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Standardizing Accelerated Aging Testing Conditions for Silvered-Glass Reflectors

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Abstract. To assure the quality of accelerated aging tests for solar concentrators, their standardization is crucial. It guarantees the employment of adequate testing, measurement and characterization procedures and the comparability between results. A committee of the Spanish AENOR standardization agency is working on the draft “Reflector Panels for Concentrating Solar Technologies”. This work focuses on the evaluation of the procedures defined in this standard. The reflector material from the Japanese company Nishio Glass Mirror Co., which showed severe degradation after 7 years of outdoor exposure in Abu Dhabi, is tested according to the AENOR standard to check if its poor outdoor performance could have been predicted by accelerated aging testing. However, after completion of the accelerated tests, in some cases even for considerably longer test durations than the minimum required by AENOR, no considerable degradation was detected. The results suggest that the proposed testing program by AENOR is not aggressive enough to identify material failure. Ways to improve the current standard are proposed through development of more realistic tests.

INTRODUCTION

Durability testing of mirrors for concentrated solar power (CSP) plants using accelerated aging has two main purposes: first, quality control for the mirror manufacturers and their production process and second, estimation of lifetime of the components for plant development companies in a reasonable time. So far, no consistent standard exists that is used and accepted throughout the CSP industry. Standardization of testing is of utmost importance to select adequate procedures and to make test results more comparable and reproducible. The Spanish AENOR committee is working on a standard for mirror testing [1] in the framework of the standardization sub-committee AEN/CTN 206/SC. AENOR adopted the parameters and minimum duration of testing from other industries and previous experience by manufacturers but the standard does not contain any pass/fail criteria.

One critical point in the development and improvement of accelerated aging test is the availability of representative outdoor data. Analyses have to be done on material that was exposed to realistic conditions for a reasonable time. Exposure durations of a minimum of five to ten years are desirable. This is especially important for silvered glass mirrors, because in the first years of exposure only minimal degradation is detected [2]. At the same time, non-aged material of the same manufacturing batch (e.g. by storing samples indoors) has to be available for the accelerated tests to permit a comparison between outdoor and indoor degradation results.

In this work, results of the Nishio reflector material which was exposed during seven years at the Masdar Institute Solar Platform (MISP) in Abu Dhabi are compared to lab testing of the same material from the same batch in new state, referred to as “as-received”. The samples used for accelerated aging have been stored in a warehouse during 7 years and were hence protected from aggressive environmental stresses. The analysis aims to improve the

proposed testing parameters by AENOR towards a more representative standard which correlates better to outdoor exposure.

METHODOLOGY

To evaluate the proposed parameters of the AENOR tests, results from outdoor exposure and accelerated laboratory tests are compared. To obtain outdoor reference data, samples were collected from the MISP, where 33 heliostats were installed in a Beam-Down Tower demonstration plant in July 2009. The reflector material is a 5 mm-thick silvered-glass mirror manufactured by Nishio Glass Mirror Co. The backside coating is composed of a silver layer, a copper-containing primary paint, a secondary paint, and a transparent protective resin covering the edges as well as the back surface. The material presents no protective copper layer, usually acting as sacrificial anode and protection for the coatings from the transmitted UV light through the silver layer. For more details on the material see [3].

After seven years of exposure on site, all of the 1419 mirror facets showed some level of degradation. On many, the corrosion of the silver layer affects a considerable portion of the mirror surface. One of the facets was collected for analysis in the OPAC laboratory at the Plataforma Solar de Almería (PSA). The degradation is detected with optical microscopy and the loss in reflectance is measured. Facets from the same manufacturing batch as the ones exposed on the heliostats were also stored in a warehouse and thus protected from aggressive environmental conditions. These as-received samples were subjected to the accelerated tests proposed in the current version of the AENOR standard.

If the parameters of the standard are correctly chosen to represent a realistic outdoor environment, the material should suffer degradation in the AENOR accelerated aging tests.

