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N. Valette

G. Chauveau

C. Grèzes-Besset

V. Costes

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PACA2M MAGNETRON SPUTTERING SILVER COATING: A SOLUTION FOR VERY BIG MIRROR DIMENSIONS

N.Valette[1], G.Chauveau[1], C.Grèzes-Besset[1], V.Costes[2], I.Savin de Larclause[2], K.Gasc[2], F.Lemarquis[3]

[1] CILAS Etablissement de Marseille, 600 avenue de la Roche Fourcade, Pole ALPHA Sud - Saint Mitre, 13400 Aubagne – France ; [2] CNES, 18 Avenue Édouard Belin, 31401 Toulouse- – France ; [3] Aix Marseille Université, CNRS, Centrale Marseille, Institut Fresnel, UMR 7249, 13013 Marseille – France

I. INTRODUCTION

Today high angular resolution telescopes for space observation satellites require primary mirrors with larger and larger dimensions. For such mirrors, high-performance silver coating is required and is a key part of the component. For more than 20 years, CILAS has developed an expertise in the field of thin film optical coatings in order to answer a wide range of spectral performances and produce high quality thin film coatings. Among the various coating technologies that may answer such demand, magnetron sputtering is particularly well-suited to produce dense layers with improved mechanical performances and a high level of uniformity.

For this purpose, CILAS is equipped since several years with a very large magnetron sputtering machine that can address components up to 2 meters by 2 meters dimensions. In particular, such machine called PACA2M which acronym stands for PulvérisAtion CAthodique de 2Mètres is used to produce the metallic amplifiers reflectors for the laser Megajoule project for the French atomic agency (CEA) in which CILAS is involved with the responsibility for the production of the amplifiers.
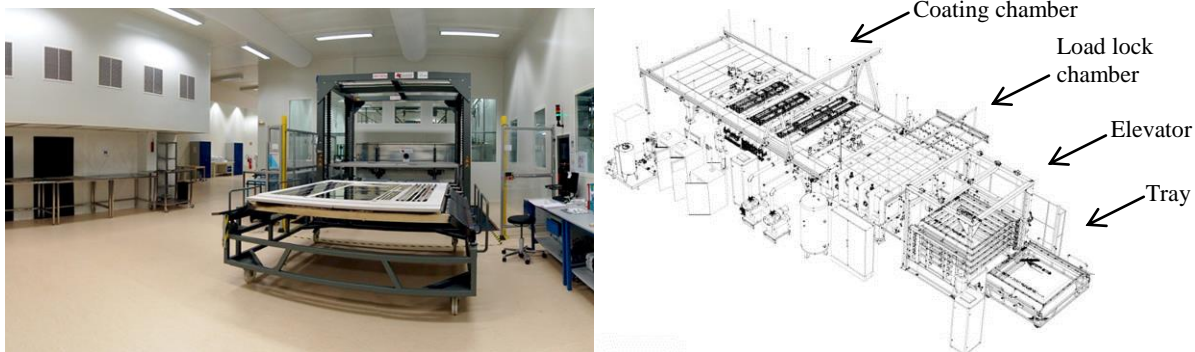

Fig.1. PACA2M deposition magnetron sputtering machine

Space environment is particularly severe for optical components. Optical coatings are required to present extreme performances and very stable properties from the beginning to the end of life of the components. In particular metallic coating is commonly used for space applications. This coating must withstand qualification environmental tests. In the frame of Research and Development activity in collaboration with the French space agency (Centre National d'Etudes Spatiales, CNES), CILAS has developed and validated a specific silver coating for space telescopes mirrors.

In a first step, a lot of different characterizations and tests have been led on samples including environmental qualification tests, stress and spectral measurements. As this campaign was successful on samples, a second step has been decided. This step was the coating of a full 600 mm mirror demonstrator.

II. PACA2M: 2-METER SPUTTERING MACHINE

PACA2M sputtering coating machine is an inline coating system equipped with seven planar 2.5 meters-long magnetrons in a 14 m³ vacuum chamber. As illustrated in the following figure (Fig.2), the substrates are scrolling under the material targets in a back and forth motion with a speed which is monitored to deposit layers from a few tenths of nanometers to hundreds of nanometers.

