POLYMER RADIATION TESTING GUIDELINES FOR SPACE APPLICATION

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GLOSSARY

EOL	End Of Life
GEO	Geosynchronous Equatorial Orbit
MLI	Multi Layer Isolation
TID	Total Ionizing Dose
TRB	Test Review Board
TRR	Test Readiness Review

ABSTRACT

Materials used to manufacture spacecraft may experience a significant radiation stress during their live flight. The absorbed dose profil into these materials depends on the material nature, its location on the spacecraft and the mission parameters (duration, type of orbit). In order to assess the material functionality degradation due to radiation and estimate the end of life (EOL) properties, a qualification phase is necessary involving on ground experimental tests. This paper provides a synthetic compilation about the main steps of a qualification and the test parameters to determine and fix for the irradiation experimental tests. The aim is to provide adequate guidelines for testing. Recommendations are made to optimize the pre-irradiation, experimental and post-irradiation phases of the material qualification. These recommendations are based on experimental tests especially performed for this work, as well as on European prime contractor internal qualification processes.

Following these recommendations, warnings are presented, in order to raise awareness on the various points that may affect the definition of the test plan and the representativeness of the results obtained with this test.

INTRODUCTION

In space applications, materials are exposed to specific environments: hard vacuum conditions and temperatures, charged particles (from the radiation belts), the solar spectrum, atomic oxygen (in low orbit, <1000 km), micrometeorites and debris.

Among these different environmental parameters, the charged particles (constituted by electrons or protons trapped in the radiation belts, rash and solar wind) generate an ionizing absorbed dose profile into these materials.

The interaction of particle radiation with an organic material causes the phenomena of chain scission and/or crosslinking via ionization mechanisms. The effects are linked to the accumulation over time of the ionizing dose (cumulative effect), causing surface and/or volume changes into the material. The induced macroscopic effects consist mainly of colour change and the modification of mechanical and electrical properties performance.

To ensure materials keep their functionality during the entire mission, the evaluation of the EOL properties is necessary.

To do this, the material properties are measured before and after performing irradiation ground tests that simulate the space environment. These ground tests are not fully representative of space environmental conditions and the choice of parameter conditions have to be carefully defined.

In space domain, there is no specific fixed rule concerning the irradiation ground tests conditions on materials only a guidance through ECSS-Q-ST-70-06C [1]. Thus different testing logics can be applied without knowing the representativeness of the ground test campaign compared to the real in orbit irradiation. Several studies were conducted in order to defined the impact of key parameters irradiations on the evolution of properties and have shown that any general conditions can be fixed, however some precautions and recommendations have to take into account in order to be the best representative as possible.

In this context, this paper presents the global approach for qualifying a material exposed to irradiative environment and provides some warning and recommendation on key parameters to consider for environmental tests. The paper proposes:

- to review and detail the mains steps of the usual radiation qualification process commonly used in the European space industry
- to provide simplified radiation testing guidelines for material space applications
- for each step, to discuss the impact of the main parameters that have to be chosen
- to provide some recommendations and warnings to help to choose the best appropriate parameter tests

GLOBAL APPROACH FOR THE QUALIFICATION OF MATERIAL UNDER RADIATION

The global synoptic for the qualification of a material to radiation is presented on the figure hereafter.