The following instruments are used to quantify the solar reflector optical degradation in the test campaign:

- A Perkin-Elmer Lambda1050 spectrophotometer with an integrating sphere of 150 mm diameter is used to measure the hemispherical reflectance in the wavelength range of $\lambda = [280,2500]$ nm, using 5 nm intervals at an incidence angle of $\theta_i = 8^\circ$. The data were evaluated with a 2nd surface reference reflectance standard (calibrated in the range 280-2500 nm). The hemispherical reflectance spectra are weighted with the solar spectrum from the ASTM G173-03 standard to obtain the solar-weighted hemispherical reflectance, $\rho_{s,h}([280,2500]nm; 8^\circ; h)$.
- A portable specular reflectometer model 15R-USB, manufactured by Devices and Services. This instrument measures monochromatic specular reflectance with an incidence angle of 15° and in a wavelength range between 635 and 685 nm, with a peak at 660 nm. The measurements were taken with an acceptance angle of 12.5 mrad. The nomenclature used for this parameter is $\rho_{\lambda,\varphi}(660\text{ nm}; 15^\circ; 12.5\text{ mrad})$. Reflectance measurements are taken before, during and after testing to evaluate the optical quality of the different materials.
- A 3D light microscope model Axio CSM 700, manufactured by Zeiss. This microscope allows 5, 10, 50 and 100 times magnification and also measures roughness, defect features (size and depth) and surface profiles. Microscopic pictures are taken during and after the completion of certain tests to study the appearance and evolution of the defects.

A relatively small warehouse facet (ca. 50x20 cm²) was available for the accelerated tests. 10 Samples were cut from this facet with dimensions of roughly 10x10 cm². Two samples were used per test. The edges of the samples that were cut in the laboratory were protected with insulating tape, as these edges pose an unrealistic attack point for degradation and detachment of the protective resin was observed. All samples include at least one original edge with the protective system as delivered, which was not covered with a tape.

There are three standard parameters that are measured to evaluate the degradation of samples: the reflectance difference, the formation of corrosion spots in the silver layer that exceed 200 μm in diameter and the maximum penetration of the edge corrosion into the surface in mm.

The tests that are included in the AENOR standard are presented in Tab. 1 together with their corresponding minimum testing durations and parameters. For more details on testing procedures see the corresponding standards.

TABLE 1. Standard tests proposed by AENOR with durations and main parameters.

Test	Minimum duration	Summary of testing conditions
Neutral Salt Spray (NSS) ISO 9227 [4]	480 h	T: $35 \pm 2^\circ\text{C}$, pH: 6.5 to 7.2 at 25°C Sprayed NaCl solution of 50 ± 5 g/l with condensation rate of 1.5 ± 0.5 ml/h on a surface of 80 cm^2
Copper-accelerated acetic acid salt spray (CASS) ISO 9227	120 h	T: $50 \pm 2^\circ\text{C}$, pH: 3.1 to 3.3 at 25°C Sprayed NaCl solution of 50 ± 5 g/l and 0.26 ± 0.02 g/l CuCl_2 Condensation rate of 1.5 ± 0.5 ml/h on a surface of 80 cm^2
Condensation ISO 6270-2 [5]	480 h	T: $40 \pm 3^\circ\text{C}$ RH: 100%
Combined thermal cycling and humidity	10 cycles	4 h at 85°C , 4 h at -40°C , Method A: 16 h at T: 40°C and $97 \pm 3\%$ RH, Method B: 16 h at T: 85°C and $85 \pm 3\%$ RH or 40 h at T: 65°C and $85 \pm 3\%$ RH
UV/Humidity ISO 16474-3 [6]	2000 h	1 cycle: 4h at UV exposure at $60 \pm 3^\circ\text{C}$ followed by 4h at 100% RH at $50 \pm 3^\circ\text{C}$

The laboratory weathering chambers employed to perform the accelerated aging tests are listed below:

- Salt spray chambers: These chambers perform tests according to the ISO-9227 standard [4]. The chamber model VSC450 by Vötsch is used for copper accelerated salt spray testing (CASS), while an Erichsen chamber model 608 is used to carry out the neutral salt spray (NSS) test.
- A humidity chamber from the company Ineltec is used to perform the Condensation test, according to the ISO 6720-2 standard [5].
- The SC 340 MH chamber by ATLAS is employed to perform weathering tests involving the control of temperature and humidity, such as the thermal cycling test. For this work method A was performed.
- The UV-Test chamber by ATLAS is utilized to perform the UV/Humidity test, according to the ISO 16474-3 standard [6].