PACA2M sputtering machine has been designed for producing uniform large coatings on 2 meters by 2 meters components, with a thickness up to 40 centimeters and a weigh of 1.5 ton. With its various cathodes that may be powered in different modes (RadioFrequency (RF), Mid Frequency (MF), Direct Current (DC) or Pulsed DC) [1,2,3,4,5,6], it allows to address a wide range of applications using optical multidielectric and

metallic functions such as anti-reflection coatings or high reflection mirrors from ultraviolet to near infrared [7,8,9].
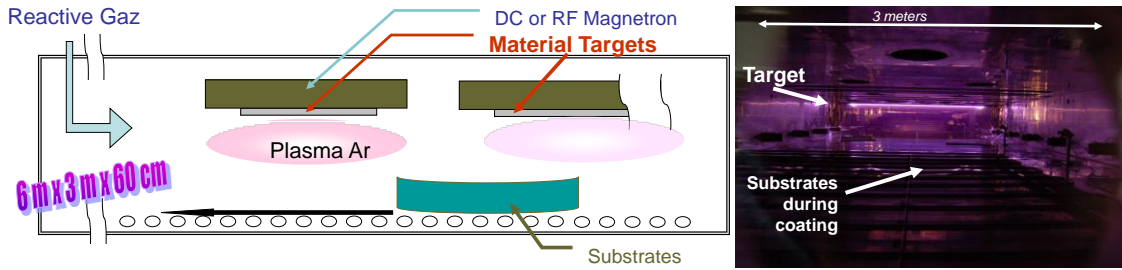

Fig.2. Inline coating system principle (left) and view of plasma inside the 2mx2m PACA2M chamber (right)

On schematic view of the coating machine (Fig.1), the elevator of trays with 5 positions can be noticed as a front part of the 9 m^3 load lock chamber which is coupled with the machine.

Moreover, PACA2M is equipped with an effective optical broadband monitoring (BBM) that enables us to monitor in situ the optical performances with an excellent reproducibility [10]. Such all-fibered system developed in collaboration with Institut Fresnel permits to measure the in situ optical performances over a [280-2200nm] spectral range and to stop the deposition and automatically trigger to the next layer. It also gives access to an accurate characterization of material parameters such as refractive index dispersion curves.

Coupled with the BBM as a stop criterion, automated processes are achieved in an industrial environment without any operator during the process, with a high repeatability and a realization error less than 1% [11,12].

Next to PACA2M coating machine, CILAS has a set of equipment adapted to large heavy components inside and outside the clean room. In particular, a large cleaning machine (called KOMBI650) with eight-tanks (ultrasound, detergent, water) is used to successively wash, rinse and dry large pieces; the final rinse is done with high quality deionized water, followed by a lift-out of a few millimeters per second under filtered hot air.

Fig.3. Automatic cleaning machine for large components

Various tooling for handling and motorized hoist are also available as illustrated below.
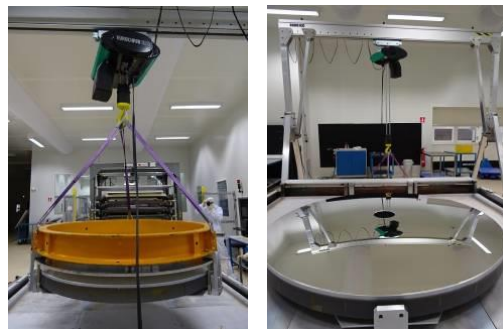

Fig.4. Handling and lifting large components. Here a Zerodur® mirror manufactured by AMOS for Javalambre Astrophysical Observatory (OAJ)

III.   REALIZATION OF SILVER COATINGS FOR SPACE APPLICATIONS

Among the many capabilities that PACA2M offers and the various materials available for the silver-protective layers, a tradeoff between spectral performances and robustness has been done with the French space agency (Centre National d'Etudes Spatiales, CNES), in the frame of Research and Development activity. It has led to the selection of a design based on silver with protective oxide layers, which exhibits the best industrial maturity of the process.

The first phase of our Research and Development activity consisted in a qualification step on samples including adhesion tests, with a characterization and optimization of the process parameters in order to reach high performances of uniformity and spectral performances. During this phase, as it can be responsible for coating degradation in severe environment, we also validated the mastering of the uncoated area at the edge of the component and the presence of a well-defined strip. Finally the stress of each material involved in the selected design has been studied in order to model and predict the surface deformation.

At last, we also studied several technological aspects such as electrical grounding implementation of the coating and coating removing.

