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A NEW ECSS STANDARD FOR ENVIRONMENTAL DURABILITY TESTING OF OPTICAL COATINGS FOR SPACE APPLICATION

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I. INTRODUCTION

Many different environmental factors can have an effect on optical coating durability for space applications. This includes in-orbit effects such as vacuum exposure, UV radiation, particle radiation, atomic oxygen, thermal cycling, contamination and orbital debris, as well as ground based effects such as cleaning, contamination and humidity [1]. There are a large range of national and international standards available which define test requirements and methods for coating durability, mostly for ground applications. Space projects currently utilize combinations of these existing standards, and adapt them according to specific requirements for the project. However there can be discrepancies between projects about the specific standards used, leading to conflicting definitions about when a coating is "space qualified". The supplier and customer often needs to re-negotiate very general aspects of coating qualification for each new project, leading to increased time and effort.

A new ECSS standard is being developed which will capture the best practice across the large range of existing national and international standards, in order to define general requirements for coating use in space applications. This will include a minimum set of tests required for coating evaluation, qualification and production as well as providing information about tests which may be required in specific missions scenarios (e.g. air-vacuum shift, atomic oxygen, VUV, radiation, laser induced damage and contamination induced effects). The ECSS working group is composed of coatings experts, optical engineers, materials engineers and quality engineers from various European industries and space agencies.

II. BACKGROUND

This section summarises some of the background issues addressed by the working group during the early discussions, including lessons learnt from previous projects, the overall motivation for the standard and the expected scope.

A. Ambiguities arising from use of multiple standards

Many ambiguities currently exist about exactly which test parameters to use from the multiple standards which are available for durability testing. Some examples are provided in Table 1.

A. Early screening

Failures encountered part way through a qualification campaign on a new coating can be costly. Therefore, early screening is beneficial. The very simple test sequence adhesion-humidity-adhesion can quickly highlight potential problems during the design or manufacture of the coating. Other specific tests can also be performed during the evaluation phase to assess specific aspects of the coating. For example, it may be beneficial to perform a quick immersion test in LN2 prior to embarking on a lengthy and expensive thermal vacuum test campaign.

B. Vacuum effects

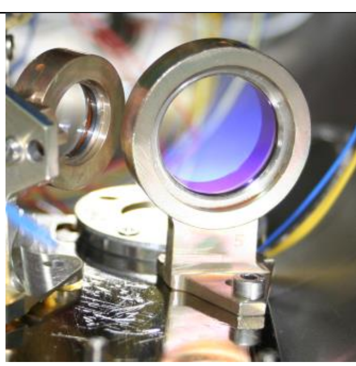
One of the key differences between testing for ground based and space applications is the vacuum level. There is often a need for compromise regarding the tests to be performed in vacuum. On the one hand, suppliers do not always readily have access to the required vacuum facilities, and often opt to perform testing in an inert atmosphere instead. In some cases, this is a valid approach. However the type of coating and criticality of the application must be taken into account when defining the test, as air-vacuum effects can have serious undesired consequences on the performance of space instruments (see section VII for further details of air-vacuum testing).

Table 1 Examples of some ambiguities existing from use of multiple standards for durability testing

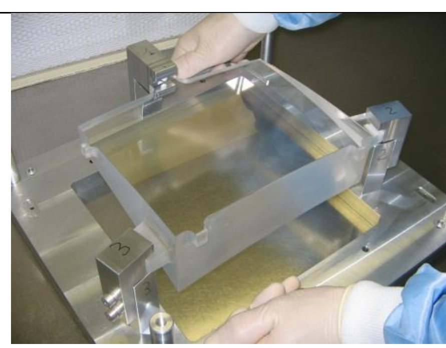
TEST SEQUENCE	• Which is the correct sequence ? • Should adhesion test be performed first to reveal defects, or at the end of the campaign, to test residual resistance ? • Should the abrasion test be performed before or after humidity testing ?
ADHESION TEST	• At what rate should the tape be pulled off : Slow, quick or snap ? • Which tape should be used ? • Is the tape applied on the edges of the coating (some coaters have a mask for best fit and edge exclusion)
HUMIDITY TEST	• Duration (24h, 5 days, 7 days ?) • Temperature (50^{O}C or 55^{O}C ?) • Relative Humidity (95%, 95 % or 100 % ?)
COSMETIC INSPECTION	• Scratch & Dig of MIL-PRF-13830B or 5/ *N x A* of ISO 10110-7? (According MIL-PRF13830B, Dig number refers to the actual diameter whilst Scratch number is "as it appears"; ISO 14997, that works in conjunction with ISO 1110-7, states that defects shall be measured and quantified) • Use microscope measurements or qualitative visual comparison with calibration standard ?
VACUUM EXPOSURE	• When should vacuum exposure occur in the test sequence ? (after humidity is sometimes suggested to stress humidity outgassing) • Can the vacuum test ever be omitted ?
RADIATION TESTS	• A lot of uncertainty exists, and sometimes over-testing occurs, or substrate effects invalidate the results (e.g. by use of non-rad hard materials) • Is gamma radiation beneficial / necessary for thin coatings ?

