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Procedia

Energy Procedia 69 (2015) 1519 - 1528

International Conference on Concentrating Solar Power and Chemical Energy Systems, SolarPACES 2014

Critical constraints responsible to solar glass mirror degradation

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Abstract

The degradation of solar glass mirrors is analyzed as a function of various constraints such as temperature, humidity, rain and solar irradiation. In order to understand the degradation mechanisms, two kinds of tests have been performed: simple tests to identify the effect of each constraint and complex tests to highlight the effects of synergy and to reproduce the natural ageing. The study of the paints coat degradation evidences that UV exposure is one of the most aggressive constraints for paint binder. But results show also a strong effect of liquid water on paint degradation and particularly on pigments loss and blistering phenomenon. Whereas temperature in dry environment is not a very degrading factor for coats, presence of liquid water is a strong accelerating constraint at high temperature. Agreement between outdoor ageing and indoor ageing with a new procedure involving UV, temperature and rain also shows that liquid water is the critical constraint responsible to edge corrosion of silver layer and paint delamination. This work shows also that relation between paint degradation and corrosion of silver layer is not obvious. Homogeneous corrosion by pitting was observed without any deep chemical deterioration of the paint coat system.

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Keywords: CSP mirrors, lifetime, degradation, paint coat, FTIR ATR, artifial ageing.

1. Introduction

The improvement of mirrors lifetime is one of the levers for improving the installation cost of concentrated solar power plants (CSP). Mirrors represent about 10 % of this cost [1, 2] and have obviously a key role since they are in

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charge of sun rays concentration towards the receiver. The loss in mirror performance is directly linked to the decrease of plant productivity. The objective of this study is to better understand the mechanisms of mirror's degradation. The challenge is to set up indoor ageing tests which are representative of outdoor ageing. Until now, the effect of sun rays was the much studied and the irradiation tests are the typical ageing tests to estimate the lifetime of mirrors [2]. Water is often presented as a critical factor but its effect on mirrors degradation remains under study [3, 4]. In particular, the differences between humidity and liquid water constraint are not well explained. Solar power plants are located in desert sites: humidity is low and precipitations are rare. But, it was shown that condensation phenomena may appear [5]. Above all, as long as the cleaning of mirrors by water under pressure is the main procedure used, this factor cannot be neglected, the cleaning being made about one time a week in CSP plants.

A critical issue for durability studies is the trial period which can be very long. Results must be compared to those obtained in natural ageing to validate the relevance of the accelerated test. Additionally, mirror technologies are constantly evolving because of the changes of environmental standards (removal of lead and copper) or the improvement of mirror performances (reflectivity, shape, cost, etc.). In order to have a fast experience feedback, a model mirror is tested. This mirror is supposed to be used indoor. Evidently, ageing on solar mirrors are also performed. Based on FTIR-ATR spectroscopy, the study is dedicated firstly to the mechanisms of paints degradation according to the constraints like temperature, UV, humidity and liquid water. Independent and cross effects are highlighted. The main property of the solar mirror is the specular reflectivity thanks to silver layer. The second part of the study is focused on mechanisms of silver corrosion.

2. Experimental procedure

Three commercial glass mirrors detailed table 1 were studied: #1 is adapted to indoor use, whereas #2 and #3 are solar mirrors. The paint protection system is made by two or three kinds of paint coats called: primer, base coat and top coat. The mirrors are 7 squares centimeters and are cut from larger mirror; thus, they have no edge protection. Table 2 displays outdoor and indoor ageing tests performed.

Table 1: The different constituents of the mirrors

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	Clear glass	Reflective layer	Copper layer	primer	basecoat	Topcoat			
#1	yes	silver	no	alkyd	no	Alkyd			
#2	yes	silver	yes	alkyd	alkyd	solar white paint			
#3	yes	silver	no	alkyd	no	solar white paint			

Table 2: Ageing test procedure

Name	Constraints	Material and remarks
Ext1	Outdoor ageing : Alps climate (Le Bourget du Lac, France)	Bottom paint is never exposed to sun rays
Ext2	Outdoor ageing : Mediterranean climate (Cadarache, France)	Bottom paint is temporary exposed to sun rays
100°C	100°C constant (Humidity<40% RH)	Venticell
Н	Humid environment (gaseous state) 45°C, 95%RH	Vötsch VC0018
DH	Damp heat test (85°C/85%RH)	Vötsch VC0018
CLT	Condensation 45°C, 95%RH	Vötsch VC0018
LT	85°C, 65W/m ² between 300 and 400 nm.	SUNTEST XXL+ (ATLAS)
LTR	Constant irradiation at 65W/m ² between 300 and 400 nm; cycle of 27 min in dry	SUNTEST XXL+ (Test is based on standard
	environment followed by 3min rain. Temperature varies from 45 during rain	ISO 11341. Cycle duration was shortened to
	phase to 85°C during dry phase.	increase the number of rain phase.)