The second phase of the activity consisted in coating a large component provided by CNES. The aim of this phase is to validate the coating with a full size mirror. The validation criteria were the quality of the coating and also the mechanical behavior of the mirror after coating

A.   Characterization of coating uniformity over large dimension

A set of dedicated samples in silica and Zerodur® which characteristics are given in the following table has been defined for the qualification tests.

Material	Samples	Dimensions (mm)	Bevel	Strip	Grounding
Zerodur®	Validation	25 x 25 x 3	Yes	Yes	Yes & No
Zerodur®	Divoli	25 x 25 x 3	No	No	Yes & No
Zerodur®/Silica	Stress	φ 25.4 x 0.5	Yes	Yes	Yes & No
Silica	Uniformity	φ 25 x 2	Yes	Yes	Yes & No

Square "Validation" samples with bevels all around the edges and uncoated area have been used for studying peripheral area as well as square "Divoli" samples with sharp edges and no uncoated strip.
Circular "Stress" samples have been used for stress characterization.
"Uniformity" samples have been positioned along circles with 500mm, 1m, 1.5m and 2m diameters all over the tray of the coating machine for uniformity characterization of thickness and reflectivity (see Fig.6).
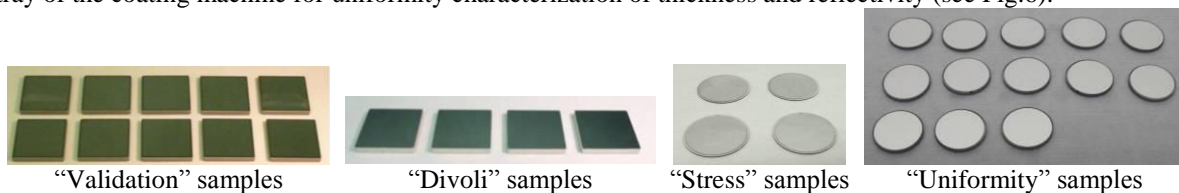

"Validation" samples   "Divoli" samples   "Stress" samples   "Uniformity" samples
Fig.5. Photographs of dedicated samples coated on the 2m x 2m PACA2M tray

A characterization of thickness uniformity over the 2mx2m coating tray has been done including all the samples that have been simultaneously coated, for the qualification phase and for the coating phase on the large 591mm diameter component.
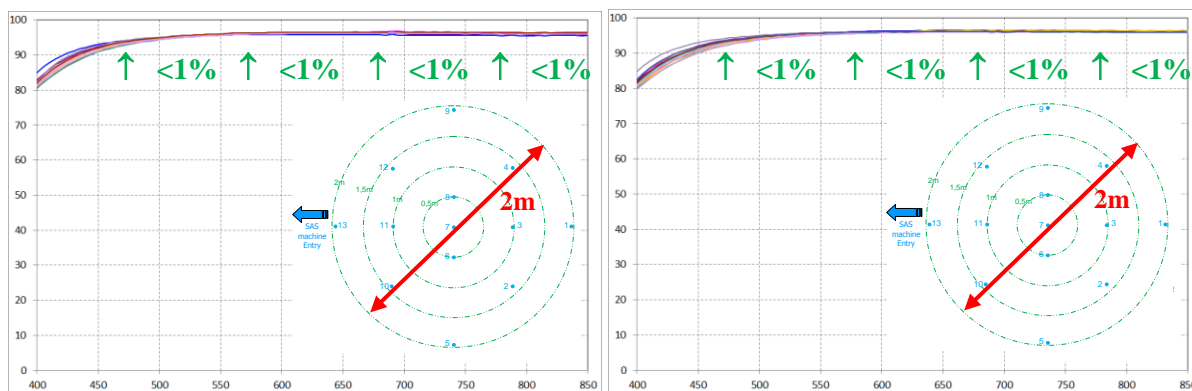

Fig.6. Spectral reflectivity over 2m x 2m measured on samples for several productions of the R&D Activity

As presented in the previous graphs (Fig.6) where we have calculated the variations of the reflectance responses at several wavelengths, we can see that uniformity of the spectral performances is better than 1% on all the samples over the 2m x 2m dimensions.

Moreover, we can notice that the spectral responses of all the samples are very similar and reproducible from one production to the other.

B. Qualification tests

A test plan has been established following specifications defined by CNES. The tests are performed to ensure that moisture and thermal cycle do not affect adhesion or spectral performances of the coating.

