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High performance silver coating with PACA2M magnetron sputtering

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ABSTRACT

The performances that are required today for optical components are increasingly demanding, both in terms of the spectral response and the conditions to which the components are subjected. So-called dense deposition techniques for thin film coating currently make it possible to respond in many cases to this ever-increasing requirement. Indeed, conventional evaporation techniques that can address a large number of applications such as metallic coatings, anti-reflection coatings, mirrors, are no longer sufficient and we currently rely on the dense deposition techniques such as, for instance, magnetron sputtering which allow implementing layers stable and low-ageing behavior. In this paper, we will review the recent achievements at CILAS by magnetron sputtering technique on high-performance silver coatings for space applications.

In our premises in Aubagne, CILAS has implemented a large dimension in-line magnetron sputtering coating machine with which such silver coatings are realized. This equipment with oversized dimensions involving a 6 meters by 3 meters vacuum chamber, 12 turbomolecular pumps and 7 planar magnetrons over 2.50m long will be presented in detail. In particular, the wide range of configurations will also be illustrated with complex coatings achievements with respect to the capability of the machine which is equipped with magnetrons, that may be powered in Radio Frequency, Mid Frequency, Direct Current or pulsed Direct Current modes.

For several years, CILAS has improved its coating processes to produce high quality silver coatings, taking into account spectral performances, environmental conditions and also coating uniformity over 2meter by 2 meter dimensions.

Further developments have been led by CILAS with the support of the French Space Agency as part of Research and Development activities to improve environmental performance; we present here characterization results on samples before and after environmental qualification (temperature, humidity, vacuum heat ...) and on components with dimensions up to 600mm. A multi-year test campaign including stress measurements and adhesion tests on samples and mirror demonstrator will also be presented.

Keywords: thin film coatings, magnetron sputtering, telescope mirrors, large dimensions

1. INTRODUCTION

For several years, CILAS is involved in coating development and production for space applications. A lot of specific optical functions have been studied and improved to be able to reach high performances for severe environments.

Among the various physical vapor deposition technologies that are available for coating, CILAS has focused its expertise on dense technologies such as ion assisted deposition (IAD), ion beam sputtering (IBS), dual ion beam sputtering (DIBS) or plasma sputtering. Involvement of CILAS in space projects is illustrated as presented in the following paragraph.

For instance, ion assisted deposition which consists in a technology based on evaporation of the materials with electron guns [1] and densification of microstructure with the help of an ion beam gun, has been demonstrated to be really efficient. For example, CILAS has produced using this technology the laser dichroic of the CHEMCAM instrument that was a part of Mars Curiosity mission in 2006 [2] and more recently a similar dichroic for the Mars 2020 mission.

Dual ion beam sputtering which is based on a technology using an ion beam gun to sputter the materials involved in the multilayer is an appropriate technology for tremendous spectral performances as well as high quality of layers microstructure and smooth interfaces [3,4]. As an example, CILAS is involved in the Microcarb mission whose objective is to map the sources of the main greenhouse gas (CO2) and is in charge of the development, qualification and realization of narrow band pass filters (Figure 1) and dichroics for which ion beam sputtering technology is implemented.

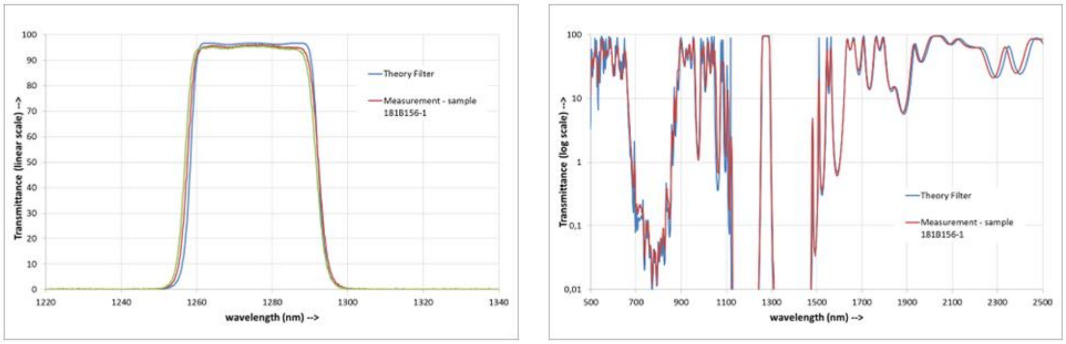


Figure 1. Realization of complex filters in DIBS technology for Microcarb mission (comparison theory - measurement)

Magnetron sputtering technology has also proven for the last years its capacity to produce multilayers coatings with a very high number of layers and a huge agreement between experimental and theoretical curves. For instance, PH2 filters for TARANIS mission have been successfully realized (Figure 2) and implemented in the scientific observation satellite of the French spatial agency (CNES).

