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### ENGINEERING TOOL FOR THE QUALIFICATION OF OPTICAL COATINGS

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#### ABSTRACT

For the needs of the European Space Agency, SESO is developing in cooperation with the Institut Fresnel an Engineering Tool for the Qualification of Optical Coatings. The goal is to develop a standard methodology for testing the behaviour and stability of optical coatings during the air to vacuum transition.

The Engineering Tool is indeed designed to achieve in vacuum reflectance and transmittance measurements between 600 and 1700 nm.

It is also designed to evaluate during the vacuum cycle partially the nature of the outgassing elements, using mass spectrometry.

We will present in our paper the concept of this equipment and the associated test method.

The preliminary characterizations will be done in June 2006 on reflective coatings, one anti reflective coating and dichroic filters.

#### 1. CONCEPT OF THE EQUIPMENT

#### 1.1 Scope

It is well known that during the first vacuum to air change, which occurs on completion of a vacuum deposited coating, the metallic and oxide optical layers can trap or adsorb a certain amount of humidity, depending on their density and stoichiometry. Any water adsorbed into the layers will create a modification of the effective refractive index and thus of the optical properties of the coating. If the coating is subject to new air to vacuum transition(s), the water adsorbed by the layers will be partially desorbed, which can in turn lead to:

- further modifications of the optical performance properties (the refractive indices of the layers will change again, causing a potential shift in the wavelength centering, the transmittance, the

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reflectance and the optical absorption of the coating as a whole),

• outgassing of constitutive elements from the coating.

#### 1.2 Specification for the equipment

In order to characterize modifications of the optical properties and performance of coatings induced by air to vacuum transitions, the Engineering Tool is able to:

- realize a vacuum of 10<sup>-5</sup> Torr (1.3 mPa),
- realize in-situ transmittance measurements,
- realize in-situ reflectance measurements at 7° and 45° angles of incidence,
- measure during the vacuum cycle partially the nature of the outgassing elements, using mass spectrometry.

The device includes a heating circuit to increase the temperature of the sample by up to 50°C in order to accelerate the outgassing process.

It also includes a cooling device using liquid nitrogen in order to characterize the optical coating at low stabilized temperature. This is one of the specificity of the Engineering Tool.

The equipment is able to realize transmittance and reflectance measurements for different states of polarization.

It is also possible to perform a visual inspection of the sample surface during the vacuum cycle.

The state of the art of the air to vacuum transition on optical coating mainly concerns narrowband filters. The qualification of optical coatings with the Engineering Tool will be performed on:

- a wide spectral range of 600 nm to 1700 nm, to cope with the general objectives of space applications,

- other types of coatings (reflective coatings, one anti reflective coating and dichroic filters).

#### 2. TEST METHOD

#### 2.1 Description of the Engineering Tool

The main part of the Engineering Tool is the vacuum chamber which has been designed in order to minimize the leak rate and to achieve a vacuum of  $10^{-5}$  Torr. The chamber is about 200 mm diameter and is equipped with a vacuum pump and a vacuum gauge for measuring the static vacuum.

The chamber is equipped with optical windows of one and two inches diameter in order to allow reflectance and transmittance measurements and also the observation of the sample during the test ("Fig. 1").



Fig. 1. The vacuum chamber.

The windows are uncoated so that the only coating exposed to the air to vacuum transition is the coating of the sample which is in the vacuum chamber. In addition, this ensures that the windows have a well known and stable transmittance.

In order to avoid possible problems induced by residual parasitic images, the input and output optical windows are slightly tilted.

The design of the equipment for specular reflectance and transmittance measurements is mainly based on the goniometric method.

The sample is put inside the chamber which is set on the central stage of a goniometer. The front (incident) surface of the sample is co-planar with the rotation axis of the goniometer.

The illumination source device is attached to the fixed arm of the goniometer, and the detection device is attached to the rotating arm of the goniometer ("Fig. 2").



## Fig. 2. Schematic representation of the goniometric method.

The recording of the base-line of the set-up is performed in transmission without any sample and without use of any kind of standard reflector for calibration purposes.

The source device is made of:

- the light source which is a stabilized tungsten halogen lamp with a continuous emission spectrum from 360 nm up to 2000 nm,
- the input optical fiber operating in the visible and the near infrared spectrum,
- an achromatic objective optimised for the spectral range and that focuses the incident light in the input plane of the sample,
- and a polarizer for giving access to the polarization dependence of the transmittance or reflectance coefficients.