For the humidity test, the samples have been placed in a climatic chamber and it has been checked that no condensation occurred. The conditions have been applied during 48 hours with relative humidity between 90% and 95% and a temperature of 40°C. Twenty thermal cycles have been realized between -15°C and 70°C under atmospheric pressure with a slope of 10°C per minute and waiting duration of about 30 minutes (Fig.8).

Humidity test, thermal cycle and adhesion test have been carried out on dedicated samples, as spectral measurement and visual inspection have been made before and after each test on all the selected samples and WFE measurement has been carried out on "stress" samples.
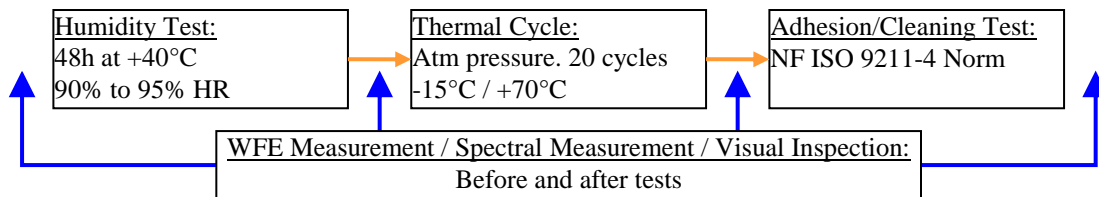

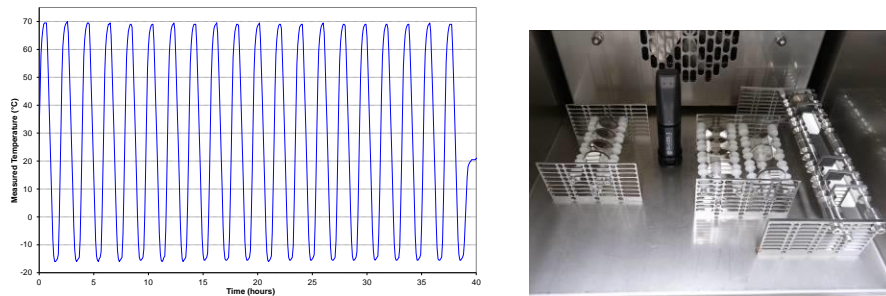**Fig.7.** Test sequence for qualification

Fig.8. Diagram of temperature during thermal cycles with samples ready for thermal cycle

The graphs below give the spectral measurements of "Validation" samples after each qualification step (coating, humidity test and thermal cycle) and show that there is no degradation after test.
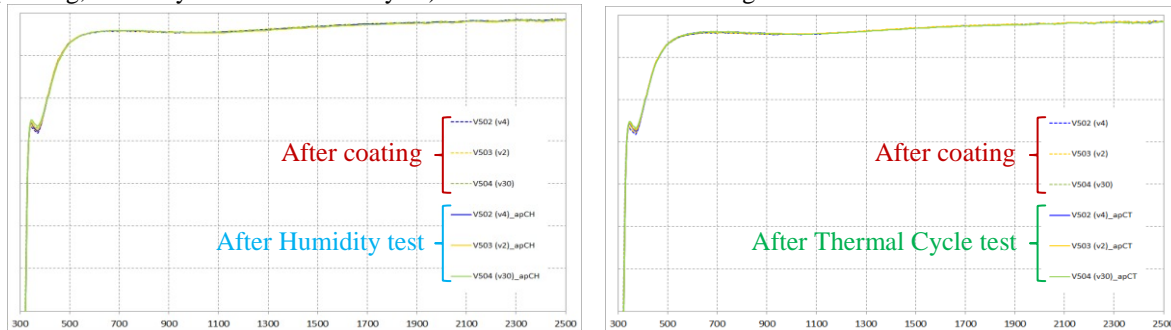

Fig.9. Spectral measurements before and after humidity test and thermal cycle

The analysis of all the results obtained during the two phases of the project shows:
- A very good stability of the spectral response
- No degradation in adhesion test at level 2
- No degradation in adhesion test at level 3 (which was not requested ISO 9211-4 norm)
- No evolution of the cosmetic performances

As a conclusion of these tests, these successful qualification tests demonstrate that CILAS magnetron sputtering silver coating is capable of space harsh environments.

