ECSS-Q-ST-70-17C :
A NEW ECCS STANDARD FOR DURABILITY TESTING OF COATINGS

Adrian P. Tighe(1), Christian Durin(2), Christine Amar(3), Christopher Semprimoschnig(4), Elisabeth Renard(5), Jean-Marc Agullo(6), Leonardo Chicarella(7), Marika Orlandi(8), Markus Glier(9)

(1) Adrian.Tighe@esa.int, ESA ESTEC, P.O. Box 299, 2200 AG, Noordwijk (ZH), NL
(2) Christian.Durin@cnes.fr, Service Technologies, Matériaux et Procédés, Sous-Direction Assurance Qualité , Centre National d'Etudes Spatiales 18, avenue Edouard Belin
(3) Christine.amar@thalesaleniaspace.com, Thales Alenia Space, Toulouse
(4) Christopher.Semprimoschnig@esa.int, ESA ESTEC,.PO. Box 299, 2200 AG, Noordwijk
(5) Elisabeth.renard@airbus.com, Airbus Defence and Space, Qualité - Technologies – Matériaux, Poste 89 46, Toulouse, France
(6) Jean-marc.agullo@airbus.com, Airbus Defence and Space, Qualité - Technologies – Matériaux, Poste 89 46, Toulouse, France
(7) leochicarella@gmail.com
(8) Marika.orlandi@esa.int, ESA ESTEC,.PO. Box 299, 2200 AG, Noordwijk
(9) Markus.glier@ohb.de, Optical Systems, OHB System AG, Manfred-Fuchs-Str. 1, D-82234 Weßling / Operpfaffenhofen, Germany

ABSTRACT

A new ECSS standard was published in February 2018 which captures the best practice across the large range of existing national and international standards, in order to define requirements for coating use in space applications. This includes a minimum set of tests required for coating evaluation, qualification and production, as well as providing information about tests which may be required for specific mission scenarios (e.g. air-vacuum effects, thermal endurance, atomic oxygen, VUV, radiation, laser induced damage and contamination induced effects).

The paper describes the motivation behind the standard, providing examples of "lessons learnt" from previous space projects and typical pitfalls which can be encountered during coating qualifications. The minimum set of required tests and methods for coating qualification are summarised, and a general overview of the various mission specific tests is provided. The concept of the coating qualification model is also be discussed. This is required when simple flat samples are not fully representative (e.g. for curved surfaces, sharp corners or edge masks).

Results of coating test campaigns from some recent space projects are presented, in order to highlight the need of testing using representative configuration (Test-As-You-Fly), particularly with respect to the substrate used. For example, during high temperature thermal endurance testing of white coatings for the BepiColombo project it was observed that copper from the aluminium Al2219 substrate gradually started to diffuse into the white coating after prolonged periods at high temperature, and this affected the optical performance of the coating. In another example, we show that degradation of a mirror coating during high energy proton testing was due to interaction of the protons with the substrate and not a degradation of the coating itself.

General requirements for ensuring maintenance of coating qualification are also discussed, as well as potential factors to consider in assessing the need for delta qualification or re-qualification (e.g. change of coating materials and processes or modification of coating equipment). We show that a standardised approach to testing is particularly important for recurrent missions, when nominally identical coatings need to be re-manufactured after a gap of several years.

1. INTRODUCTION

1.1 Motivation and objectives

Many different environmental factors can have an effect on coating durability. This includes in-orbit effects such as UV, particle radiation, atomic oxygen, thermal cycling contamination and orbital debris, as well as ground based effects, such as cleaning, contamination and humidity [3].

Many different standards exist for durability testing of coatings, mostly for ground based applications. Historically, space projects have been free to choose their own test requirements, based on a combination of existing standards and specific requirements for a given project. This approach can lead to major discrepancies between projects and leads to misleading definitions about when a coating is "space qualified". The supplier

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and customer often needs to re-negotiate very general aspects of coating qualification for each new project. The key motivation behind the new ECSS-Q-ST-70-17C standard is to standardise the qualification approach for the most commonly used classes of coatings for space applications, as well as to provide guidelines for the different types of test [1]. The specific objectives are to :