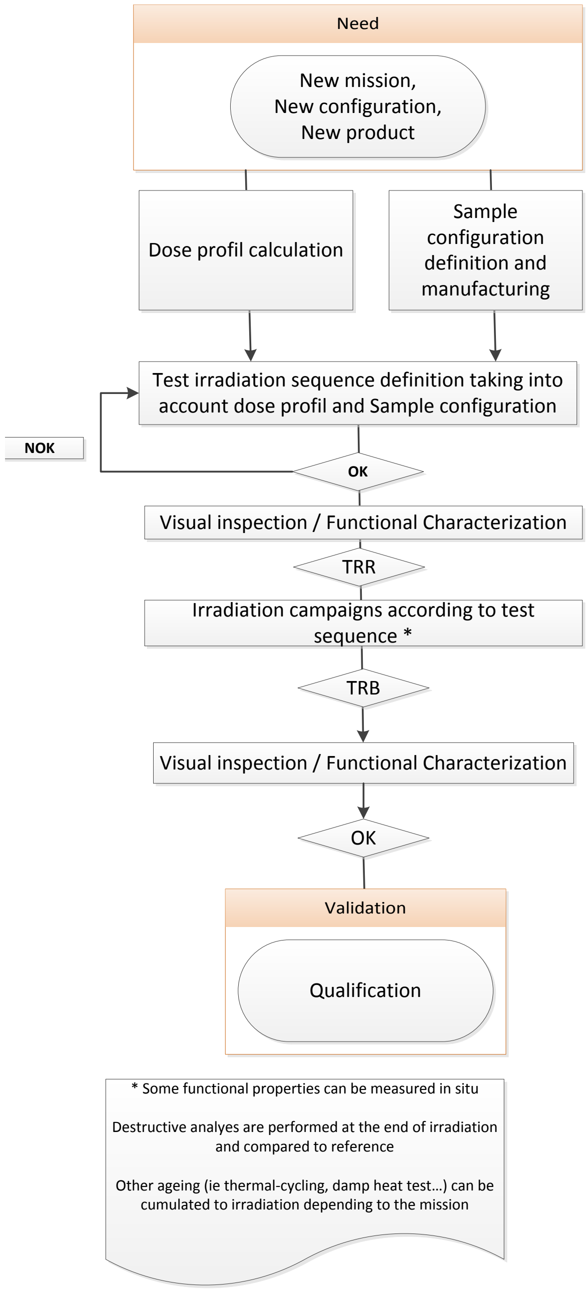

Fig. 1. Global Synopic

First, the need shall be defined at project level. A global review of the context and exact need has to be performed. The need is generally linked to the use of a new product, or the use of a material for a new mission, or the use of a material on another location on the satellite.
The topics to consider are the following ones:

- Projects environments
- Heritage and knowledge on materials
- Margin
- Material specific uses / critical property

The outputs of the project review allow subsequently to define the qualification test plan. It will be presented in detailed in next paragraphs and includes:

- the sample configuration for irradiation tests and manufacturing conditions,
- the dose profile calculation,
- the test irradiation sequence
- the definition of functional properties and success criteria.

Once the test plan defined, the samples are manufactured and the irradiation campaign can be launched including functional characterization at different stages of irradiation, sometimes in-situ depending on the properties to monitor.

QUALIFICATION TEST PLAN DEFINITION

During the test plan definition phase the following points have to be addressed:
- The calculated dose profile for each material to qualify
- Sample configuration (test vehicle)
- Functional test conditions and acceptance criteria
- The test campaign itself including:
  ▪ the irradiation test sequence
  ▪ irradiation test conditions
  ▪ additional environmental testing

3.1 Calculation of the dose profile

The dose level received during the flight depends on several parameters:
- Mission (flight duration, orbit...)
- Material type (density and chemical composition)
- Location on the spacecraft (spacecraft structure, MLI…)
- Material configuration (2D or 3D, thickness of coating or sheath,...)

The dose profile for each material to qualify shall be calculated. Different methods exist to compute dose. The one to be applied has to be agreed at project level.
The aim of the TID profile calculation is to assess the dose level experienced on orbit, in order to be able to define representative experimental parameters for the ground test qualification.

3.2 Sample configuration and manufacturing

Sample configurations shall be defined taking into account:
- The property to test

- Dimension and numbers required for functional test,
- Dimension compatible with radiation facility (the irradiation facilities have limited capacity and the place and/or sample configurations have to be optimized)
- The deposited dose: the thickness of the sample has to be defined in order to deposit a dose corresponding to the mission dose profile

3.3 Functional Test conditions & success criteria

The characterizations enable to validate or not the material qualification. It is important to perform it in appropriate conditions in order to have usable results.
Test methods and conditions shall be described in the test plan and the success criteria shall be fixed.
Among all the characterization tests, the following ones are commonly monitored depending on the application:

- Visual inspection
- Mechanical characterization
- Electrical characterization
- Thermo-optical characterization
- Physico-chemical characterization

As mentioned previously, the properties can be measured at different stages of the irradiation, and sometimes in-situ measurement are performed (for example for thermal-optical properties).

3.4 Test campaign

3.4.1 Experimental irradiation test sequence

Once the mission dose profile computed, the radiation ground test should be designed in order to be representative of the dose level expected during the flight.
The test sequence ie. Type of particle, flux and energy of particle is defined regarding the mission dose profile, the material nature and density and the sample configuration as well as ground facilities constraints. Commonly, ground test facilities deliver mono-energetic beams in different range of energy and flux.
For an established test sequence, the total deposited dose inside the material is calculated and compared to mission dose profile. The sample configuration for qualification shall be considered (sometimes different from configuration on satellite). Finally, the test sequence shall be set up in order to make the two dose profiles fit as much as possible (real dose profile for material mission and dose profile calculated for the material test on ground).