C.   Characterization of stress induced by the coating

A characterization of the stress has been done in order to quantify the deformation induced by the coating; for this characterization, "Stress" samples have been used as they have been designed to present a very thin 500μm thickness for a 25mm diameter. Wave front error (WFE) measurements have been performed at the Institut Fresnel with a NewView 7300 ZYGO optical profilometer on a field of 10x14 mm².

At first, each uncoated sample has been characterized before any operation. Then, a monolayer of each material involved in the reflective coating (grounding material, adhesion layer, silver layer, protective materials) has been deposited on these samples and WFE measurement has been done after coating on each sample in order to characterize the stress for each monolayer.

For this operation, a specific tool has been developed to guarantee a perfect repositioning of the samples using a mark that has been implemented on the "Stress" samples.

In order to eliminate the initial non-planarity of the samples, WFE measurements have been corrected by subtracting the surface topography which has been measured prior to any operation as shown in Fig.10.
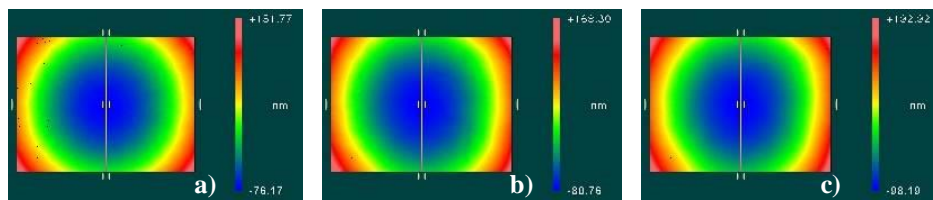

Fig.10. WFE measurements a) after coating, b) after humidity test, c) after thermal cycle

The analysis of the WFE measurements is done with the help of the Stoney formula, given hereafter, where the radius of the deformation is directly related to the mechanical stress of the layer.

$$\sigma_{layer} = \frac{E_{substrate} t^2_{substrate}}{(1 - v_{substrate}).6.t_{layer}.R}$$

$E_{substrate}$ represents the Young modulus of the substrate
$v_{substrate}$ represents the Poisson coefficient of the substrate
$t_{substrate}$ represents the thickness of the substrate
t_{layer} represents the thickness of the monolayer
R represents the radius of the deformation
σ_{layer} represents the mechanical stress of the layer

It must be noted that the mechanical stress σ_{layer} in the layer contains an intrinsic part due to the layer material itself and an extrinsic part depending on both the layer material and the substrate corresponding to the differential expansion existing between the layer and the substrate. As a result, these stress values are corresponding to a given temperature and for a given substrate.

A thermal chamber has been implemented on the profilometer by Institut Fresnel to allow measurements at different temperatures up to 65°C regulated at 0.1°C.

Assuming perfect adhesion of the layers at each interface and an additivity of the various mechanical stresses, we can then calculate the theoretical radius of curvature R of the complete stack by summing the various individual curvatures (1/R$_i$) calculated for each layer taking into account its thickness:

$$\frac{1}{R} = \sum_i \left(\frac{1}{R_i} \right)$$

D.   Results of stress study on samples

As previously discussed, an optimization of the process parameters for protective layers has been done during the first phase and applied to the multilayer stack to optimize the global performances of the mirror coating; in particular, our developments have been drawn by a minimization of stress and an improved environmental stability. On the following table, we give the comparison of the measurements of the curvature radius done on mirror-coated samples and the theoretical prediction of the deformation for several different batches.

These results show that there is a good agreement between measurements and prediction, which demonstrates the validity of our model.

Batch	Grounding	Measured Radius (m)	Estimated Radius (m)
1	No	-176.0	-150
2	No	-166	-173
3	Yes	-321	-333
3	No	-163	-156

Fig.11. Measured and estimated radius of curvature after coating on silica "Stress" samples (ambient t°)

Moreover, it has been noticed that stress compensation is obtained between the various layers constituting the mirror, mostly between the grounding layer and the protective layers that allows reaching a lower global deformation than without the grounding layer (see Fig.12). These results have been confirmed by the prediction based on the individual stress analysis of each layer.
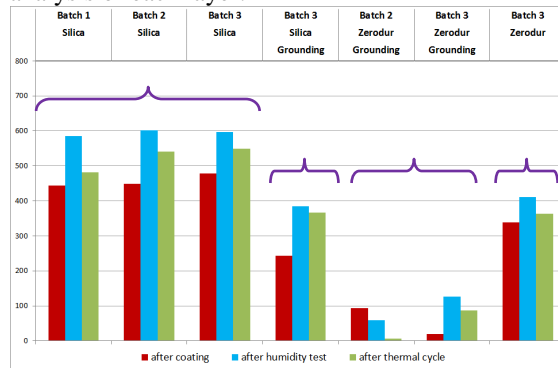