Mirror degradation is characterized by specular reflectance measurements using an UV-NIR-Vis Perkin Elmer Lambda 950 equipped with ARTA (Absolute Reflectance and Transmittance Analyzer) accessory [6]. It allows measurements at different incidence angles and at each wavelength on the whole solar spectrum from 280 nm to 2500 nm. Analysis is performed on 2×1 mm² surface on the glass mirror center. The percentage of solar reflectance % ρ_s is calculated at 8° using ASTM G173-03. The variation of % ρ_s is called Δ % ρ_s . Mirrors degradation is also analyzed by an Olympus camera and using a reflecting ZEISS AXIO Imager microscope. The characterization of paints is performed by Fourier Transform Infrared Spectroscopy (FTIR) between 500 and 4000 cm⁻¹ with a Nicolet 510P spectrometer equipped with an Attenuated Total Reflectance (ATR) Nicolet instrument. A ZnSe crystal (refractive index = 2.4) and a 45° incident angle of the IR beam were chosen in order to analyze the superficial area of the coatings (analyzed thickness of some micrometers). Paint is polished to perform FTIR ATR measurements on undercoat (primer and base coat).

3. Results and discussion

3.1. Paint used as protective coat for solar mirror glass

Paint is a composite material with a polymer binder and organic and inorganic pigments. In this study, the characterization of the paints is qualitative and is focused on the binder and the mineral pigments. The primer is basically an alkyd polymer as well as the base coat. The topcoat is either polyurethane or an acrylic paint and both technologies require a high content of anti-UV such as TiO₂ pigments. Acrylic and urethane copolymers may be used as binder by mirror manufacturers. FTIR-ATR analyses permit to define the type of the binder and the main pigments. All the pigments cannot be detected and the binder/pigment ratio is not measurable by this spectroscopy. However, IR spectroscopy is still a powerful and fast method to understand the effect of the outdoor constraints on the paint degradation. Infrared spectra of the mirror paint coats are displayed in Fig. 1. The main vibration bands associated at each coat are listed in table 3. Primer #1 #2 and #3, basecoat #2 and topcoat #1 are alkyd paint associated with melamine polymer (815 cm⁻¹, 1540 cm⁻¹). These paint contained also mineral pigments such as calcium carbonate, talc and barium sulfate. Topcoat #2 and #3 are mainly compounded by polyurethane binder, titanium oxide, talc and barium sulfate. The minerals pigments have mainly a spherical shape but silicate pigments such as talc are lamellar to extend the path between the exterior and the metallic layers.

Table 3: IR band assignments

Vibration band	Wavelength (cm ⁻¹)	Detection				
Binders						
C=O stretching	1720 [7, 8]	Every paint				
C=O stretch (biuret in polyurethane paint)	1686, 1638 [8]	Top coat #2 and #3				
N-H deformation and C-N stretching of melamine	1540[9]	Primer #1, #2 and #3; Basecoat #2; Top coat #1				
N-H deformation and C-N stretching of amide II	1520 [8]	Top coat #2 and #3				
C-H bending	1450, 1380 [7, 10]	Every paint				
C-O-C (asymmetric bending)	1247 [8, 10]	Every paint				
C-O and C-C stretching	1180, 1160, 1110, 1070 [7, 8]	Every paint				
Crosslinks between alkyd and melamine	815 [9]	Primer #1, #2 and #3; Basecoat #2; Top coat #1				
C-H rocking	740 [7]	Primer #1, #2 and #3; Basecoat #2; Top coat #1				
Mineral pigments						
Talc $(Mg_3(Si_4O_{10})(OH)_2)$	3674, 1013 et 668 [7, 9]	Every paint				
CaCO ₃	1400-1430, 875, 710 [7, 9]	Primer #1, #2 and #3; Basecoat #2				
BaSO ₄	1180, 1110, 1070, 980, 640 et 610	Top coat #1 #2 and #3; Primer #1 and #2				
	[7, 8, 9]					
TiO ₂	large band around 500-700 [9]	Top coat #2 and #3				