- Capture the best industry practice across the large range of existing national and international standards
- Define generic test criteria for standard testing of each class of coating (e.g. sample lot size, substrate requirements, standard tests to be performed for qualification and lot acceptance)
- Define test guidelines to verify use of specific coating technologies (e.g. porous coatings, very thin or fragile coatings, coatings on substrates with unusual geometry)
- Define a minimum set of durability tests for new coating technology developments
- Define test criteria for non-standard situations (e.g. repair of flight hardware or re-verification of supplier/process equipment)
- Define requirements for delta qualification, or re-qualification (e.g. after process change, design change)

1.2 Coating classification and applications

There are many different types of coating, which are used in a huge range of different space applications. It is not claimed that the standard will be relevant for all coating types, but the intention is to cover the most commonly used coatings. Three categories of coating are defined :

- Thin film optical coatings
- Thermo-optical and thermal control coatings
- Other metallic coatings

This list was derived after an extensive review by the ECSS working group of materials and processes lists from previous projects, to identify the most commonly used coatings. Of course, there will always be some ambiguities for certain coatings about which category to apply. For example, is a metallic mirror a “thin film optical coating”, or “other metallic coating” ? Or is a thin film black coating for stray light reduction an “optical coating” or a “ thermo-optical coating” ? It is simply not possible to standardise the approach for all coating types, and good engineering judgement also needs to be applied when classifying the coating type.

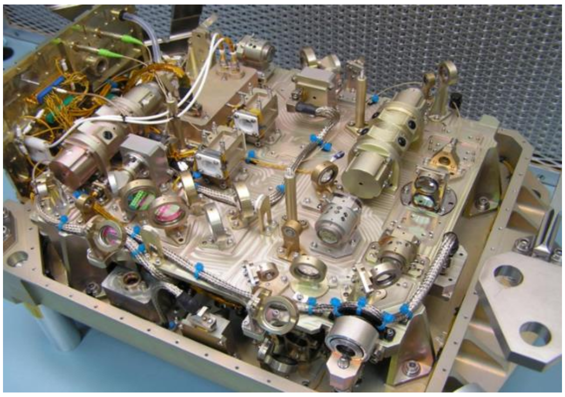

Figure 1 Thin film optical coatings are one of the most commonly used class of coatings for space application. The example shown here is the ALADIN laser instrument on the recently launched Aeolus satellite, which contains coatings on more than 80 optical surfaces ! (ESA/Leonardo Company)

1.3 Categories of use

Many of the existing standards for durability testing of coatings have been developed for ground based applications. On the one hand, some ground environments are clearly not relevant for typical space applications. For example, most space coatings will not see prolonged exposure to the outside elements such as wind or rain. In fact, space hardware is typically well protected on ground under controlled humidity and temperature conditions. On the other hand, there are additional environmental factors which may need to be considered for space applications, especially vacuum and radiation, as well as other mission specific environments such as atomic oxygen and high intensity UV radiation. Therefore, the standard has provided “categories of use”|, in order to differentiate the different environmental factors which may affect the coating :

- Category A: Coating within a sealed, pressurised unit
- Category B: Coating is exposed to vacuum but shielded inside spacecraft
- Category C: Coating is exposed to vacuum with view to space

1.4 Limitations

The following applications were specifically excluded from the standard, because they are already dealt with elsewhere :

- Cover glass coatings on solar arrays (see ECSS-E-ST-20-08C)
- Surface treatments and conformal coatings on EEE parts (ECSS-Q-ST-60/70)

Another notable exclusion from the standard is the qualification of coatings for “long term” ground storage. This is currently a topic of high interest for some satellite programmes especially in the telecommunications sector. Satellites are manufactured “in advance” and stored on ground, ready for launch as soon as the need arises. Storage periods may be as long as 20 years. This is an important topic to address for long term survivability of optical coatings. However the definition of the test approach and related scaling laws (e.g. for accelerated humidity testing) is still an area of on-going research.

2 CASE STUDIES : EXAMPLES OF COATING FAILURE AND LESSONS LEARNT

This chapter provides some examples of coating failures and lessons learnt from previous projects. This is not an exhaustive review but it highlights some of the recurring issues encountered during coating qualifications, providing the motivation and rationale behind the development of the new standard.