Fig.12. Evolution of the sagitta after environmental tests (WFE measurement)

In conclusion, elementary and global stress measurements on samples have permitted to characterize and minimize the global stress of the coating. This also allows us to predict the mirror mechanical deformation and demonstrate:
- Master of the deformation
- Equivalent deformation after coating and after environmental tests
- Validation of the process reproducibility
- Repeatability of stress from batch to batch
- Validation of modeling stacks deformation

IV.   COATING OF ZERODUR® 591MM DIAMETER MIRROR
The last phase of the project consisted in coating and characterizing a Zerodur® 591mm diameter mirror provided by CNES.
A.   Description of the process
Our standard production flow which is carried out for large dimension components has been followed including unpacking, removal of protecting film, preliminary cleaning aimed to remove long time storage and/or shipping contamination residues, visual inspection to check the surface quality before any operation, and then coating involving electrical grounding implemented in two steps: a) coating of a conductive layer on the whole surface b) mirror coating over the useful area after installation of a mask.

Fig.13. Visual inspection after cleaning (right)

Final coating has been deposited on the mirror following this process and final cosmetic inspection has shown that no defect has been generated by the coating; only, defects that were still present on the uncoated surface have been observed or slight stains due to the cleaning has been revealed by the coating. Moreover, a uniform strip about 4mm large from the bevel has been realized as defined.
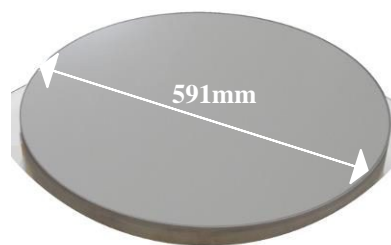

Fig.14. Coated M1 Mirror on the tray (Zerodur®, 591mm diameter)

B.   *Spectral response*

Another parameter that must be taken into account is the shape of the mirror that has an impact on deposited thickness. A characterization of this parameter has been done with the help of witnesses located in a tooling that simulates the shape of the mirror and witnesses located on the periphery of the mirror.

An estimation of the average reflectivity on the mirror is done with the weighted sum of the witness reflectivity by the area of the ring on which it is located as given hereafter:

$$R_{estimated} = \frac{\sum_{i}(R_i \cdot S_i)}{S_{Total}}$$

Fig.15 gives the estimated curve of the spectral response compared to the minimum and maximum curves measured on witnesses.
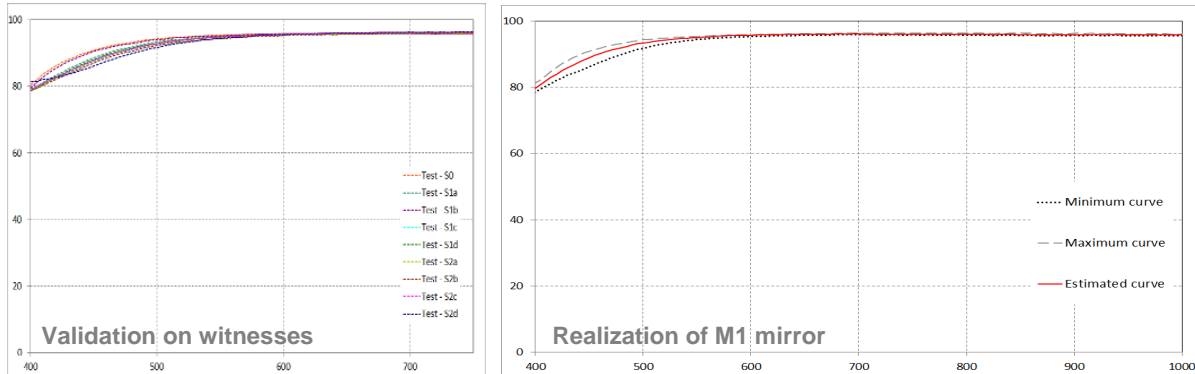

Fig.15. Spectral measurements of witnesses during the validation test on the left and estimated average reflectivity of the mirror on the right

C.   *WFE measurement on the mirror*

A characterization of the wave front error induced by the coating has been realized on the mirror by the Laboratoire d'Astrophysique de Marseille (LAM) with:

-        a mechanical spherometer for radius measurement to characterize the curvature variations
-        a Fizeau interferometer equipped with a simultaneous phase-shifting to perform shape measurements with spatial sampling of about 0.93mm (analyzed pupil diameter: 600 pixels). The contrast of the fringes is adjustable by the tuning of the relative intensities of the two beams by polarizing effect with an attenuator.