C. Geometry of optics

Optics for space instruments come in a vast range of different shapes and sizes (Fig. 1). Poor adhesion can be caused by the complex shape of the optical surface. When the size and / or the curvature or any unusual design of the sample is such that the manufacturing process could induce coating morphology heterogeneity, coating thickness variation and deformations of the substrate, a coating qualification model is necessary to complete the qualification. In this case, the geometry is more representative of the flight hardware (or in some cases, it must be fully representative). Specific design configurations may also need to be taken into account (e.g. including same masking and electrical grounding points, if any). Special tests may be also necessary to verify the coating quality (typically out of the scope of the standard) and the qualification samples may be adapted for this purpose.



Small components in a laser instrument (HR coating from ALADIN project, Leonardo company)



Component with large size and complex shape (portion of lens with spheric faces from Gaia project, Leonardo company

Fig. 1 Optical components come in a vast range of different shapes and sizes, and the durability test samples need to be adapted accordingly

III. CATEGORIES OF COATING USE FOR SPACE APPLICATIONS

Categories of use are defined in order to determine the severity of durability testing required. For example in the ISO 9211 standard [2], many different categories are defined, covering various ground based environmental situations (eg. dust, sand, high humidity, salt spray, icing/frosting). In general, for space applications, the on-ground environment is typically less severe, and optical components are well protected in clean-room environments, with cleaning limited to the minimum. Therefore, the severity of the testing can be adapted accordingly. For example, the coating may not have to withstand severe abrasion, or exposure to salt spray and sand. In orbit however, the environment will vary depending on the mission, and it is very important to take this into account when defining the test programme. For example, the optical components may be in sealed units, where no vacuum testing is required, they may be under vacuum but shielded from the space environment, or they may be directly exposed to space. These categories of use are summarized below.

Category A

This category refers to components which are mounted internally within sealed (pressurized) units. It is likely that access to the coating will be restricted once the instrument is assembled, so cleaning will be very limited. In orbit, the components will not be exposed to vacuum and will normally have a less severe radiation exposure due to shielding from the enclosure.

Category B

This category refers to components which are embedded inside an optical instrument, but not fully sealed. In-orbit, the components will be exposed to vacuum, but not directly exposed to space. They will normally have a less severe radiation exposure due to shielding from the instrument or spacecraft walls.

Category C

This category refers to components which have a direct view to space (e.g. telescope mirror). On ground, the components will generally only be exposed to a controlled environment (e.g. in clean-room). However, additional cleaning of the coating may need to be performed during the instrument integration phase. In-orbit, the components will be exposed to vacuum with a direct view to space. Radiation shielding may be limited, and other environmental factors may need to be considered (e.g. UV radiation, atomic oxygen)

Category O

This category refers to applications which require special, non-standard, specifications. Since the specification of the components in such cases will not exactly fit into one of the categories A to C, the recommended way to specify in such a case is to indicate first the category in which most requirements are satisfied. The exceptional requirements can then be specified from other categories or by indicating the test degree of severity [2].

IV. TEST LOGIC

The level of testing required depends on the project phase. The different factors to take into account are described in the following sections and the overall logic is depicted in Fig. 2.

A. Evaluation

This is a first approach to characterise new coatings at low Technology Readiness level. A reduced test programme is used as an early screening to intercept weak coatings before starting the core of the expensive qualification test programme (see also section II). The minimum set of tests to be performed are humidity, adhesion and thermal cycling.