3.2. Chemical degradation of protective paints

Alkyd-melamine paint degradation (#1 mirror):

Paint degradations change drastically as a function of constraints exposure (Fig. 2). Results obtained by FTIR-ATR show that temperature does not affect the paint structure even after 5700h at 100°C (Fig. 2b). At low temperature (45°C) and with high humidity level (95% RH), the chemical structure of the paint does not change either (Fig. 2c). In the same conditions, but with liquid water (Fig. 2d), the paint binder remains unchanged but a depletion of pigments content is observed for CaCO₃ (1400 and 875 cm⁻¹) and BaSO₄ (980, 640 and 610 cm⁻¹).



Fig. 1: Spectra of paints obtained by FTIR-ATR between 500 and 2000cm⁻¹.

The absorption of water into the paint may favor migration of mineral pigments towards the surface. No reduction of talcum content was observed because these silicate pigments have a lamellar shape; they are consequently much less mobile than others.

At low temperature and at relatively short time (1000h), humidity has no effect on paint degradation but at high temperature (85° C), the paint is rapidly deteriorated (Fig. 2e). IR measurements after 1200h of DH test show binder degradation whereas the pigment contents are not affected. The decrease of intensity at 1720 cm⁻¹ (C=O) and the appearance of a new vibration band around 1620 cm⁻¹ are caused by the degradation of the polymer binder and the formation of hydrolysis products [11]. At the same time, the decrease of the band at 815 cm⁻¹ evidences the break of crosslink between alkyd and melamine. The ageing under irradiation with glass exposition to the light allows us to understand the combined effect of liquid water and temperature. The analysis was performed on the water retention area (Fig. 2f). This ageing induces a vibration band appearance around 1650 cm⁻¹ due to binder hydrolysis. But in addition to the polymer degradation, a loss in the CaCO₃ content is observed (1400, 875 and 710 cm⁻¹).



Fig. 2: Spectra of alkyd paint (mirror #1) obtained by FTIR-ATR between 500 and 2000 cm⁻¹ after different kind of ageing test (exp. means exposure). Arrow corresponds to a new band vibration caused by degradation products.

When paint is directly exposed to irradiation (LT), the degradation level is very high (Fig. 2g). The vibration bands of C=O bonds around 1720 cm⁻¹ disappear associated with the growth of a large band around 1650 cm⁻¹ caused by appearance of photo-oxidation products [8]. At the same time, the crosslinks between alkyd and melamine disappear also (815 cm⁻¹). Despite the strong degradation of polymer matrix, the pigments remain into the paint. In the presence of liquid water (LTR), the degradation is even more severe. A loss of pigments is associated to the degradation of the binder due to irradiation. It is clearly evidenced by IR spectra but also by the observation of chalking. The complete deterioration of the binder and the loss of pigments induce an erosion of the paint until a complete disappearance of the top coat. The degradation mechanism is very fast and it is enhanced by the spreading and flow of water on paint surface.

In order to correlate these artificial ageing tests to natural ageing, measurement was performed on samples exposed to Ext1 and Ext2. The paints are never exposed directly to sunlight on Alps site (Ext1). After two years of ageing, the binder remains unchanged whereas a loss of CaCO₃ pigments is observed (1400, 875 and 710 cm⁻¹) (Fig. 2i). These changes in paint composition are similar to the ones observed under CLT test except that only CaCO₃ pigments are lost. On Mediterranean site, the paints are occasionally exposed to irradiation and mirrors are subjected to water retention. IR measurements after two years of exposure show a loss of CaCO₃ pigments (1400, 875 and 710 cm⁻¹) and the appearance of degradation products with the increase of absorption around 1650 cm⁻¹ (Fig. 2j). The paint degradations are very similar to the ones after LTR test (when glass is directly exposed to light). But it is difficult to uncorrelated hydrolysis and photo-oxidation products. Consequently, irradiation effect cannot be excluded at this stage of the study.

