after tests and spectral measurements in reflection (Fig. 7 and Fig. 8) have been done before and after the tests, as presented on the following figures where we can notice that there is no evolution after test.

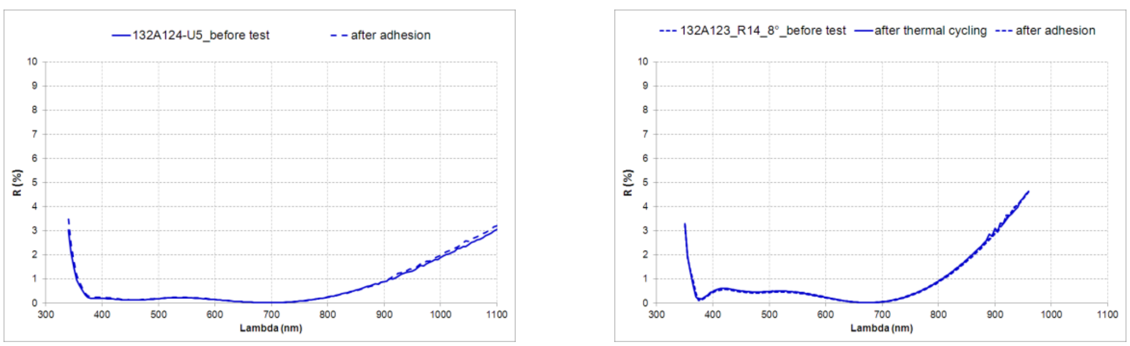

Fig. 7: Spectral measurements of reflectance at 8° of incidence before and after qualification tests

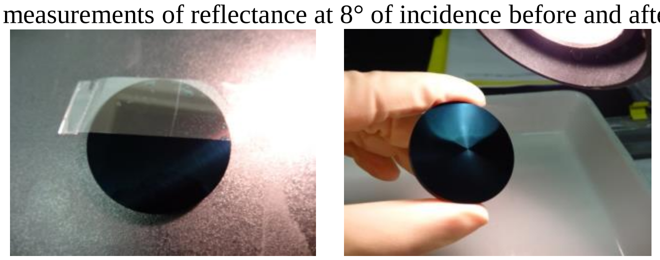

Fig. 8: Coated samples during adhesion and cleaning tests

Both qualification tests sequences were successful on the two types of surface finishing as no degradation of the surface cosmetic properties after adhesion test and no change on the spectral measurements have been observed.

V. SCATTERING CHARACTERIZATION

At last, scattering behavior of such multilayers solution has been numerically studied and characterizations have been performed on representative components. The metrological study is performed with SALSA (Spectral and Angular Light Scattering Apparatus), the new scatterometer developed by the Institut Fresnel [10, 11, 12].

This set-up (Fig. 9) allows the recording of angularly and spectrally resolved scattering patterns on the spectral range [420-1100 nm] with an effective detection limit close to 10^{-8} str^{-1} (Rayleigh diffraction of air particles physical limit) and so it is perfectly suited to the broadband characterization of low scattering and absorbing coatings.

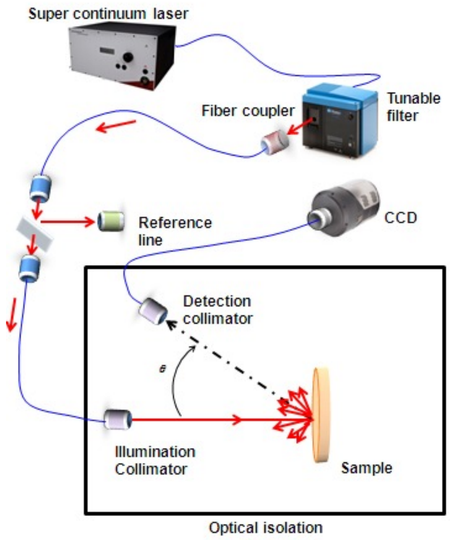

Fig. 9: SALSA (Spectral and Angular Light Scattering Apparatus), at Institut Fresnel

The source used is a super-continuum source emitting in the range [450 nm- 2 µm] with a 6W total power available. The laser light is then filtered by a tunable filter leading to a wavelength adjustable on the [450-1000 nm] range with an output spectral width of 2 nm.

The outgoing beam is coupled into a 50 µm diameter multimode optical fiber, then in a 100 µm diameter fiber after passing through adjustable densities and sampling a reference signal. The illumination collimator has an angle of incidence which may be arbitrarily chosen. The detection collimator is positioned on a motorized arm which rotates in the plane of incidence around the illuminated surface of the sample.

The set of θ angles which can be addressed by measurement, is comprised between [5°- 185°], the 0° position corresponding to the incident beam. For each scattering angle θ and each illumination wavelength λ_C, the scattered light is collected and then injected into an optical fiber through an achromatic collimator symmetrical to that used for lighting. The fiber output is then imaged onto a very high sensitivity CCD array.

All measurements are given with reference to the calibration sample measurement and the detection limit of the instrument. The level of scattering is given in terms of BRDF as a function of scattering angle θ which is defined with respect to the normal to the sample as shown in Fig. 10.