As mentioned previously, it is impossible to reproduce exactly the same dose profile on ground than on flight due to facility limitation. So a compromise has to be taken to be as close as possible. Once the two dose profiles are sufficiently close, the radiation test sequence is approved. This exercise is done for each material. A test sequence can be defined in order to cover several materials. In other cases, test sequence can be stopped at different level in order to achieve different dose needed for different materials.

Sometimes it is difficult to find the good compromise to select the best appropriate sequence as illustrated by the following examples.
For a specific environment (GEO), two possible testing strategies are presented below: a worst (Fig. 2) and an averaged test (Fig.3).

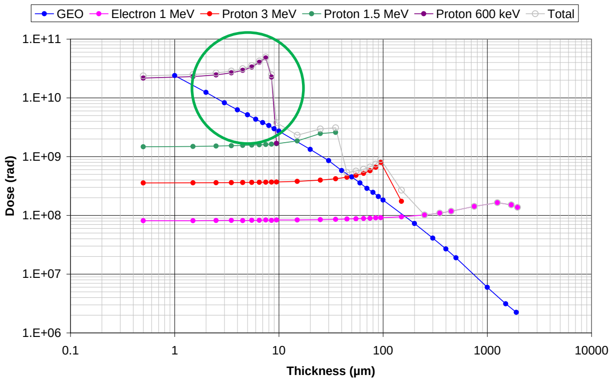

Fig. 2. Worst case test sequence covering the GEO external dose level

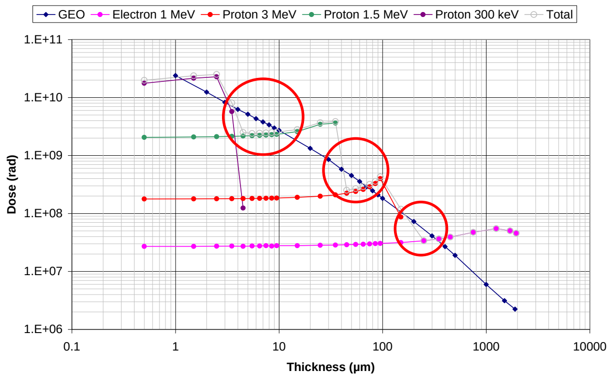

Fig. 3. "Averaged" test sequence not fully covering the GEO external dose level

If the test is performed according to the surface dose level (Fig.2), the material volume may be over-irradiated (green circle), whereas if the test is performed according to the volume dose level (Fig.3), the material surface may be under-irradiated (red circles). As a consequence, several test conditions (particle type, energy and fluence) may be used in order to reproduce more accurately the dose decrease in the material thickness. The functionality of the material can also be consider and used as driver to choose one of the two sequence.

3.4.2 Irradiation test conditions

In the test plan, the irradiation test conditions shall be defined. The key parameters to fix are the followingd:

- Temperature of irradiation. An operational temperature has to be fixed for the irradiation. Thermo-couple location or any other temperature measurement facilities have to be agreed.
- Atmosphere: ambient air, nitrogen atmosphere or vacuum. The irradiation atmosphere has to be fixed in function of the material tested and the need. For instance, if the characterizations to monitor consist in thermo-optical measurements, it is strongly recommended to do the irradiation free from oxygen.
- Flux: iteration has to be done between the facility and the project to agree on an irradiation flux
- The test sequence order has also to be fixed.
- ….

3.5 Samples management

The samples packaging is important. Some irradiation test facilities can be located in foreign countries and samples could have to travel between each phase of the test campaign.
Samples have to be packaged in a non-contaminant material (SCC 1000 material typically) and, in function of the parameter evaluated, they could be packaged under nitrogen or partial vacuum.
The samples storage or travel duration have to be reduced to the minimum possible between each phase of the test campaign (between two irradiations or just before characterization tests).
Intermediate testing may be required to track any evolution of critical parameter associated to storage period and conditions.

After the final test campaign (after characterization tests), the samples shall be stored in controlled environment in order to be able to use them for additional tests in the future if needed.

ANALYSE / WARNING

This paragraph presents some warnings, preconisations and precautions to consider for defining the test plan, especially irradiation parameters based on work previously published by the same authors on this topic [2] and on bibliography.

4.1 Materials

As already explained in the project review, the material background has to be considered at the early stage of the study.
Depending on the properties assessed, the presence of charges, the exact formula in a given chemical family, manufacturing processes can induce different results in term of properties variations.
For example: after irradiation at 500MRad, two epoxides bi-component adhesives show a different behavior in term of mechanical degradation regarding dose rate variation [2]. Generalization into a same materials family cannot be done.