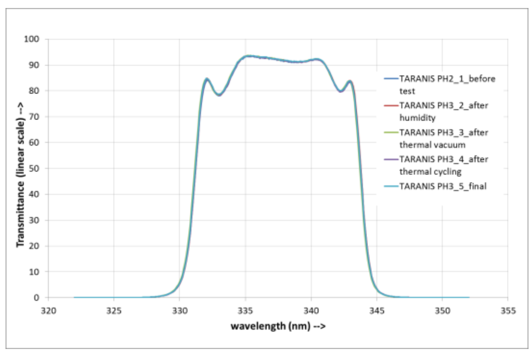


Figure 2. Realization of complex filters in magnetron sputtering technology for Taranis mission (no evolution after environmental testing)

Improvement of optical properties of such complex coatings has been made possible thanks to the development of in situ optical monitoring which can be now considered as a guarantee to produce very demanding spectral performances [5,6,7,8].

2. PACA2M COATING MACHINE FOR LARGE COMPONENTS

So-called dense technologies which are really valuable for complex functions can be relied on for metallic coatings, as they also exhibit a real improvement of mirror coatings characteristics, in particular considering ageing problematic.

For several years, CILAS has studied in collaboration with CNES improved silver coatings for space applications. The main characteristics that must be fulfilled are not only the spectral reflectance which may be required as well for UV visible region as for infrared, or for a wide range of incidence leading to polarization effects, but also uniformity performances for large components, cosmetic, low ageing behavior and repeatability of production.

CILAS developed its expertise for the production of large optical coatings for several years thanks to the large PACA2M coating platform which has been installed in 2010 in its Aubagne premises in the south of France [9].

Such coating platform is constituted with a large magnetron sputtering machine that can address optical components up to 2 meters by 2 meters dimensions, 40 centimeters thick and to 1.5 ton weight as well as a large ultrasonic cleaning machine. A broadband optical monitoring especially designed has been implemented on the coating machine allowing to reliably achieving spectral performances in an industrial environment on large surface, and also in situ material characterizations and thickness determination [10].

Designed as an inline coating system, allowing components to move back and forth under the materials to be deposited, such coating machine is constituted with a 14m^3 vacuum chamber, equipped with seven planar 2.5 meters-long magnetrons and a 9 m^3 load lock chamber, with twelve turbo pumps and a cryogenic pump.

Figure 3. View of the PACA2M deposition magnetron sputtering machine, with PACA2M acronym for PulverisAtion CAthodique de 2M.

Since 2010, CILAS produces the metallic reflectors for the laser Megajoule project [11,12] for the French atomic agency (CEA) using magnetron sputtering technology with PACA2M. The company took advantage of this equipment to develop a catalog of optical functions [13,14]:

- Metallic mirrors constituted with Al or Ag reflective layer, protected or enhanced with dielectric multilayers
- Antireflection coatings or dielectrics mirrors and beam splitters
- Absorbing metal dielectric coatings for parasitic light reduction [15,16]

3. SILVER COATING REALIZATION WITH PACA2M

3.1 Spectral response

The main advantages of magnetron sputtering technology are its robustness versus severe environment and also its characteristics of uniformity and process stability [17]. These properties have been widely demonstrated and are illustrated by the results we get in production for metallic coatings for instance. With the experience gained for Laser MegaJoule reflectors, we have deepened our expertise and in particular, we have worked with the help of CNES to improve the performance of silver coatings for space applications [18]. A development was conducted according to a trade-off analysis between the main characteristics (spectral reflectivity, ageing behavior, stress, …).

3.2 Qualification tests

A test plan defined upon CNES specifications has been applied on samples representative of final components in order to qualify environmental behavior of the coating, with spectral measurements at each step. The various tests are listed hereafter:

- Humidity Test: 48h at +40°C±2°C with 90% to 95% relative humidity
- Thermal vacuum : 34 cycles [-15°C ; +70°C] under vacuum with less than 10°C/min variations and 30 min step
- Thermal cycles : 20 cycles [-15°C ; +70°C] under atmospheric pressure
- Adhesion tests at each step in accordance to ISO 9211-4 standard, severity level 2 and 3

Spectral measurements on witnesses show similar responses after coating and after each qualification step, as presented in the following graph (Figure 4).