White paints degradation (top coat - #2 #3 mirror):

Infrared measurements show that both white paints have similar behavior regarding climate constraints. Thus, only the results for mirror #2 are displayed in Fig. 3. White paints remain unchanged under temperature (100°C), humidity at low (H) and high temperature (DH) and when exposed to Ext1 natural ageing.

But paint is deteriorated as soon as liquid water is a constraint. At low temperature (CLT), there is only a decrease of absorption at 1070, 1110 and 1180 cm⁻¹ (Fig. 3f). The much probable hypothesis is a loss of BaSO₄ pigments. At high temperature with liquid water retention (LTR with glass exposed to light), there are a binder degradation and a loss of BaSO₄ pigments. The decrease of the C=O band at 1720 cm⁻¹ and the appearance of intense vibration at 1650 cm⁻¹ evidence hydrolysis of polymer binder (Fig. 3g).

The paints exposed to irradiation (LT) are deteriorated by photo oxidation mechanisms (Fig. 3h). The enlargement of the C=O band (1720 cm⁻¹) corresponds to this mechanism. The decrease of absorption at 1520 cm⁻¹ indicates a degradation of urethane bonds.



Fig. 3: Infrared spectra of top coat #2 obtained by FTIR-ATR between 800 and 2000cm⁻¹ after artificial ageing test.

One can also observe that the intensity at 1150 cm⁻¹ decreases whereas the vibration band at 1070, 1110 and 1180 cm⁻¹ remain unchanged after LT ageing. Thus, the BaSO₄ pigments are not affected by photo-ageing and the decrease at 1150cm⁻¹ is attributed to polymer degradation. When paints are exposed to continuous irradiation but with rain cycle (LTR) the same kinds of photo-oxidation are observed (Fig. 3i). However, the decrease of the absorption between 1100 and 1200 cm⁻¹ indicates a loss of BaSO₄ pigments due to spreading and flow of water.

As can be expected, Solar white paints are much more resistant to water stress factor than interior paint. But the degradation mechanisms are quite the same. White paints are deteriorated under irradiation exposure and when liquid water wets the paint surface at low or high temperature. It is interesting to notice that liquid water is one of the major degrading factors of the paints. There is always a loss of pigments when water wets the paint surface. Associated to the binder degradation caused by irradiation but also temperature, there is a strong degradation of paint with chalking.

3.3. Mirror degradation

The study of chemical degradation of the paints is insufficient to characterize all the degradations. In order to complete this study, the work is focused on silver corrosion and physical paint degradation with phenomena of delamination and blistering. The main degradations observed are displayed in table 4, 5 and 6. Three degradations have to be highlighted:

- Blistering
- Corrosion and delamination from the edges
- Homogeneous corrosion by pitting

Table 4: Summary of the main degradations of the top coat and the silver layer of mirror #1

Test	Chemical degradation of paint	Physical degradation of paint	Δ%ρ s 8°	Silver degradation
Ext1 (2 years)	Loss of mineral pigments	None	-3.0%	Homogeneous corrosion by pitting
Ext2 (2 years)	Loss of mineral pigments	Chalking	0%	Edge corrosion
	Hydrolysis of the binder	Delamination		
100°C (5700h)	none	None	-1.0%	Homogeneous corrosion by pitting
H (1000h)	none	None	0%	none
DH (1200h)	Hydrolysis of the binder	Punctual defects	0%	Punctual corrosion
CLT (1000h)	Loss of mineral pigments	None	0%	none
LT (2000h)	Photo oxidation of the binder	None	0%	none
LTR (1000h)	Loss of mineral pigments	Delamination	0%	Edge corrosion
Glass exposition	Hydrolysis of the binder			
LTR (1000h)	Loss of mineral pigments	Chalking - Paint erosion	0%	Edge corrosion
Paint exposition	Photo oxidation of the binder	Delamination		Punctual corrosion

Table 5: Summary of the main degradations of the top coat and the silver layer of mirror #2

Test	Chemical degradation of paint	Physical degradation of paint	Δ%ρ _s 8°	Silver degradation
Ext1 (2 years)	none	None	0%	none
Ext2 (2 years)	No measurement	None	0%	Edge corrosion
100°C (5700h)	none	None	-2.2%	Homogeneous corrosion by pitting
H (1000h)	none	None	0%	none
DH (1200h)	none	Punctual defects	-3.9%	Homogeneous corrosion by pitting
CLT (1000h)	Loss of mineral pigments	Blistering	0%	none
LT(2000h)	Photo oxidation of the binder	None	0%	none
LTR (1000h)	Loss of mineral pigments	Delamination	0%	Edge corrosion
Glass exposition	Hydrolysis of the binder	Blistering		-
LTR (1000h)	Loss of mineral pigments	Chalking	0%	none
Paint exposition	Photo oxidation of the binder	-		