2.1 Substrate effects

The type of substrate and the condition of the surface (e.g. roughness, cleanliness) can have a significant impact on the final quality and performance of the coating. For example, one of the options considered for the high temperature edge shields on the Bepi Colombo solar array was a white ceramic coating on an aluminium alloy substrate (Figure 2). High temperature UV testing was performed to verify the long term performance of the white coating. This testing showed unacceptable degradation in the thermo-optical properties of the coating. However further analysis demonstrated that the degradation was in part caused by diffusion of copper from the Al2219 aluminium alloy substrate (Figure 3). This example demonstrates the importance of testing the coating using exactly the same substrate as that intended for flight. It is likely that other alloys of aluminium with lower copper content would not have shown the same problem.

Figure 2 Edge shields with white thermal control coating on the Bepi Colombo Solar Array (ESA/Airbus)

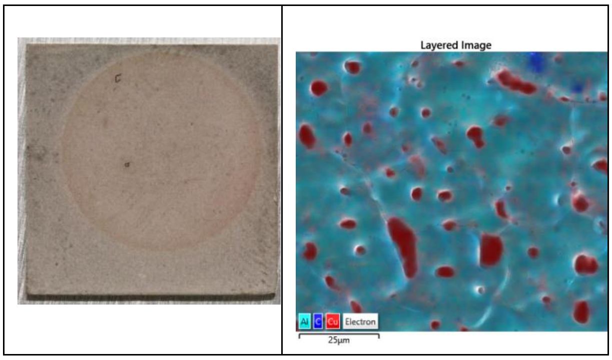

Figure 3 Evolution of copper within the coating after long term thermal ageing test (ESA)

The following example also illustrates the critical role the substrate can play in radiation testing of coatings. After an accelerated high energy proton radiation test on a beryllium mirror with an SiO2 coating, a blue haze area appeared. Analysis with the AFM showed blisters on the surface (Figure 4). This is thought to be a test artefact, caused by the interaction of the beryllium substrate with the high energy protons (>1MeV) used for the test. The test was repeated successfully with a lower energy more representative of the mission – the degradation in the substrate did not occur.

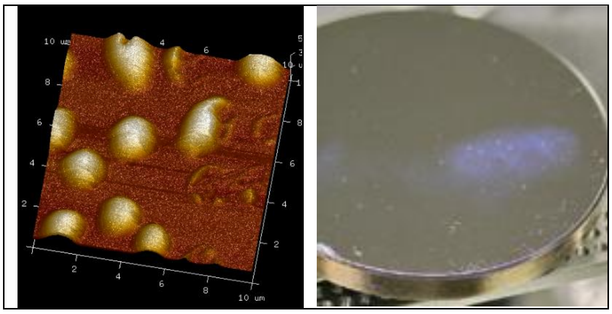

Figure 4 Defects in the SiO2 coating on a Beryllium mirror after high energy proton radiation test (ESA)

2.2 Humidity effects

The humidity test is used as a simple quality control test to check the mechanical resistance and /or stress in the coating (see Section 3.4.3). In general this is a relatively simple test to perform. However, the humidity level during the test is typically higher than 90% and it is important to ensure that condensation does not form on the surface of the substrate. This can cause rapid failure of the coating (see Figure 5). This is prevented by mounting the samples vertically, shielding the samples from the “wet” surfaces of the chamber, and keeping the samples at elevated temperature until the humidity level is decreased.

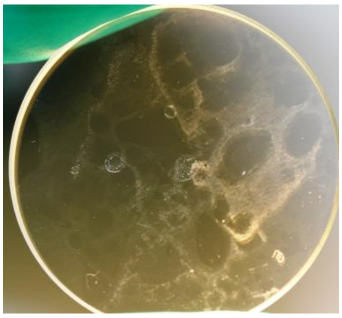

Figure 5 Degradation of an optical coating due to inadvertent condensation during a humidity test (ESA)

The humidity test can also be used to detect defects in humidity sensitive coatings, one common example being the silver coating on the rear of optical solar reflectors (OSR). For example, localised micron sized defects have been observed on "pristine" OSR coatings after prolonged exposure to a standard cleanroom environment. Formation of sulphur containing dendrites are surrounded by areas where the underlying silver coating is depleted. Initially, these defects are on the micron scale, and can only be observed using the electron microscope (Figure 6). However an extended humidity test can be used to accelerate the corrosion, revealing the defects. For example, Figure 7 shows the development of localised corrosion on an OSR after a long duration humidity test (168 hours, 95%, 50C). This is more prevalent near to the edge of the component, where the silver is exposed.