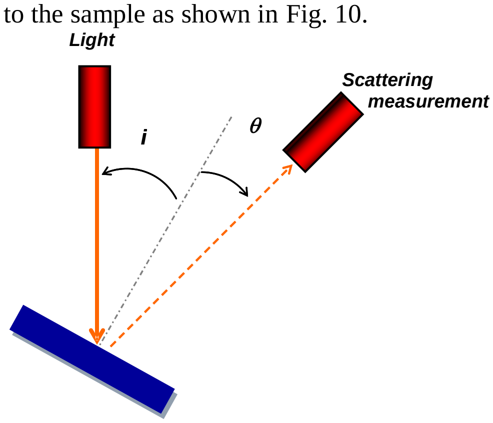

Fig. 10: Definition of angles of incidence (i) and scattering (θ)

Calibration of the system is done by measuring a standard calibration sample. This sample is a component whose angle resolved scattering pattern in the plane of incidence follows a Lambertian law of the form:

$$I(\theta,\lambda)=\frac{\rho(\lambda)}{\pi}\cos\theta$$

where $\rho(\lambda)=1-A(\lambda)$ is the albedo of the sample at the considered wavelength, and $A(\lambda)$ the absorption.

The measurement of a standard Spectralon reference sample (albedo of 99% ± 1% over [250 nm-2500 nm]) allows the validation of proper operation of the scatterometer by comparison of measurements to theoretical response of the component.

With this set-up, we have characterized 2 samples with different roughnesses and we give on the following graphs (Fig. 11 and Fig. 12), the measurements for different angles of incidence.

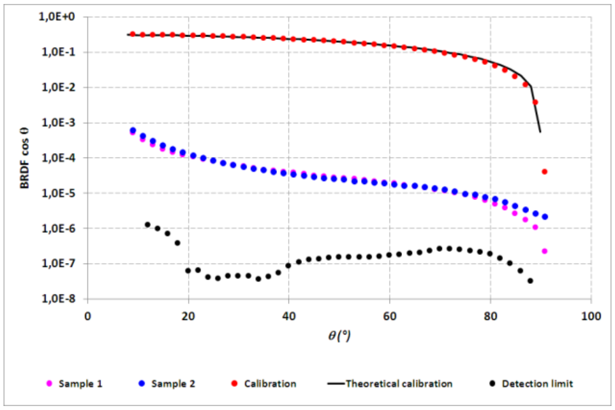

Fig. 11: Scattering measurement of 2 samples with different roughnesses, λ = 600 nm at 0° of incidence

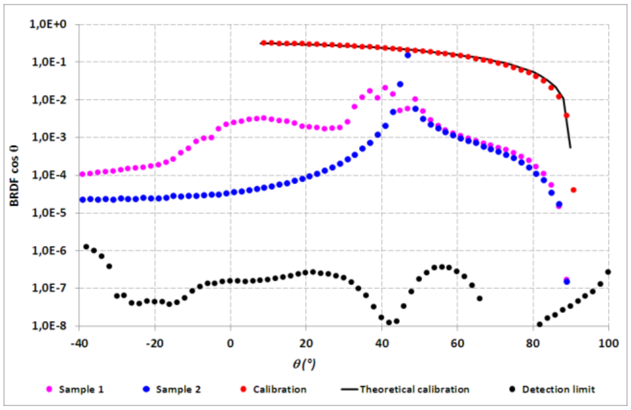

Fig. 12: Scattering measurement of 2 samples with different roughnesses, λ = 600 nm at 50° of incidence

We can see the low level of scattering obtained, with a good agreement with calculation as presented on the following graph where we have superimposed theoretical calculation on the measurement at 0° of incidence.

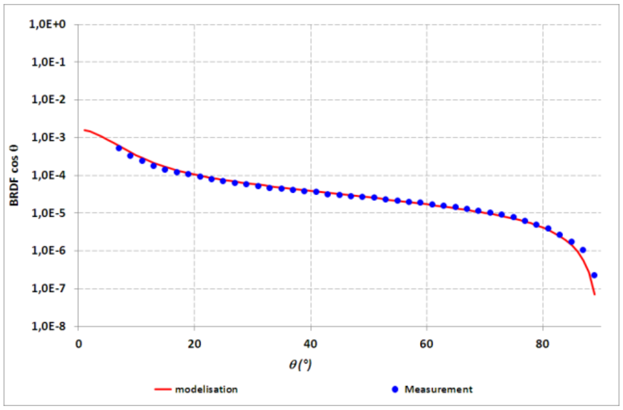

Fig. 13: BRDF measured (black dots) compared with modelization (red curve) of scattering losses of absorbing optical coating – Unpolarized illumination light at 650 nm wavelength under normal incidence

VI. CONCLUSION

In this paper, we have presented absorbing multilayer coatings that have been developed in the frame of research and development activity for the French space agency (Centre National d'Etudes Spatiales); we have seen that broadband optical monitoring is particularly interesting for the production of such metal-dielectric functions as it allows determination of refractive index.

Environmental tests led on various types of substrates (glass or metallic) have been achieved and spectral characterizations done at the different steps of tests have shown the stability of the performances in severe conditions compatible with space environment. Capability of cleaning on coated Al samples has been demonstrated. Finally, scattering characterizations have been performed and show that, even in the case of high roughnesses components, very low scattering levels can be reached by the optimization of absorbing coating formula.

The authors thank Sodern for supplying the Aluminum 6061 T6 samples, representative of vanes baffles in the frame of research and development activity for the French space agency (CNES).

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