Test	Chemical degradation of paint	Physical degradation of paint	$\Delta\%\rho_s 8^\circ$	Silver degradation
Ext1 (2 years)	none	None	0%	none
Ext2 (2 years)	No measurement	None	0%	none
100°C (5700h)	none	None	-1.5%	Homogeneous corrosion by pitting
H (1000h)	none	None	0%	none
DH (1200h)	none	Punctual defects	0%	none
CLT (1000h)	Loss of mineral pigments	None	0%	none
LT (2000h)	Photo oxidation of the binder	None	0%	none
LTR (1000h)	Loss of mineral pigments	Blistering	0%	none
Glass exposition	Hydrolysis of the binder	-		
LTR (1000h)	Loss of mineral pigments	Chalking	0%	none
Paint exposition	Photo oxidation of the binder	-		



Fig. 4: Blisters formation on topcoat #2 after 1000h of CLT test.

Blistering:

Blister formation appears on white paints exposed to water retention at low (CLT) or high (LTR) temperature. For both temperatures the phenomenon appears very fast before 100h of test. Blisters are formed at the interface between top layer and the paint layer below (base coat or primer). Some of the blisters burst and let appear the surface below. It is the case after 1000h of condensation test where the burst blisters are grey (Fig. 4). The blister formation is caused by liquid water absorption. No corrosion was observed on silver layer and blisters appear between the paints coat. Blistering is not linked to punctual corrosion of the metallic layer which is the major cause of blistering phenomena mentioned in the literature [12]. Water absorption may locally increase the volume of the paint which may form irreversible deformations.

At the stage of this study, the test duration is too short to induce metallic corrosion. But the degradation of the top coat induces weakness area located at the burst blister. The alkyd paint exposed directly to the environment should be degraded more quickly according to the previous part on chemical ageing of paints.

Corrosion - delamination from the edges

Silver corrosion starts from the edges of the mirrors and may go along with delamination of the paint system. These phenomena were firstly observed in natural ageing Ext2 (Fig. 5a). Two kinds of degradation were observed depending on the fixation system of the mirrors. Under the fixation of the mirror there is corrosion from the edge but no delamination. On the other hand, the corrosion from the edge goes along with delamination. These phenomena happen only with water retention on the paint and when ageing induces temperature and wetting/drying cycles. From this fact observed in Ext2 site (Fig. 5a), we develop a new artificial test with alternation of dry phase and rain phase. This test called LTR in this study is based on the standard ISO 11341.

Optical micrographs Fig. 5b and 6 after LTR ageing for mirror #1 evidence the silver corrosion and the delamination of the paints system. The degradations are in really good agreement with what occurs in outdoor ageing Ext2. This confirms assumptions that water retention associated with temperature cycle are the critical constraints responsible to silver corrosion and paint delamination. Mirror #2 is also subject to these phenomena (Fig. 7). Silver corrosion is observed in Ext2 ageing whereas silver corrosion and delamination is already observed on LTR after 1300h of ageing. Mirror #3 is not corroded at all in Ext2 and in LTR test. This is another fact which confirms the relevance of the LTR test. All the mirrors do not have edge protection by paint, polymer or scotch because samples are cut from larger mirror. The well edge resistance of mirror #3 shows that a better edge protection can be guarantee by the mirror technology itself without applying an edge protection system.

Results show that the degradation mechanism from the mirror edge starts with the silver corrosion due to water retention. This corrosion decreases the adhesion between the metallic layer and the paints system. The paint degradation (Fig. 2) induces modifications of chemical properties of paints such as wettability and water absorption. Physical properties like coefficient of thermal extension or tensile and compression strength may also be affected. Temperature changes and cycle of humid and dry phases induce strains which cause delamination of the paint when corrosion of metallic has already begun.



Fig. 5: Photographs of the mirror #1 degradation after 2 years Outdoor (Ext 2) and after artificial ageing (LTR).