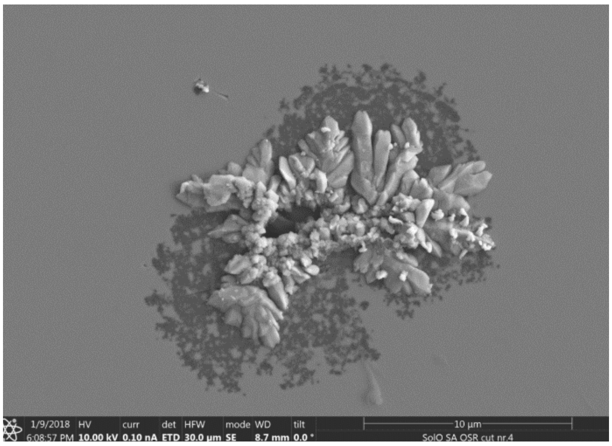

Figure 6 SEM micrograph showing localised delamination of the silver coating and growth of sulphur containing dendritic structure on an Optical Solar Reflector (OSR) after exposure to a standard cleanroom environment (ESA)

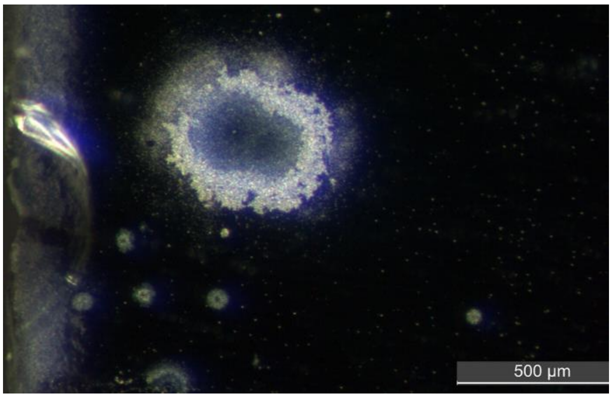

Figure 7 Localised corrosion of the silver coating on an Optical Solar Reflector (OSR) after a long duration humidity test (ESA)

2.3 Geometry

Coating qualification tests are typically performed on flat samples. However some geometrical features such as curved surfaces or sharp edges can affect the coating quality (e.g. adhesion), and specific samples may be manufactured to qualify this aspect of the flight hardware.

For example, Figure 8 shows delamination of black paint from the curved edge of an optical lens. This type of anomaly would not necessarily be detected during qualification tests on flat samples. In another example (Figure 9), delamination has occurred around the sharp edges of cable clamps which have been glued onto a structural panel. Again, this type of failure would not be detected using tests on simple flat samples. In this case, a representative coating sample would be required, including the cable clamp.

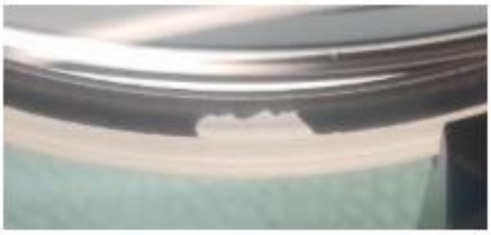

Figure 8 Delamination of black paint on the curved edge of an optical component (ESA/Airbus)

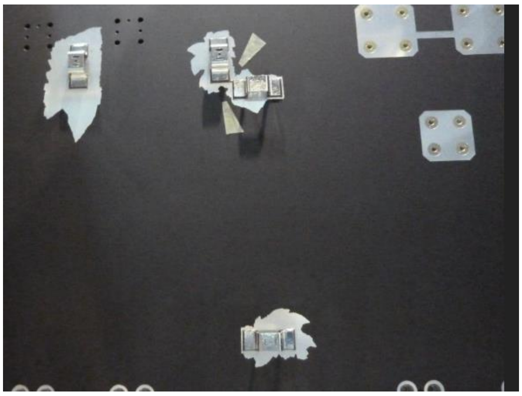

Figure 9 Delamination of black paint on a structural panel, at sharp edges next to cable clamps (ESA/Airbus)

2.4 Cleanliness and contamination

Many coating related issues can occur due to contamination, either during the coating process or in subsequent testing. Poor substrate cleanliness can be a cause of coating delamination and failure.