B. New Qualification

A new qualification is necessary for a new manufacturer, new materials or completely new coating process. In this case, a full test programme is required, with a dedicated test plan. The qualification matrix (see section V) shall guarantee :

-Repeatability of the process is verified (e.g. by performing 2 coating runs for the qualification samples)
-Essential tests that are unavoidable
- Correct test sequence
-Not too many resources in terms of samples quantity, number of tests, campaign duration, number of spectral measurements (avoid repetition and redundancies)

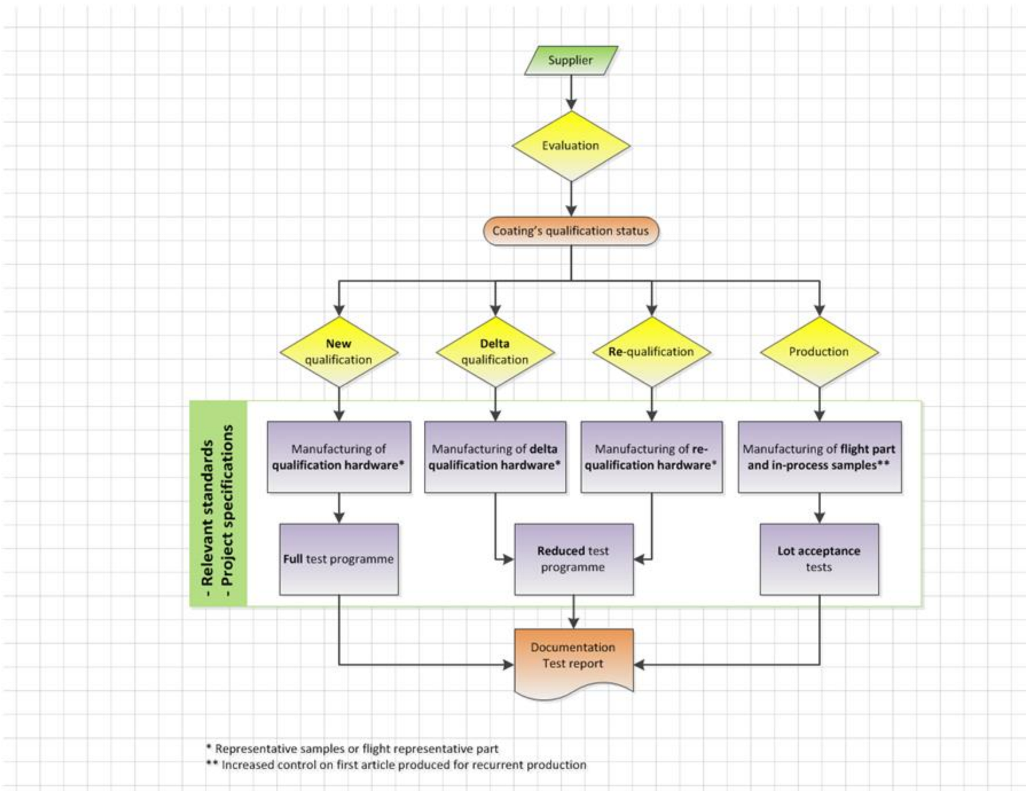


Fig. 2 Overall test logic for durability testing of optical coatings

C. Re-Qualification

A re-qualification is required in case of major changes to implementation of an existing coating process e.g.
-change of equipment, site
-previous major anomalies
-process not implemented for prolonged period
In this case, a reduced test programme may be accepted, according to existing test plan (for example, it may not be necessary to repeat radiation testing if the design of the coating has not changed)

D. Delta Qualification

A delta qualification is required in there are small changes to the coating process, for example :
-minor changes to substrate material (e.g. different alloys in same class)
-different environment
-different substrate geometry or surface preparation
-minor differences in coating design (e.g. layer thickness or number)
In this case, a reduced test programme may also be accepted, according to modification of existing test plan

E. Production

For a coating which is already fully qualified, lot acceptance tests are performed on samples selected from the production lot. The standard tests to be performed are similar to those for qualification (adhesion, humidity, thermal cycling), but not all of the mission specific tests will be required. Extended acceptance tests could be performed in case of first article approval of recurring production.

V. MINIMUM TEST REQUIREMENTS

A minimum set of durability tests are required for verifying the quality of the coating process for space applications (Fig. 3). The aim of these tests is to ensure that the coating has been produced in a well controlled manner according to a known design. In this respect, these durability tests may not precisely replicate the “mission environment”, and in fact in many cases the coating will be “over-tested” (for example, tape will not normally be applied on a flight coating!). However they are well established “industry standard” tests which demonstrate the robustness of the coating, whilst highlighting potential problems as quickly and as simply as possible. Project specific tests will often be required in addition depending on the mission requirements, and examples are described in section VII.