Fig. 6: Optical micrograph of corrosion of the silver layer and delamination of paint system after 450h of artificial ageing LTR.

LTR 1300h				Ext2 (2years)			
	Bottom surface		Front surface			Front surface	
							-

Fig. 7: Photographs of the mirror #2 degradation after Outdoor (Ext 2) and artificial ageing (LTR).

Homogeneous corrosion of the silver layer

The homogenous degradation of silver layer is a whole corrosion of silver by pitting (Fig. 8). Results displayed at tables 4, 5 and 6 show that homogeneous corrosion of silver layer happens after some thousands of hours at 100°C for all the mirrors, after two years of Ext1 ageing for mirror #1 and after only 100h of damp heat test for mirror #2. Except for mirror #1 subjected to Ext1 ageing, the silver corrosion is not associated with a chemical degradation of the paint.

Ageing at 100°C or damp heat test for mirror #2 does not evidence paint degradation according to infrared measurement (Fig. 3). No liquid water participates to these mechanisms; the degradation is caused by oxygen and humidity. We may suppose that temperature induces an increase of the free volume of the paint which favors the diffusion of gas through the paint to reach the metallic layer. During damp heat test, the partial pressure of water is very high. That's why the corrosion of the mirror #2 under damp heat test is much faster than the corrosion at 100°C.

The homogeneous degradation of model mirror (#1) after two years in Ext1 can be attributed to the loss of mineral pigments which favors the diffusion of corrosive species. This kind of degradation is not observed for mirrors exposed in Ext2. The higher humidity level and rain frequency at the Ext1 site may cause this global degradation. It is interesting to notice also that one part of the mirror is not exposed to solar irradiation due to fixation mechanism. The silver area below this fixation mechanism is not corroded. These results suggest that UV rays catalyze chemical reaction of corrosion. Authors had already shown that UV rays above 450 nm are partially transmitted through silver layer and can degrade silvered PMMA mirrors [13]. On one hand, transmitted UV can deteriorate the paint and on the other hand they can catalyze chemical reaction with corrosive species. We need to have more feedback on solar mirror technology to confirm this fact but this result suggests that mirrors have to be exposed to UV in artificial ageing test in order to evaluate their lifetime. However, one has to remember that model mirror #1 have no copper layer between silver layer and paint coat. As long as copper layer or any kinds of anti-UV coats remain unchanged, this kind of degradation should not occur.



Fig. 8: Optical micrograph of silver layer after 2 year in Ext1.

4. Conclusion

The effect of environment constraints on mirrors paint coat degradation has been identified. Direct exposure to sunlight and synergy between temperature and liquid water induce polymer binder degradation. Loss in pigments has also been observed when liquid water wets mirror paint. The calcium carbonate is the most mobile pigment whereas lamellar silicates are very stuck to the coat. The depletion of talc by chalking was only identified when polymer matrix is highly deteriorated. Liquid water affects not only the protective paint but also the silver layer by initiating its corrosion. Besides, the artificial ageing with cycle of rain and temperature modified from standard ISO 11341 enables to reproduce the effect of corrosion and delamination observed in outdoor ageing.

The whole degradation of the silver layer by pitting is not necessary associated to high paint degradation according to FTIR measurement even if some minor degradation was observed by colorimetric measurement [14, 15]. This phenomenon is obviously caused by diffusion of corrosive species through the paint. The diffusion is favored by defects or changes in binder properties during thermal ageing. The defects may occur during the mirror

process or during the ageing such as loss of pigments or micro-hole. Thermal ageing may induce an increase of free volume of the paints and an increase of diffusion coefficient of corrosive species.

Infrared measurement by FTIR ATR method is a good tool to characterize the degradation of the protective paint coats. It allows us to understand the main constraints and to correlate outdoor and artificial ageing. But, this study evidences also that it is not sufficient to understand corrosion of the reflective layer. The characterization of paint properties at the temperature of ageing is necessary in order to understand all the mechanisms.

This work requires much time of outdoor and artificial ageing to better understand mirror ageing but gives tools to understand the various kinds of degradation observed outdoor. The outdoor ageing established in desert area will give us better references to understand the durability of mirror for CSP application [16-18].

Acknowledgements

This work is supported by a public grant overseen by the French National Research Agency (ANR) as part of the "Investissements d'Avenir" program (reference: n° ANR-11-EQPX-0014).

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