In some cases, residue on the substrate prior to coating can cause stains in the coating, which may alter the cosmetic appearance of the coating, even though the functional performance durability of the coating is good (Figure 10). In this case, it is important to establish clear acceptance criteria for cosmetic defects.

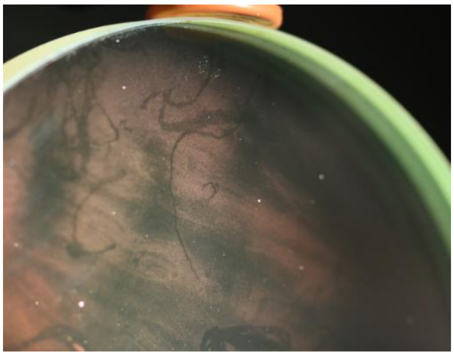

Figure 10 Stains on an optical filter due to substrate contamination prior to coating (ESA)

Contamination which occurs on a coating during thermal vacuum testing at instrument level is often considered as an external environmental effect, which is not considered during sample level qualification testing of the coating [4]. However, there are some cases where the design of the coating, especially the porosity, can influence the contamination resistance. Then it may be necessary to perform dedicated contamination testing on the coating at sample level (Figure 11). For example, an extensive test campaign was performed to qualify the contamination resistance of the UV laser optics for the ALADIN instrument on the Aeolus satellite. It was shown that denser laser coatings manufactured using a technique such as ion beam assisted magnetron sputtering outperform the less dense ebeam coatings (Figure 12).

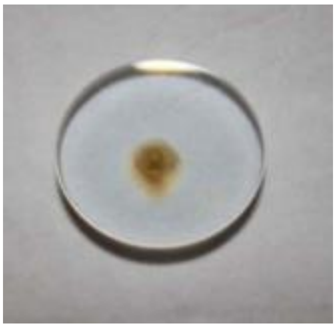

Figure 11 Typical damage on an AR coating sample due to laser induced contamination (ESA/DLR)

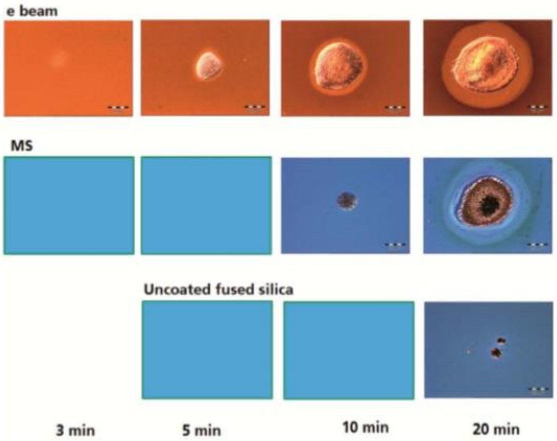

Figure 12 Optical micrographs showing the evolution of contamination induced damage on optical coatings manufactured with different coating technologies. The onset of damage is delayed on the Magnetron Sputtered (MS) coating, compared to the more porous e-beam coating (results from test with 500mJ/cm2, 1khz laser) – ESA/DLR [7].

2.5 Air-vacuum effects

For some types of porous coating, the spectral response can shift to lower wavelengths during the transition from air to vacuum (Figure 13). This can have serious implications for the performance of optical instruments operating in vacuum, and may go undetected if durability testing has only be performed on-ground at atmospheric pressure. Therefore functional testing of the coating under vacuum is always recommended for critical applications [2].

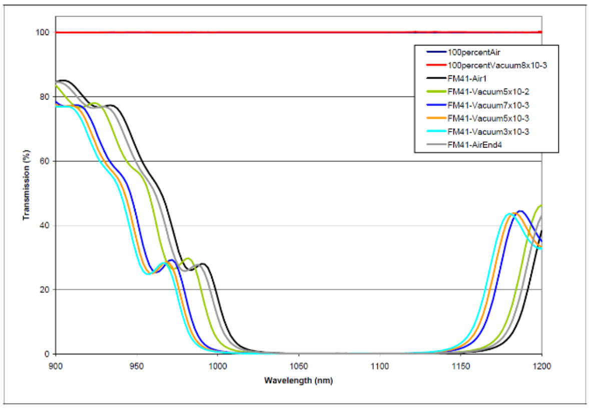

Figure 13 Example of air-vacuum wavelength shift observed in a high reflector coating at 1064 nm (ESA, reproduced from [2])

2.6 Sample storage

It is important that samples are stored correctly during and after the coating qualification testing. Figure 14 shows as example of degradation of a silver mirror which occurred after storage of the sample in a foam packaging. Samples should be well protected, and typically stored in a controlled environment (dry Nitrogen, constant temperature).