		Fast control at start of qualification campaign	Thermal resistance verification	Radiation resistance verification	Overall cumulative agents resistance verification	Doubling for statistical the Overall cumulative agents resistance verification	Untested, to be stored for potential test repetition
Seq.	Test	1	2	3	4	5	6
1	Performance	Spectral	Spectral	Spectral	Spectral	Spectral	Spectral
2	Adhesion	X					
3	Cleanability	X					
4	Moderate Abrasion	X					
5	Humidity	X			X	X	
6	Thermal-Vacuum Cycle		X		X	X	
7	Thermal Cycle (atmospheric pressure) (see text)		X		X	X	
8	Radiations			X	X	X	
9		Adhesion	spectral	Spectral	Spectral	Spectral	
10		Spectral	Adhesion	Adhesion	Adhesion	Adhesion	

Fig. 3 Test matrix for minimum set of durability tests

A. Adhesion testing

The adhesion test is normally one of the first durability tests to perform on a new coating, and one of the simplest, yet at the same time it can be one of the most ambiguous ! A tape of specific adhesive strength is applied directly onto the surface of the optic, and pulled off by hand at a specified rate. Unless otherwise required in the relevant specification, the tape is not applied within 2 mm of any rim of the specimen [2]. For a good coating, there should be no evidence of the coating on the tape. For optical coatings, typical tapes which are used are 3M 600 or 3M 810, with adhesive strengths in the range 3-4N/cm. Of course, it can be argued that the implementation of the test is somewhat “operator” dependent (e.g. the rate of pulling off the tape is not measured), and there may be some debate about pass/fail criteria in some situations, for example when a tiny piece of the coating is noticed on the tape. However, it is a well established test, very quick and easy to perform, and it can give very reproducible results as long as it is performed under controlled conditions by trained operators.

B. Humidity testing

Optical coatings for space applications are typically stored in a controlled atmosphere on ground (e.g. cleanroom or nitrogen purge). Nevertheless, a short term humidity test (e.g 24 hours at relative humidity higher than 90%) is used as a simple quality control test to check the 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. There is no firm scaling factor that correlates the duration of the test with the life in a given environment, because the degradation mechanism that takes place in highly accelerated testing is not the same one that causes the long term degradation [3]. However, the test is the best technique to accelerate the ageing process simply and repeatably and to evaluate if a coating is reasonably durable. The test should be controlled so that no condensation occurs on the coating during the cooling phase. 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.

C. Thermal cycling

The goal of the thermal cycling test is to check the mechanical resistance of the coating under extremes of temperature and vacuum. Typically, an optical coating will fail during the first few cycles (usually due to thermal expansion coefficient mismatch). Therefore, a limited number of cycles (e.g. 20) can be performed in the first instance as a quality control. Of course, this may be far short of the actual cycles an optical component may encounter in orbit. If fatigue related issues could be critical, then extended testing may be required. This needs to be assessed depending on the mission requirements.

The main disadvantage of thermal vacuum cycling is the very high cost and long schedule implications (the duration of the test could be several weeks). It is important to ensure that the optic reaches the actual test temperature, and this takes more time for large, glass optics which are not thermally conductive. Sometimes, it may be possible to replace some of the vacuum cycles with thermal cycles at atmospheric pressure.

An alternative thermal shock test could be considered to quickly assess the quality of a new coating (e.g repetitive immersion in LN2 and hot solvent). The advantage of thermal shock testing is increased cycling speed (see also section II). However it is more severe, because :
-it is subject to atmospheric pressure conditions which can form ice condensate and exert disproportionate stress on the coatings.
-it is uncontrolled with time and excursion temperature rates.

D. Radiation testing

Radiation test parameters depend on the mission environment and the configuration of the optical system. For example, an optical component embedded within an instrument may be shielded to a greater extent than an optic exposed directly to space. The Category of Use for the component needs to be taken into account (section III), and it may be necessary to perform a radiation analysis to predict the radiation level. To define the radiation test parameters, the thickness of the coating must also be known, so that the absorbed dose for a given energy can be estimated. In general, testing must be performed with the actual radiation species encountered in the space environment (e.g. electrons, protons). Simulation of the total absorbed dose using gamma radiation will only be of very limited use as most of the radiation will not be absorbed in the thin coating. Other factors to take into account when performing the radiation test are :
- annealing effects (the spectral measurements need to be made in-situ or as soon as possible after the exposure)
-degradation of the substrate (a reference uncoated substrate should always be tested at the same time).