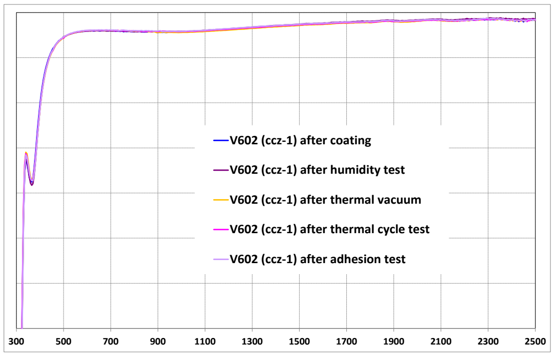


Figure 4. Spectral measurement on a sample after each qualification step

3.3 Coating removal and recoating

For space application, another requirement for mirror coatings is the capability to remove the coating and to re-coat the component without any degradation. Our qualification study has covered this requirement as it has been demonstrated that it is possible to chemically remove the coating and to recoat the substrates with the same level of performances.

This is illustrated in the following table (Figure 5) where we present the roughness measurements at each step of our tests plan. The topography of the substrates was recorded at different scales by interferometry in white light using microscope at Institut Fresnel, before coating (Step 1), after coating (Step 2), after coating removal (Step 3), and after re-coating (Step 4).

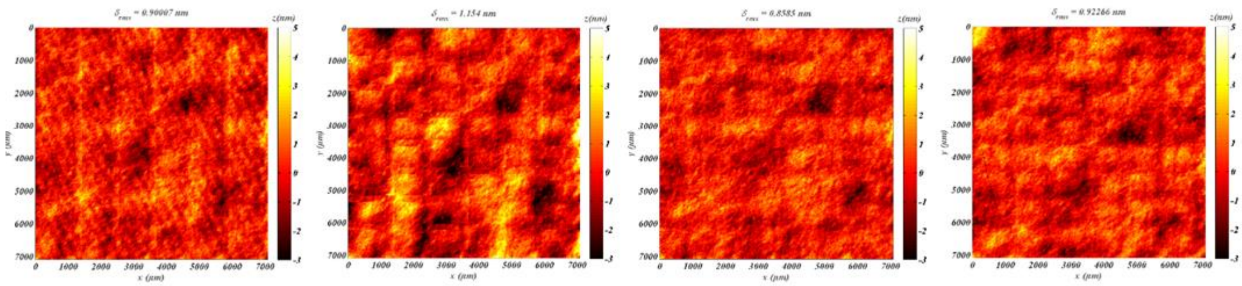


Figure 5. Surface topography maps by interferometry using white light microscope at Institut Fresnel (ref.V602 sample)

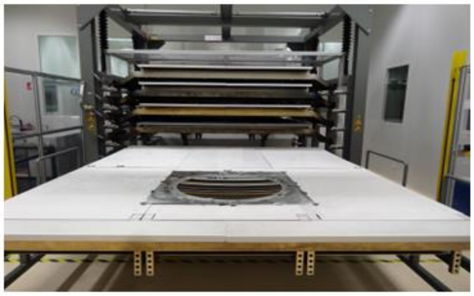
The roughness values given in the following table (Table 1) show that there is no degradation, which demonstrates the validity of the method thus developed.

sample ref#	before coating (nm)	after coating (nm)	after coating removal (nm)	after recoating (nm)
#V601	0.85	0.97	0.89	0.93
#V602	0.90	1.15	0.86	0.92

Table 1. Quadratic roughness (nm) values after each step (#V601 and #V602 samples)

3.4 Ageing behavior

We have also tested the robustness of the coating on a real mirror (Figure 6) to qualify ageing over time, which is of great interest for space applications. This mirror is a zerodur ® 591 mm diameter M1 mirror and has already been presented [18].



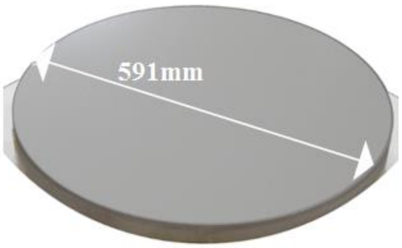
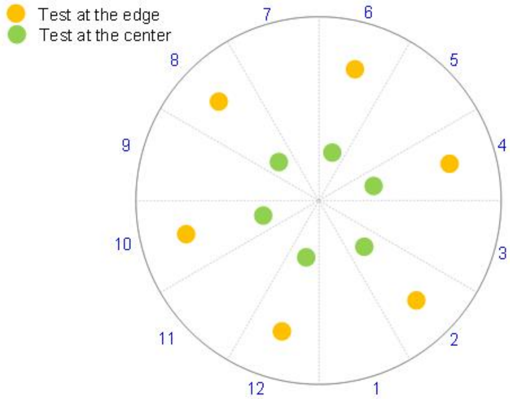


Figure 6. Coated M1 Mirror on the tray (zerodur®, 591mm diameter)

For the present study, the surface of the mirror has been divided into 12 sectors. The adhesion tests were performed at the center (orange bullet) and at the edge (green bullet) in a different sector every 2 months. In accordance with the NF ISO 9211-4 standard, the adhesion test is performed with method 2, severity level 02.