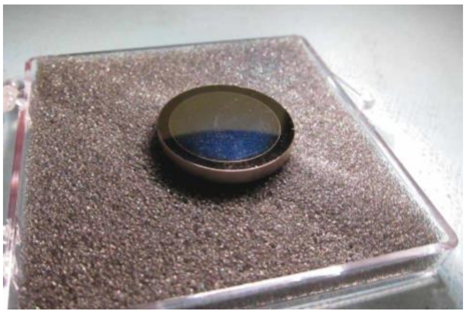

Figure 14 Degradation on an optical mirror due to incorrect storage of test sample in foam material (ESA)

3 CONTENT OF THE NEW STANDARD

3.1 Test Logic

The overall test logic is shown in Figure 15, and the following phases are identified :

Evaluation : First approach to characterise new coatings, using reduced test programme

New Qualification : New supplier, materials or process, full test programme is necessary

Re-qualification : Implemented when there are major changes to existing process e.g Change of equipment, Change of site, Previous major anomalies, Process not implemented for prolonged period (typically more than 1 year)

Delta qualification : Implemented for minor changes in existing process e.g. Minor changes to substrate material (e.g. alloys in different series),Minor differences in coating design (e.g. layer thickness or number)

Production phase : Coating is already fully qualified, and only lot acceptance tests are performed on samples selected from production lot.

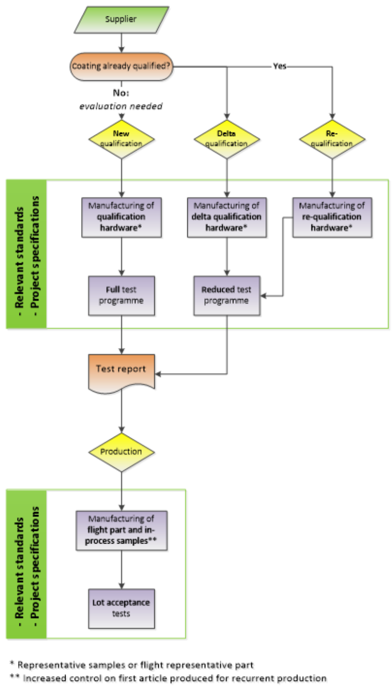

Figure 15 General test logic for evaluation and qualification of coatings (from ECSS-Q-ST-70-17C)

3.2 Sample definition

Typically, a standard coating qualification uses flat samples, manufactured using flight representative processes and materials. In the new standard, the concept of the “coating qualification model” is also defined, when specific design features in the flight hardware or operational environmental conditions can induce coating morphology heterogeneity, coating thickness variation and deformations of the substrate. This takes account, for example, of the size and curvature of the substrate, adhesion layers, primers, optical components with different coatings on each side, masking, grounding or holes for fastening (see also Section 2.3).

3.3 Minimum test requirements

The standard defines the minimum set of durability tests required to verify the quality of the coating process for space applications. The aim of these tests is to ensure that the coating has been produced in a well controlled manner according to a known design. An example of a test matrix for optical coatings is shown in Figure 16. Project specific tests will often be required in addition depending on the mission requirements, and examples are described in Section 3.5.

Test	Method description	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
Performance		1, 5	1, 3	1, 3	1, 5	1, 5	1
Adhesion	Clause 6.2	6	4	4	6	6	
Cleanability	Clause 6.5	2					
Moderate abrasion	Clause 6.6	3					
Humidity	Clause 6.3	4			2	2	
Thermal vacuum and cycling	Clause 6.4		2		3	3	
Particle and UV Radiation	Clause 6.7			2	4	4	
Additional tests	in accordance with requirement 5.2i						

NOTE 1: The numbers in the columns indicate the sequence(order) in which the tests are performed.
For example, the test sequence for Sample1 is performance (1), cleanability (2), abrasion (3), humidity (4), performance (5), adhesion (6)