RESULTS AND DISCUSSION

In the results mainly the degradation during outdoor exposure and accelerated aging are evaluated. A meaningful accelerated test should be able to reproduce the degradation happening in the field.

Outdoor Facet

The facet that was analyzed in the laboratory was previously exposed for more than 7 years and presents strong corrosion affecting a considerable part of the facet surface. Microscopic analysis (Fig. 1) shows a degradation pattern similar to the one found in previous studies [7].

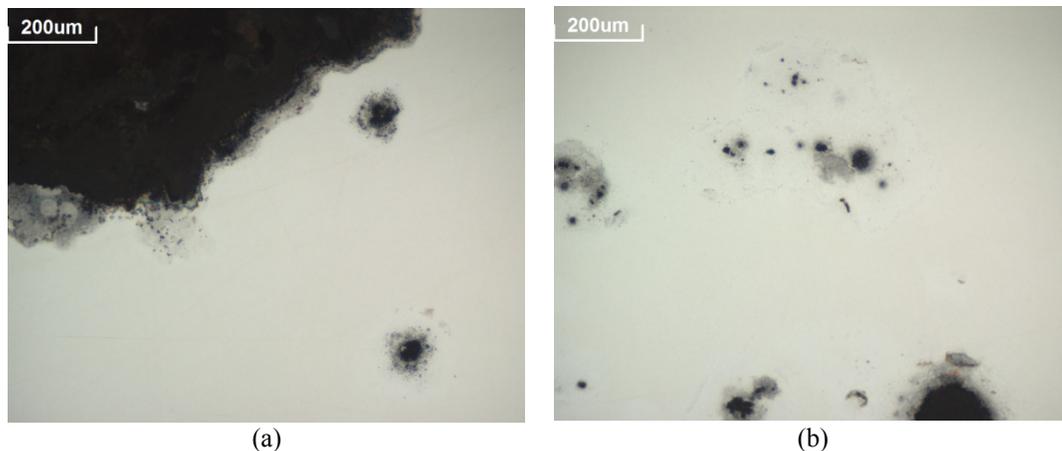


FIGURE 1. Microscopic images of silver layer corrosion after outdoor exposure in Abu Dhabi for 7 years

As there is no commercial instrument to measure the reflectance of the whole surface, a combination of different techniques is used to estimate the loss in reflectance and the affected area. An image processing software was used to analyze a photo of the facet that was taken with a white homogeneous background, see Fig. 2 (a). A brightness threshold was chosen to identify the optically affected areas and a degraded area of 14.1 % of the entire surface was calculated, see Fig. 2 (b).

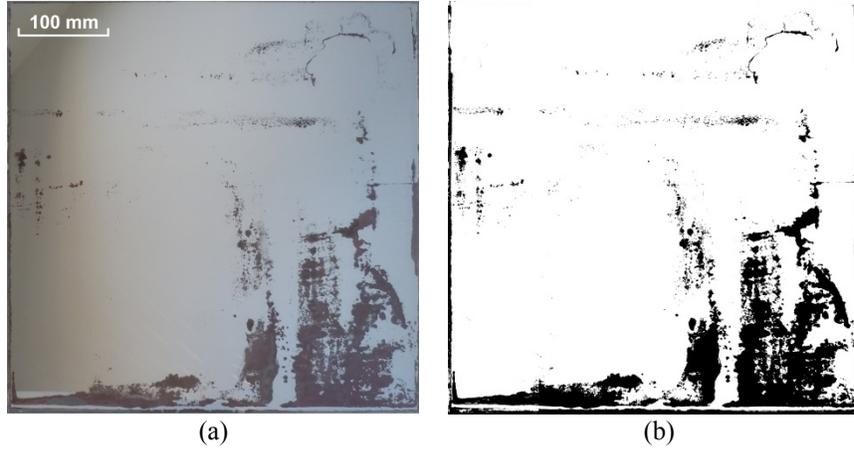


FIGURE 2. (a) Photograph of the degraded facet, (b) Analysis of degraded area fraction (black: degraded, white: undegraded)

Specular reflectance measurements were taken with the D&S on degraded and undegraded areas. For the undegraded areas, spots were chosen where no visual change on the reflective surface could be identified. Measurements were taken on 13 spots and the average value is 0.838 with a standard deviation of 0.003, indicating that the values are homogeneous.