The diameter of the spot on the sample is about 4 mm.

The light transmitted or reflected by the sample is then collected by the detection optics ("Fig. 2"). This second objective is mounted on the rotating arm of the goniometer which is able to rotate and to be locked at a precise position.



Fig. 3. The detection device.

The detection device ("Fig. 3") is made of:

- an achromatic focusing objective, which collects and focuses the transmitted or reflected light from the sample under test in the output fiber,
- the output optical fiber operating in the visible and the near infrared spectrum,
- an analyser for giving access to the polarization dependence of the transmittance or reflectance coefficients,
- and the spectrophotometer which is the Optical Spectrum Analyzer ANDO AQ 6315-A of the Institut FRESNEL. The spectral range of the spectrophotometer extends from 600 to 1700 nm. When equipped with the detection optical fiber of 600 microns core diameter, the resolution reached is better than 4 nm.



Fig. 4. The goniometer equipped with the chamber and the lighting and focusing objectives.

The sample holder has a mechanical rotation around the axis of rotation of the goniometer. This rotation is used to measure the reflected light at  $7^{\circ}$  and  $45^{\circ}$  angles of incidence, and to measure the transmittance at every angle of incidence.

The mechanical translation of the sample along the vertical axis is also possible. This translation is used for:

- the IN and OUT displacement of the sample for performing the recording of the baseline,
- scanning the coated area along the vertical axis.

These movements are manually driven from the outside of the chamber and is located on the top of the chamber as shown on "Fig. 4".

The equipment is also designed to evaluate during the vacuum cycle partially the nature of the outgassing elements from the coating, using a mass spectrometer capable of measuring mass from 1 to 100. This is a novel approach and a relevant parameter to assess because it can quantify any permanent degradation of the coating due to the removal of constitutive elements from the layers.

In order to accelerate the outgassing process and provide for thermal cycling, the device includes a heating circuit to increase the temperature of the sample by up to  $50^{\circ}$ C.

In addition and to cater for the increasing demand for cryogenic optical space applications, the equipment also includes a sample cooling device using liquid nitrogen in order to characterize the optical coating at low stabilized temperature.

The Engineering Tool also includes a visual inspection capability (CCD time lapse) of the sample surface in order to detect any evolving physical degradation or surface cracking of the coatings.

In order to have the possibility to achieve other measurements (surface deformation due to vacuum condition for example), the chamber has additional two inches diameter windows. These windows are uncoated and slightly tilted in order to avoid possible problems induced by residual parasitic images.

The equipment is placed on a vibration limiting table ("Fig. 5").



Fig. 5. The engineering tool for qualification of optical coatings.

#### 2.2 Test sequence

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For each type of coating to be characterize under the air to vacuum transition, two samples will be tested according to the following test sequence:

- reflectance or transmittance measurement between 600 nm and 1700 nm after coating at room temperature and 1 atmosphere,
- surface quality inspection,
- humidity test, \_
- moderate abrasion test,
- solubility and cleanibility test,
- surface quality inspection, \_
- reflectance or transmittance measurement between 600 nm and 1700 nm after test at room temperature and 1 atmosphere,
- reflectance or transmittance measurement between 600 nm and 1700 nm in vacuum at room temperature,
- outgassing elements measurement,
- \_ reflectance or transmittance measurement between 600 nm and 1700 nm in vacuum at 50°C,
- outgassing elements measurement, -
- reflectance or transmittance measurement between 600 nm and 1700 nm in vacuum at low stabilized temperature,
- outgassing elements measurement,
- surface quality inspection.

For any kind of spectral transmittance or reflectance measurement, we will perform two recordings of the baseline, the first one just before the measurement and the second one just after this measurement, and we will use as reference baseline the mean of both spectral recordings (cancellation of the possible source drifts). At each step of the sequence, we will achieve a subtraction of the dark current contribution.

The environmental tests and the optical properties measurements will be done according to the International Standard ISO.

#### 3. CONCLUSION

For the needs of the European Space Agency, SESO has developed in cooperation with the Institut Fresnel an Engineering Tool for the Qualification of Optical Coatings for testing the behaviour and stability of optical coatings during the air to vacuum transition.

The preliminary characterizations will be done in June 2006 on reflective coatings, one anti reflective coating and dichroic filters.