NOTE 2: The reason for dividing the samples into groups is to test for the following:
S1: Resistance to moisture effects, used as a fast control at start of qualification campaign
S2: Resistance to thermal effects
S3: Resistance to radiation
S4: Cumulative effects
S5: Repeat of test for cumulative effects
S6: Stored for reference (no tests)

NOTE 3: For performance testing, see Annex D

NOTE 4: An additional sample used to test the adhesion immediately after the coating run can minimise the risk of discovering adhesion problems only at the end of the qualification testing

Figure 16 Example of test matrix for optical coatings (from ECSS-Q-ST-70-17C)

3.4 Standard test methods

3.4.1 Visual inspection

The standard provides requirements for visual inspection in order to assess the defects in the coating before and after each test step. It is emphasised that lighting conditions are critical during the visual inspection, especially for some types of optical coating with sensitivity at specific angles or in a specific wavelength range. Then the illumination angle must be carefully adjusted in order to visualise the surface of the coating, with no interference from surrounding light sources (see Figure 17).

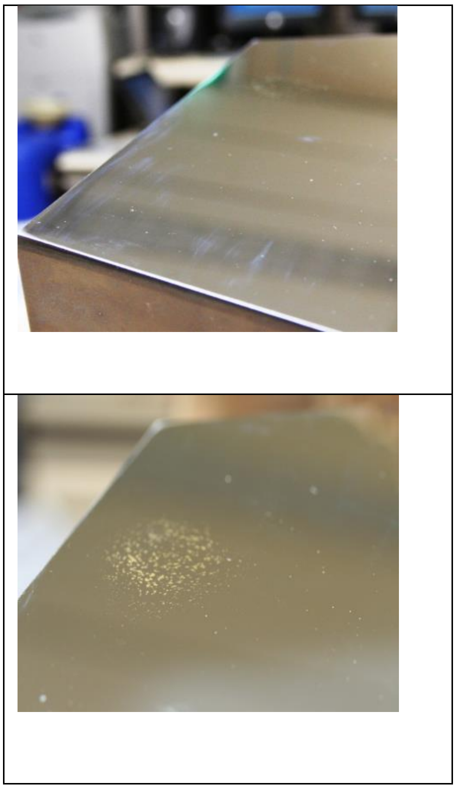

Figure 17 Defects observed on a mirror surface showing the influence of lighting conditions and angle of illumination. In the upper image, cleaning residue and sleeks can be seen on the surface. In the lower image, the mirror was tilted slightly to reveal an area of delamination (ESA)

3.4.2 Adhesion test

The requirements for the adhesion test are derived from the standard “tape test” in ISO 9211-4 [5]. It is acknowledged that this test can be somewhat ambiguous and “operator dependent”|. However it gives fast results and has long been the industry standard for coating suppliers. Unless otherwise required in the relevant specification, the tape is not applied within 2 mm of any rim of the specimen. For optical coatings, typical tapes which are used are 3M 600 or 3M 810, with adhesive strengths in the range 3-4N/cm. Further examples are provided in the annex of the standard. There was also some discussion within the working group about the existing standard ECSS-Q-ST-70-13, which is used to determine the peel and pull off strength of tapes and coatings. This test does not become obsolete with the new standard, but should be used when failures occur and the adhesion strength needs quantifying.

3.4.3 Humidity testing

The humidity test is based on the long established “MIL standards”. Again this is an industry standard test, which is easy to implement and can provide rapid results about the quality of the coating [6]. There was extensive discussion within the working group about the exact humidity and temperature parameters to specify for this test, as different industries have historically used slightly different parameters. Therefore a range of parameters are provided e.g. 40^{O} C – 50^{O} C, in order to accommodate qualifications performed by different suppliers. Technically, this does not make a critical different to the outcome of the test, *in most circumstances.* Of course, there may be some specific coatings which are humidity sensitive, and humidity test parameters are more critical. These must be assessed on a case by case basis.

It is important to emphasise that this is a *quality control test*, and not a lifetime simulation test. It is acknowledged that optical coatings for space applications are typically stored in a controlled atmosphere on ground (e.g. cleanroom or nitrogen purge). Nevertheless, the short term humidity test (e.g 24 hours at relative humidity higher than 90%) can reveal mechanical resistance and /or stress in the coating. The goal of this test is to accelerate the ageing process so that meaningful data can be acquired in short period of time.