To measure the reflectance on degraded areas, some spots were chosen where the complete measurement area appears to be affected by degradation. This measurement involves a high uncertainty as the evaluation if a spot is degraded in the whole measurement area depends on the operator and handling of the equipment. Seven measurements were taken with an average of 0.103 and a standard deviation of 0.049. The values vary strongly and range from 0.055 up to 0.198.

The average reflectance of the facet can be estimated weighting the reflectance values of the degraded and non-degraded parts with their corresponding area fractions:

$$\rho_{tot} = a_{non} * \rho_{non} + a_{deg} * \rho_{deg} = 0.859 * 0.838 + 0.141 * 0.103 = 0.734 \quad (1)$$

Where ρ_{tot} is the total reflectance of the whole surface, ρ_{non} is the reflectance of the non-degraded area (a_{non}) and ρ_{deg} is the reflectance of the degraded area (a_{deg}).

The measurements of the as-received warehouse facet showed an initial reflectance of 0.846 with a standard deviation of 0.001, so it can be stated that the outdoor facet lost about 0.112.

A deeper study of the corrosion mechanisms of the outdoor facets is presented in [3]. In the latter study, the facets coatings structure is analyzed and possible causes for the strong degradation are presented. The main possible factors are: 1) weak adhesion and manufacturing defects in reflecting layers, 2) weak protective top coating, especially against UV, 3) absence of copper layer, 4) initial presence of corrosive contaminants, chlorine and sulfur, remaining from the manufacturing process, 5) low mechanical stability of top coating especially at the edges.

Accelerated Aging Tests

The main result of the accelerated tests is that no considerable degradation of the silver layer takes place after the completion of the tests proposed in the AENOR standard. In Tab. 2 the reflectance losses are displayed for the tests at different durations. For the tests in which the appearance of corrosion was expected, also testing durations significantly higher than the ones proposed in the standard were conducted. Even with these parameters the fatal degradation detected outdoors could not be reproduced. Effectively, no degradation in the silver layer can be appreciated without microscopic techniques. For details of the individual test results see the following sections.

TABLE 2. Loss of solar-weighted hemispherical and monochromatic specular reflectance after test completion.

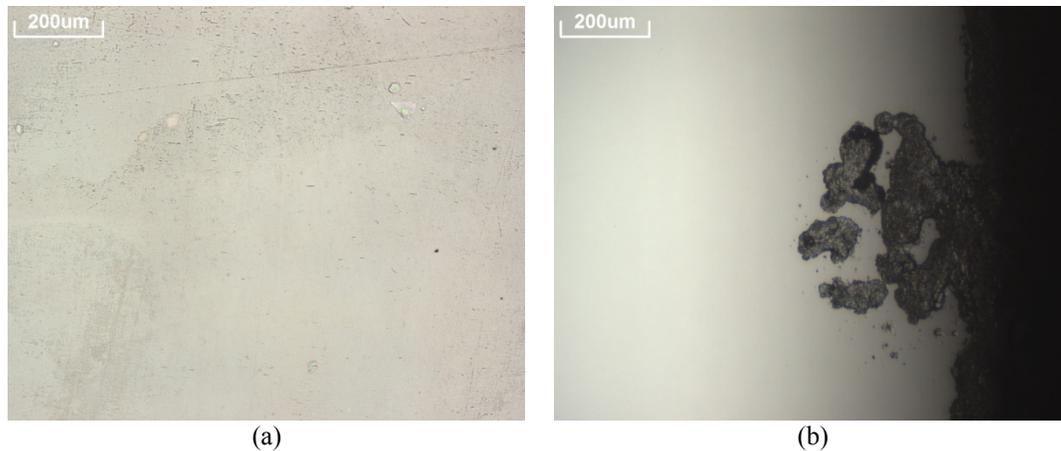
* Minimum duration suggested by the AENOR standard

Test	Duration (h)	$\rho_{s,h}(280,2500\text{nm},8^\circ,h)$	$\rho_{\lambda,\varphi}(660\text{nm},15^\circ,12.5\text{mrad})$
NSS	480*	-0.004	-0.010
	1000	-0.004	-0.014
	1550	-0.007	-0.034
	2000	-0.010	-0.045
CASS	120*	-0.001	-0.002
	480	-0.001	0.000
Condensation	480*	-0.001	-0.003
	1000	-0.001	-0.001
	2000	-0.001	-0.002
Thermocycles	10 cycles*	-0.001	-0.001
UV/Humidity	2000*	0.003	0.000
	3000	0.002	0.002