Optical coatings are often declared by suppliers as “radiation resistant” based on previous heritage. In this case, it must be proven that the materials and general composition of the coating have not changed, and that the radiation environment for the mission envelopes the previously tested coating.

E. Cleanability and abrasion testing

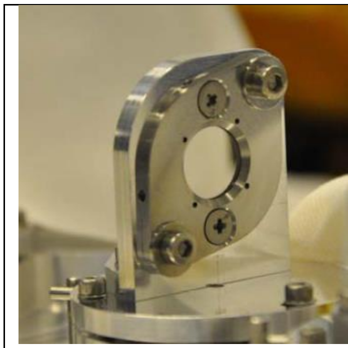
In general, these are standard tests defined in the ISO 9211 standard [2]. However the severity of the test may need to be adapted depending on the Category of Use for the optical component (section III). For example, for Category A and B optical components, the abrasion test may be omitted, if it is justified and agreed with the customer that these are sensitive coatings which will not be cleaned during the life cycle. For Category C optical components, a dedicated cleaning procedure may be required (for example, CO_2 snow cleaning), and the durability test samples should then be subjected to the same procedure, rather than simply applying the standard cleaning test.

VI. INSEPCTION AND FUNCTIONAL TESTING

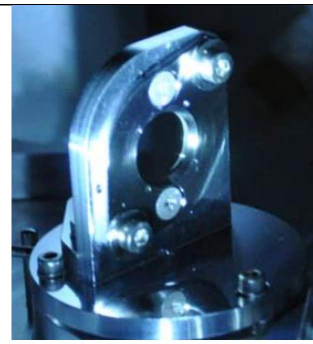
The specific requirements for visual inspection and functional testing are outside of the scope of the durability test standard. However, these tests must be performed in order to verify that the durability tests have been successful. As a general requirement, samples will always be visually inspected with the naked eye before and after each test step to verify the requirements for defects in conformance with project requirements. Lighting conditions are crucial (Fig. 4). Examples of cosmetic requirements and relative inspections are provided in ISO 10110-7 which, in its updated version, works together with ISO 14997:2011.

ISO 10110-7 provides only general indications for the arrangement of the visual inspection station, but it prescribes that, in the case of defects “worthy of study”, they shall be measured and quantified by microscopic measurements according ISO 14997.

The most common functional testing for optical coatings is spectral measurements (reflectance or transmittance), and these measurements may need to be repeated several times during the durability test campaign (e.g. after humidity, after thermal cycling etc.).



Optic viewed against white background with room lights on



Same optic viewed against black background using torch at oblique angle of incidence, which can reveal surface defects

Fig. 4 Lighting conditions must be correct to perform a good visual inspection

VII. MISSION SPECIFIC TESTS

Examples of some mission specific tests which can be used to evaluate optical coating performance in the space environment are provided in the following sections. In these cases, the test requirements are derived directly from the project specification. This is by no means an exhaustive list, but gives a flavor of the additional testing which may be required to qualify an optical coating for a specific space mission.

A. High intensity solar radiation exposure

This type of testing may be required for optical coatings which are exposed directly to high intensity solar radiation. The coating may be exposed during routine operations (for example an entrance window of a sun-pointing instrument), or it may only be exposed during non-nominal operations (for example if a telescope is pointed near to the sun for a short time). It may be possible to simulate the effects of a short sun exposure simply by rapid heating of the optic. For testing long term ageing effects, a more complex test set-up may be required, using a dedicated vacuum facility coupled to a solar simulator.

B. Laser induced contamination (LIC) / laser induced damage (LID)

LID and LIC testing is applicable for optical coatings which will be exposed to high power laser beams. Specific test methods have recently been developed in the frame of ESA's Aeolus and Earthcare satellites [4]. For LIC testing, the coating is exposed to the laser beam in the presence of organic materials, and the resulting transmission loss is measured. For LID testing, the coating is exposed to multiple laser shots with varying intensity until damage occurs, in order to measure the so-called laser induced damage threshold of the coating in vacuum.