The test should be controlled so that no condensation occurs on the coating during the cooling phase (see Section 2.2). Additionally, it will not be possible to perform this test if the substrate or coating is hygroscopic. For simulation of long term storage of optical components, or exposure to more extreme environments, extended humidity testing (e.g. 7 days) may be required, depending on the mission requirements.

3.4.4 Thermal cycling

A standard thermal cycling test is defined, consisting of a minimum number of 25 cycles, with at least 5 in vacuum. This gives the option to perform the additional cycles in an inert atmosphere. The goal of this thermal cycling test is to check the mechanical resistance of the coating under extremes of temperature and vacuum. Typically an optical coating fails during the first few cycles (usually due to thermal expansion coefficient mismatch). Therefore, a limited number of cycles are performed in the first instance as a quality control. Of course, this can be far short of the actual cycles an optical component encounters in orbit. If fatigue related issues are critical, then extended testing is necessary. This needs to be assessed depending on the mission requirements.

3.4.5 Particle and UV radiation testing

The standard provides some general requirements and recommendations for particle and UV radiation testing of coatings. This complements the requirements in the existing standard ECSS-Q-ST-70-06. For testing of coatings, it is important to consider the thickness of the coating, and tailor the test parameters accordingly. For example, simulation of the total absorbed dose using gamma radiation is usually only of very limited use as most of the radiation is not be absorbed in the thin coating. Other factors to consider are :

-Annealing effects (the spectral measurements are made in-situ or as soon as possible after the exposure).

- Degradation of the substrate (the test is always be performed using a substrate which has known degradation characteristics with respect to radiation).

-Margin of safety is agreed with the customer depending on the application (typically MOS = 2)

3.5 Mission specific tests

The standard has an informative annex giving examples of some of the mission specific tests which can be performed to qualify the coating, including :

-Atomic oxygen
-Thermal ageing
-Laser induced contamination and damage
-Solar illumination

Of course this is by no means an exhaustive list.

3.6 Quality assurance

The standard has a specific section on “maintenance of the coating process qualification”. This refers to the ability of the supplier to produce identical coatings with exactly the same process, over a period of time. This is important for recurring production, especially if there is an extended period of time between manufacture of flight hardware (e.g. over 1 year). The supplier must demonstrate that the coating qualification is still valid, and if necessary delta qualification testing must be performed.

This approach was followed for the recurrent manufacture of optical mirror coatings for an instrument on ESA’s Sentinel-3 series of spacecraft. The original coating was qualified for use on the A & B spacecraft in 2012, and the recurrent production of exactly the same mirrors was required in 2018 for the C & D spacecraft.

Three additional coating runs and delta testing was performed before manufacture of the new flight mirrors, in order to verify the repeatability of the process. Additionally, an extended 72 hours humidity test was performed to provide further confidence. There were some process issues in the first coating runs, resulting in a poor quality coating. These were solved by further refurbishment of the coating facility. An additional trial run was performed immediately prior to the flight manufacture as a final check of the coating chamber condition. All samples passed the delta test sequence successfully and the flight mirrors were successfully coated.

4 CONCLUSION

A new standard ECSS-Q-ST-70-17C for durability testing of coatings for space application has been described. The motivation behind the standard is to consolidate test requirements from the large array of existing standards and to prevent the recurrence of common problems during qualification testing. The coating community is encouraged to adopt the new standard for use in new space programmes and to provide feedback to the ECSS so that the standard can evolve in line with future programme needs.

5 REFERENCES

1. ECSS-Q-ST-70-17C, Durability testing of coatings (1 February 2018). The standard can be downloaded from http://ecss.nl

2. Chicarella L. et al, A new ECSS standard for environmental durability testing of optical coatings for space applications, International Conference on Space Optics, Biarritz, October 2016

3. Holynska M et al, *Coatings and thin films for spacecraft applications*, Advanced Materials Interfaces, 1701644, 2018

4. Wernham, D., *Optical coatings in space*, Advances in optical thin films IV, Proc of SPIE Vol. 8168, 2011

5. ISO-9211(3), *Optical coatings, environmental durability*

6. Rancourt, JD, *Optical thin films user handbook*, SPIE Press, Vol. PM37, 1996

7. Riede W, *Laser induced hydrocarbon contamination in vacuum*, SPIE Vol. 5991, 2005