NSS Test

The NSS test is the standard test for corrosion evaluation. It was conducted for a total time of 2000 h which is more than four times the minimum duration proposed in the AENOR standard. The NSS test provokes the highest reflectance drop of all conducted tests. Specular reflectance drop is 0.010 for 480 h and 0.045 for 2000 h. This drop is not due to changes in the silver layer but mainly to an effect called glass corrosion. It is known that the glass surface of a mirror can get affected when exposed to high humidity for a long time [7]. This effect was not detected after outdoor exposure.

This glass corrosion is displayed in Fig. 3 (a). Slight edge corrosion was detected after 480 h which stays in the range of several micrometers even after 2000 h. In Fig. 3 (b) the only point where the edge corrosion reaches a penetration of about 0.5 mm is displayed.

**FIGURE 3.** Microscopic images of sample after 2000 h of NSS test (a) Glass corrosion, (b) Edge corrosion in the silver layer*CASS Test*

The CASS test is an extremely aggressive corrosion test. The reflectance of the samples in the CASS test remains stable even after 480h, four times the duration proposed in AENOR. After 120 h of testing, very few small corrosion spots (less than 100 µm in diameter) appeared, see Fig. 4 (a). The number of spots grew slightly but no growth in size of existing spots was detected. Beginning edge corrosion can be appreciated after 120 h, see Fig. 4 (b), which remains far below one millimeter even after 480 h of testing.

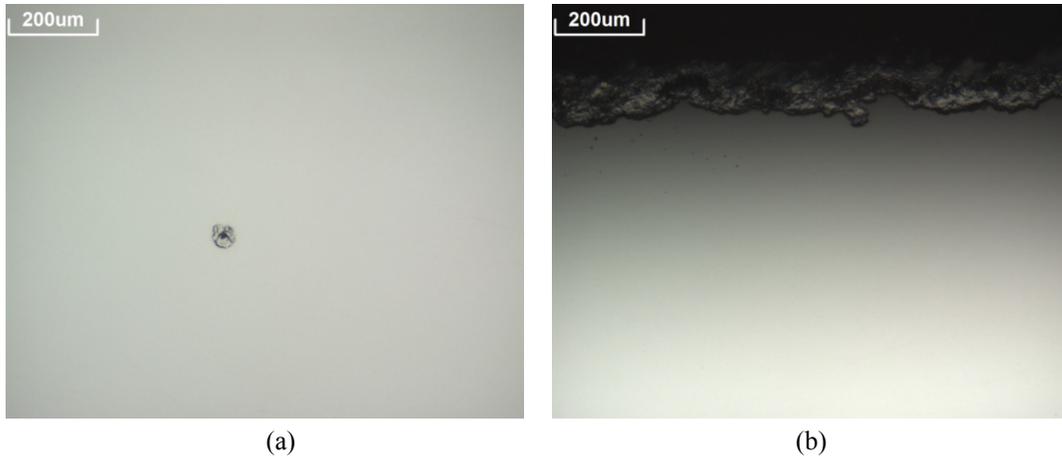


FIGURE 4. Microscopic images of sample after 120 h of CASS test (a) Corrosion spot, (b) Beginning edge corrosion

Condensation Test

The reflectance of the samples exposed to the Condensation test remains stable. No degradation of the silver layer was detected even after 2000 h, over four times the duration proposed in the AENOR standard.

Thermocycle Test

This test usually provokes mechanical stresses in the samples. Thus it could be interesting as the first step of a series of tests to form attack points for corrosion by cracking or delamination of protective coatings. In this case, no degradation was found after completion of the 10 thermocycles.