Initial measurements have been done on the mirror before coating.

The radius measurements lead to a variation of the 1800mm mirror radius after coating equal to -1.4mm, which is in the order of magnitude of the set-up accuracy.

As presented on the following maps (Fig.16), where the coating effect is obtained by subtracting the map before coating to the map after the coating, a slight astigmatism has been noticed eventually due to the structure of the mirror itself and the main result is that no high frequency is revealed.
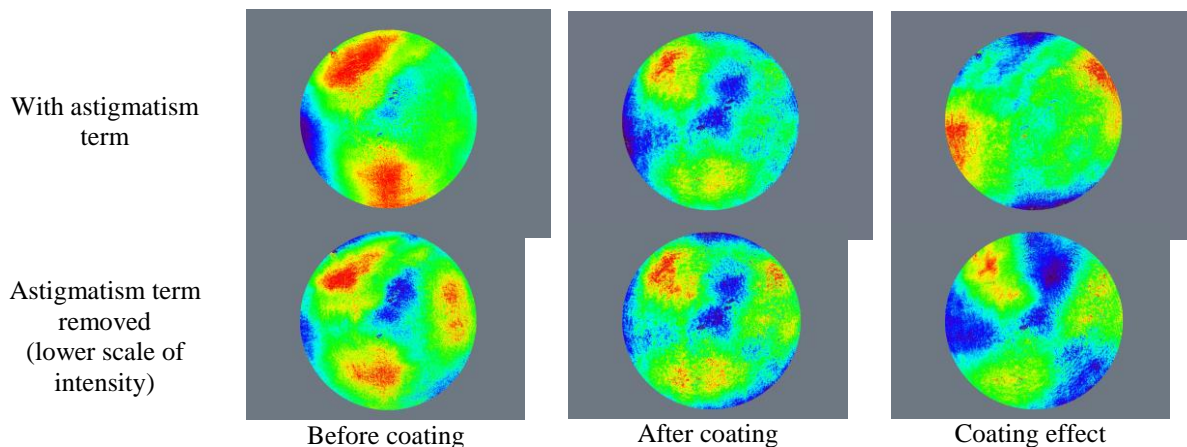

Fig.16. WFE measurements of the M1 mirror before coating after coating

The specific structure topography of WFE with astigmatism term removed (Fig.16) is due to the 3 stiff areas of the mirror for mechanical interfaces.

This WFE measurement demonstrates that no quilting is induced by the coating on such a very lightweight mirror.

V.   CONCLUSION

In this paper, we have presented the validation of CILAS magnetron sputtering silver coating for space applications. Our experimental results and characterizations on samples are detailed. The coating of a 600mm lightweight mirror is shown.

The results of the test plan recommended by CNES (humidity test/ thermal cycle/ adhesion/ cleaning) show that the spectral response, cosmetic and mechanical strength remain unchanged after environmental tests for each phase, which confirms robustness of the magnetron sputtering technology silver coating.
Uniformity of reflectivity for wavelengths greater than 550nm is better than 1% over the 2m by 2m tray. Stress analysis on thin samples shows that measurement and theoretical prediction of deformation are in good agreement and reproducible from batch to batch.
The silver coating has been optimized with mechanical strength and spectral performances improvements.
From a cosmetic point of view, the final performances are very good and the peripheral uncoated strip is well centered and uniform. Measurements of the curvature radius and wave front error before and after coating are promising as no quilting defect is appears.

As a conclusion, CILAS silver coating is validated. A qualification sequence on samples and a 600 mm lightweight Zerodur® mirror have been successfully run. The stability, the reproducibility, the homogeneity and the performances of this coating are characterized. CILAS PACA2M coating machine is a promising device for space applications and beyond.

VI.   REFERENCES

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