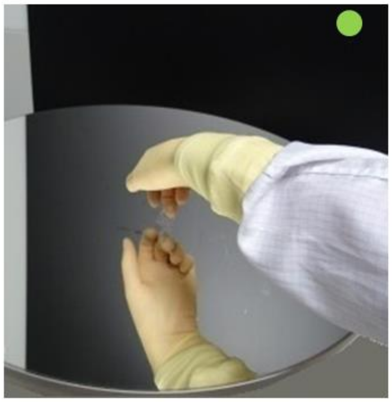


Figure 7: Adhesion tests done over 9 months period in different sectors (locations of the tests on the left) of a 591mm mirror. Adhesion test in progress at the center is shown on the right.

As presented in the following table (Table 2), no degradation of the coating appeared after the adhesion tests performed over a 9 months period.

Test #	Sector ref (cf Figure 7)	Test date	Result at the edge	Result at the center
1	2	31/05/2016	No degradation	No degradation
2	4	28/07/2016	No degradation	No degradation
3	6	22/09/2016	No degradation	No degradation
4	8	07/11/2016	No degradation	No degradation
5	10	06/12/2016	No degradation	No degradation
6	12	26/01/2017	No degradation	No degradation

Table 2. Results of adhesion tests (method 2, severity level 02) on the M1 mirror following NF ISO 9211-4.

3.5 Stress characterization

Another topic of interest concerns the constraints that may be induced by the coatings. Long-term development collaboration with CNES has allowed us studying and optimizing magnetron sputtering process parameters to reduce stress in the various layers constituting the mirror coating and to check their repeatability.

As presented in [17], stress characterization of individual materials has been performed at Institut Fresnel with a NewView 7300 ZYGO optical profilometer on a field of 10x14 mm^2 on samples specifically designed to present a very thin 500µm thickness for a 25mm diameter. Furthermore, WFE characterization on M1 mirror has been realized at Laboratoire d'Astrophysique de Marseille (LAM) with a mechanical spherometer and a Fizeau interferometer equipped with a simultaneous phase-shifting (0.93mm spatial sampling, 600 pixels analyzed pupil diameter), showing that no quilting is induced by the coating on such a very lightweight mirror.

Characterizations done on M1 mirror after 6 months and 12 months period are presented in the following table (Table 3) which gives the surface front error differences after subtraction of the initial measurement done before coating. This leads to very similar results as obtained before coating (35nm RMS-191nm PV) or after coating (24nm RMS-133nm PV) as they are included in metrology bench accuracy, which shows that the M1 mirror has not evolved after 6 months nor 12 months.

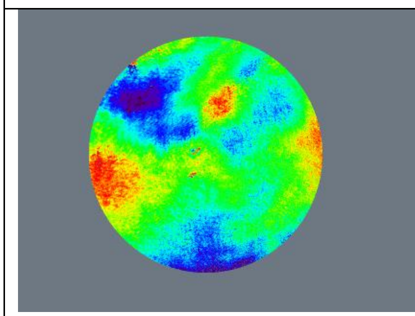
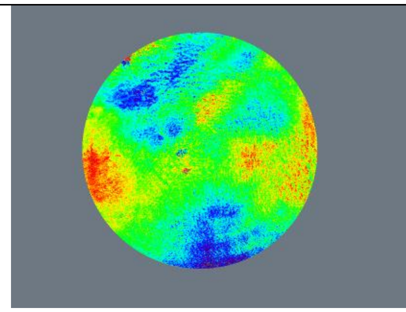
Measurement after 6 months	**Measurement after 6 months**
	
21nm RMS (129nm PV)	16nm RMS (103nm PV)

Table 3. WFE characterizations done on M1 mirror after 6 months and 12 months period after subtraction of measurement done after coating with estimation of deformations after correction of metrology bench contribution .

As some indetermination still remained on the M1 mirror, in particular slight astigmatism which could be due to the M1 geometry itself, additional work has been lead on very thin 3mm thickness for a large 300mm diameter plane substrates (Figure 8). Characterizations before and after coating have been done at LAM with Fizeau interferometer over 250mm pupil.