UV/Humidity Test

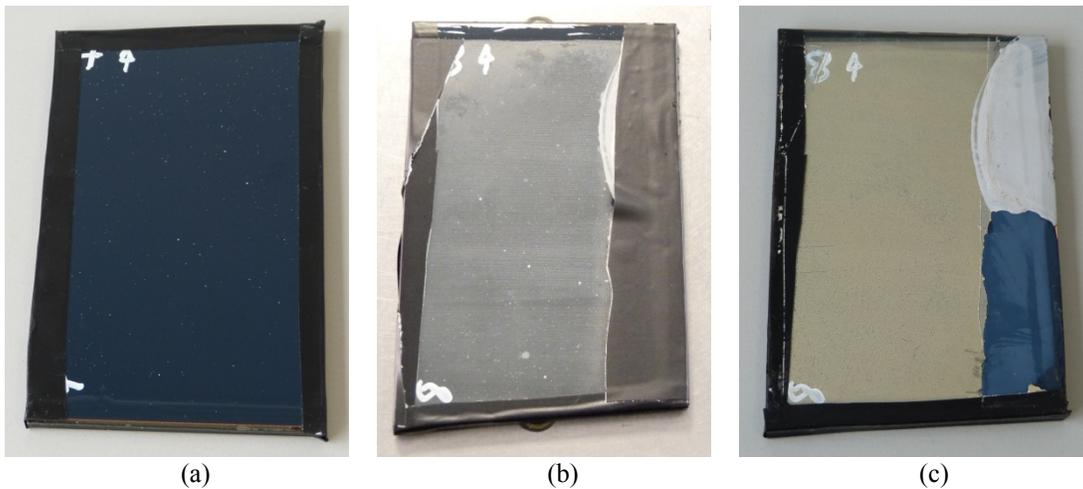


FIGURE 5. Backside of samples after UV/humidity test, (a) sample placed in chamber with backside facing out, after 3000 h (b) Sample with backside facing in, after 350 h (c) Sample with backside facing in, after 3000 h

In both samples that underwent the test, there was no considerable reflectance drop. One of the samples was placed in the test chamber with its backside facing the interior of the chamber, thus being exposed to the UV radiation and condensation directly, while the other had the backside facing out. The first one displayed the most striking result. The color of the backside coating turned from a dark blue to a lighter beige color, an effect not detected after outdoor exposure.

Figure 5 (a) shows the backside of the sample which was placed backside facing out after 3000 h. It shows no appreciable change compared to the initial state. In Fig. 5 (b) and (c), the backside of the other sample can be seen after 350 h and after 3000 h. An evolution of the degradation is visible with ongoing testing time.

In Fig. 6 (a) three main parts of the back surface of the sample with the degraded back can be distinguished. First in black the applied protective tape at the edge. Second, in blue the part where the tape was removed which resulted in the complete removal of the protective resin. Here the underlying original color is still visible. Third, the exposed part where the former transparent resin appears to be degraded to a beige color. Microscopic analysis shows that not only the color of this layer changed but also cracks and fissures spread over the entire surface, see Fig. 6 (b).

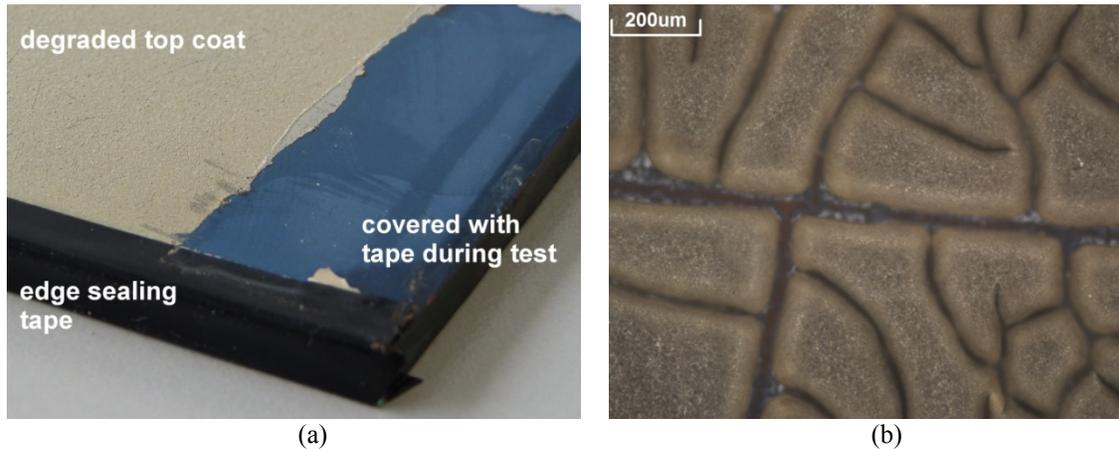


FIGURE 6. Backside of sample placed in chamber with backside facing in, after 3000 h UV/humidity test, (a) Photograph, (b) Microscopic image