4.2 Dose profile Calculation

The mission dose profile is generally computed with a Monte-Carlo method (reverse or forward). However, in specific case, the sector analysis based on equivalent aluminium shielding can be used to define a first estimation of the dose level. Depending on the result, further analysis may be done. Nevertheless, the sector analysis may not be used for material dose profile calculation.

In all cases, the environment sources that should be taken into account are:

- trapped electrons
- trapped protons
- solar protons

The experimental test sequence can also be calculated with Monte-Carlo method. The representativeness of the test sequence is a critical point (see 3.4.1)

4.3 Test conditions

Irradiation test conditions may affect the degradation of polymeric materials during their exposure into a simulated space environment. The 4 main parameters to be set for an irradiation test are:

- Type of particles
- Dose rate
- Atmosphere
- Temperature

4.3.1 Type of particles effect

In our study, no significant differences have been observed depending on the type of particles used for irradiation. However degradations of properties studied were limited, and close. Moreover, differences have been noticed during the dose rate effect study.
The thicknesses of the samples and the dose deposition profiles are the driving parameters. Not all material families were addressed during this study.
When possible, it is recommended to use the particle type the most representative of these-one of the orbit environment.

4.3.2 Dose rate effect

In most of the materials studied, we did not see any impact of the dose rate on the degradation of the

properties. However, for some materials, an impact of the dose rate has been noticed. The worst case can be either at low dose rate or high dose rate depending to the material. In addition, we note a non-similar impact depending on the radiation particles type [2].
This parameter has to be taken into account during the test plan definition and the analysis of the test results, and also the timing of test sequence.

4.3.3 Ageing atmosphere effect

The presence of oxidizing gases in the ageing atmosphere (such as dioxygen in air) can have an impact on the materials evolution. Generally, irradiation in air results in less crosslinking into the polymer samples than under vacuum [3]. The presence of oxygen allows oxidation reactions with the radicals created during radiolysis [4]. This phenomenon increases the ratio scissions / crosslinking created during the degradation of the polymer [3-5]. The main consequence is an accelerated structural degradation of the sample [6].
Moreover, healing phenomena are observed with the presence of air in the ageing chamber. Thus, modifications of the UV-Vis-NIR spectra have already been observed during a vacuum / air transition step after an irradiation sequence [7-9]. This healing can be relatively fast, ranging from minutes to hours. Even traces of oxidizing elements can cause these recoveries, with evolutions of white paints UV-Vis-NIR curves during a vacuum / nitrogen transition [9].

As conclusion, it is recommended to perform irradiation and surface characterization without oxygen, so under high vacuum or at least inert atmosphere.

4.3.4 Ageing temperature effect

Temperature is an important parameter that can impact the degradation of materials, especially polymeric materials. The mechanisms occurring during the irradiations, the radiolysis and in a second part the oxidation (if ageing under air), are generally thermo activated. The global degradation of the sample is therefore higher at higher temperature [7,10].
A localized heating can also result in changes in physical state (melting for example) and degradations (bond breaks) of polymers by specific phenomena of thermal degradations.
On the other hand, temperature also governs the healing mechanisms of colored centers (optical materials [11]).

It is recommended to control the temperature during irradiation and to define the allowable temperature range for the irradiation. It has to be noted that particle irradiation flux can have an impact on the sample temperature.

4.4 Test limitation

The technical discussions to define material radiation tests should take into account practical limitations. Due to several experimental factors, the ideal approach is not always feasible to perform the test. In a practical way, a feasibility study is performed before the test to find the best compromise between:

- the technical requirements expressed for the radiation characterization
- the practical limitations related to the activity described below

For this, the feasibility study should include the following items:

- Experimental configuration
  - Dose level (single dose or achieved in several dose steps)
  - Number and size of tested samples
  - Temperature range for the test
- Test facility
  - Beam parameters (incident energy and flux)
  - Test environment (vacuum, nitrogen, dry air…)
  - Available beam area
  - Available instrumentation (in-situ temperature measurement, electrical measurement on tested samples…)
- Project constraints
  - Allocated budget
  - Planning and delays

The final test plan should propose the best solution with respect to the technical requirements and the test budget.

CONCLUSION

This paper provides a global approach for qualifying a material under irradiative environment and points out the hard points to consider. It provides all information from this experience returns able to help for defining the most suitable test plan as possible and understanding test results. Warnings and recommendations for irradiation test are given based on experimental results.

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