C. Atomic oxygen

Atomic oxygen testing is applicable for coatings exposed directly to the space environment in Low Earth Orbit (e.g. optical payloads for Earth Observation missions). In general, most inorganic / metallic coating materials can be considered as atomic oxygen resistant, especially if there is a protective outer layer of SiO_2. However, substrates made from sensitive materials can still be exposed to atomic oxygen if there are defects or cracks in the coating, and erosion can be increased due to undercutting. Examples of coating systems which may be potentially susceptible are protected silver coatings (for example on mirror substrates or radiator fins), or thin protective coatings on polymeric films (e.g. ITO on Kapton). In general, specialist facilities are required for the atomic oxygen test, and there are a number of different techniques which can be used to generate the atomic oxygen. To test optical coatings, one key requirement of the test source is that it produces a "clean" atomic oxygen beam, without molecular or particle contamination which could interfere with the optical properties of the coating. If degradation is observed, then specific surface analysis techniques (e.g. SEM, XPS, SIMS) may be required to assess on the molecular level how the coating has reacted with the atomic oxygen.

D. Air-vacuum measurements

For some types of porous coating, the spectral response can shift to lower wavelengths during the transition from air to vacuum. 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

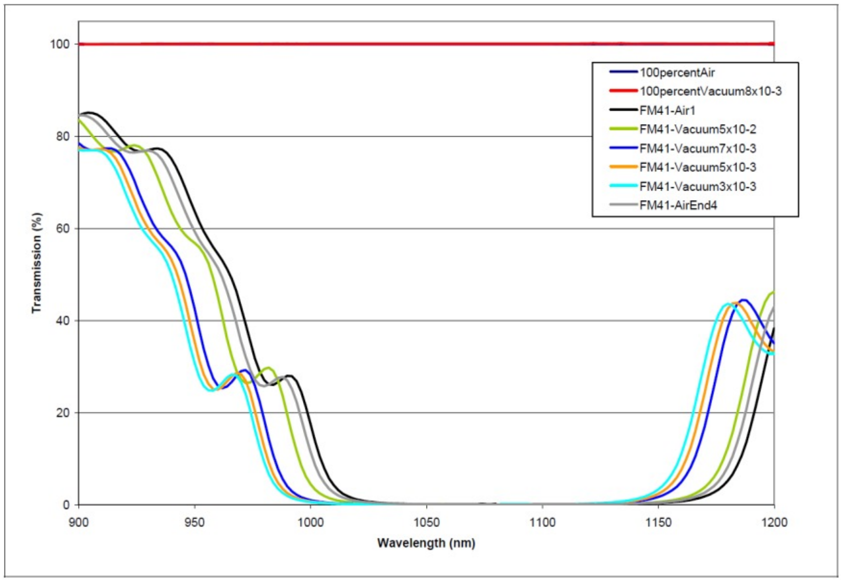


Fig. 5 Air-vacuum shift effect observed in an infrared HR coating

pressure. Therefore functional testing of the coating under vacuum is always recommended for critical applications. For small optics, a vacuum cell can be incorporated into the sample compartment of a standard bench-top spectrophotometer, and spectral scans are continuously taken as the vacuum cell is evacuated (Fig. 5). For larger samples, or measurements on flight hardware, a purpose built vacuum facility may be required, with a means to adapt the spectrophotometer onto the facility.

E. Contamination effects

Molecular contamination can be deposited onto optical coatings due to vacuum outgassing from nearby organic materials on the spacecraft. In general, this effect is not associated with the durability of the coating itself but rather the external environment. However, in some cases the coating design can change the affinity of the molecules to stick to the surface, especially in the presence of UV radiation. Testing can then be performed to assess the response of different types of coating under controlled contamination flux.

VIII. CONCLUSIONS

This paper has described the motivation behind a new ECSS standard for durability testing of optical coatings for space application. Examples of “lessons learnt” from previous space projects and typical pitfalls which can be encountered during coating qualifications have been provided. The minimum set of required durability tests and methods have been summarised, and the unique testing needs for space applications have been highlighted. The concept of the coating qualification model has been defined, when simple flat samples are not fully representative (e.g. for curved surfaces, sharp corners or edge masks). General requirements for ensuring maintenance of coating qualification have been 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, coating equipment etc.). The benefits of performing a simple set of durability tests early in the evaluation phase of a space project have also been highlighted. The ECSS standard is at the stage of the working draft, with publication aimed for end 2017.

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