Microscopic analysis of both samples shows that extremely small degradation spots appeared and spread out over the silver layer. At the edges bigger spots with a higher density are forming. After 3000 h of testing, the degradation does not reach a level that can be observed macroscopically. For microscopic pictures after 3000 h see Fig. 7 (a) for surface spots and (b) for edge degradation.

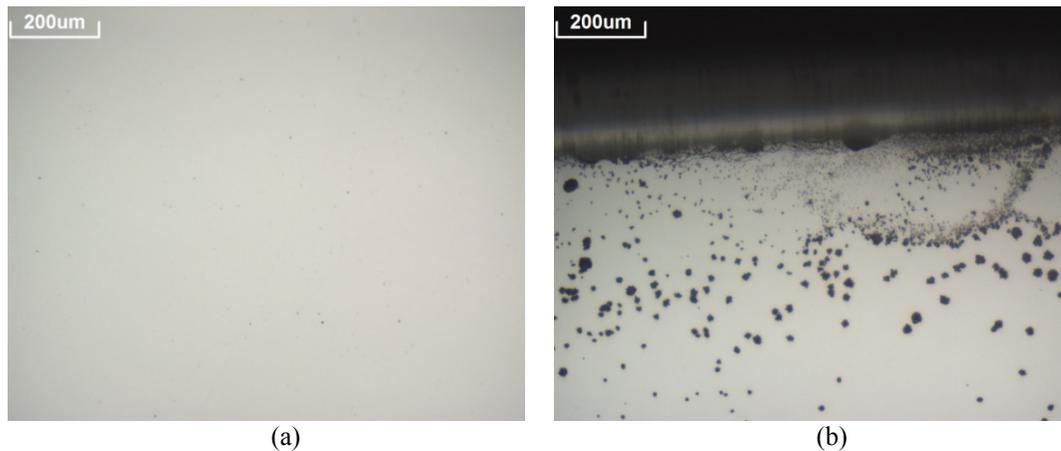


FIGURE 7. Microscopic images after 3000 h of UV/humidity test, (a) Small degradation spots on the surface, (b) Beginning of very slight edge corrosion.

CONCLUSIONS

In the AENOR standard for testing of reflector materials, a series of accelerated laboratory tests is proposed together with their corresponding minimum testing times. The proposed tests and durations are adopted from other industries and based on experiences by manufacturers and researchers. As a minimum requirement, their results

should give an indication about the quality of the tested reflectors and allow separating high from low quality materials. The work presented here concentrates on the evaluation of the testing program to fulfill this purpose.

After completion of these accelerated tests on the Nishio reflector material which has shown severe degradation outdoors, nearly no degradation was provoked. After seven years of outdoor exposure in Abu Dhabi, the Nishio reflectors showed corrosion of considerable areas of the silver layer and layer detachment accompanied with a drop of specular reflectance of more than 10 percentage points. However, in accelerated testing only very limited degradation was detected even after test durations considerably longer than the minimum ones proposed by AENOR. Changes regarding degradation spots, edge corrosion and reflectance drop are almost negligible. Reflectance losses have only been observed during Salt Spray testing as a result of glass corrosion exposed to high humidity rather than due to corrosion of the silver layer. This effect is not detected after outdoor exposure, which is also the case for the strong degradation of the back paint in the UV/Humidity test.

This leads to the conclusion that the proposed tests and parameters in the current version of the AENOR standard have to be improved. As the simple increase of the test durations does not provoke a considerably stronger degradation the most promising approach is to choose more complex tests that combine more environmental stresses and thus simulate outdoor conditions in a more realistic way. This could include cyclic testing or procedures which combine a series of tests. As the UV/Humidity tests provoked a damage of the protective backside coatings, future investigations will focus on this effect and include tests that combine these stresses with corrosion stresses as used in the NSS and CASS tests. Also, other stress factors such as abrasion due to sand and dust as well as corrosion due to chemical pollutants must be considered.

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