Figure 8. 3mm thick, 300mm diameter sample used for stress measurement

Difficulties were encountered for WFE measurements of these components due to their shape factor (ratio between diameter and thickness) which is very unusual. To overcome this issue, a dedicated specific whiffletree tool for supporting the component on the bench has been developed by LAM and the contribution of the whiffletree has also been evaluated as presented in the following graph (Figure 9).

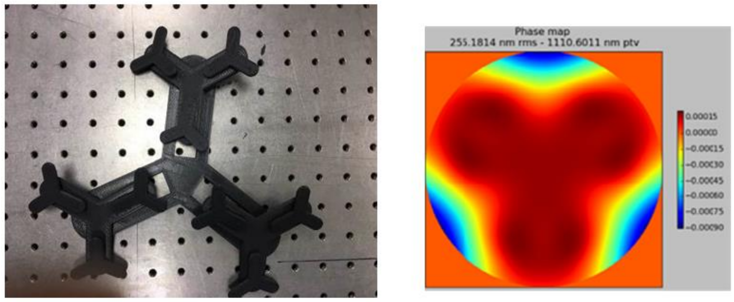


Figure 9. Whiffletree tool developed for WFE characterization at LAM (on the left). Deformation induced by the whiffletree (on the right).

In agreement with CNES, several configurations were tested for WFE characterizations using various mounts (glass window, foam and whiffletree tooling).

Metrology issues have also arisen due to the contributions of both front and back surfaces which were not separable and several measurements have been performed on one side polished and both sides polished components.

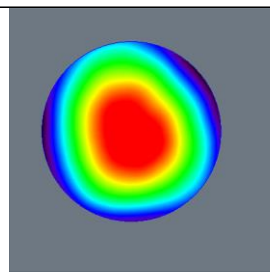
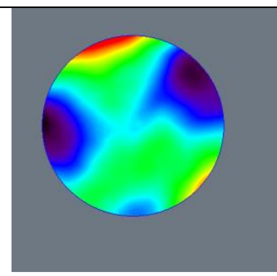
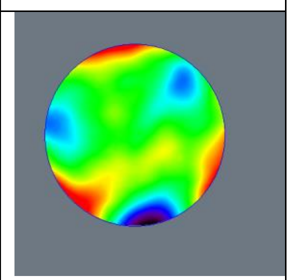
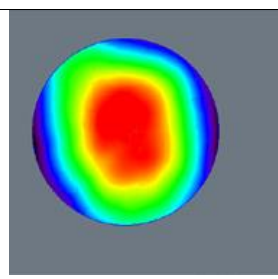
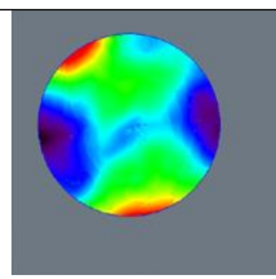
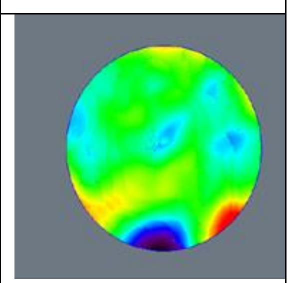
	With curvature	**Without curvature**	**Without curvature and astigmatism**
Before coating			
After coating			

Table 4. Surface front error before and after coating on one-side polished substrate (#ref L1: 3mm thick, 300mm diameter)

Despite those difficulties of evaluation, characterizations have been done showing that the dominant effect of the coating is on the curvature and slightly on astigmatism. Analysis of the results is still under investigation. In particular, the effect of whiffletree support is higher than the one predicted by the finite element model, which leads mainly to astigmatism and trefoil higher than predicted. This discrepancy can be due to the difference between whiffletree as modeled and as manufactured.

4. CONCLUSION

In this paper, we have presented tests done on samples and on M1 mirror with dimensions up to 600mm in the frame of environmental qualification of silver coating produced by magnetron sputtering technique.
Implementation of coating removal has been successfully studied with no impact on initial surface roughness, and we have also shown its compatibility with successful silver re-coating.
Adhesion tests performed over a long period of several months done on a real mirror have also been presented with no degradation.

A characterization of stress has also been carried out and measurement of M1 mirror has been presented showing no evolution after 6 months or 12 months. Deformation characterization on large thin substrates has also been studied and required a complex implementation because of their very thin thickness compared to their diameter. Measurements before and after coating required the development of a whiffletree type of support and a method of characterization adapted to these substrates. The results show a dominant term of curvature whose value is close to expected estimations and a slight term of astigmatism. Analysis of the results is still under investigation.

Qualification results presented in this paper show that magnetron sputtering technique allows producing robust and low ageing large coatings which are well adapted to space applications.

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