CONTAMINATION STUDY OF SPACE SENSITIVE SURFACES BY PACKAGING MATERIALS

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Abstract. The particulate and molecular cleanliness of sensitive components is a priority in the space industry, and in many others as well. In order to keep a surface state in clean conditions during storage and shipment, stringent requirements are necessary for the packaging: protect the sensitive device from the external environment, and not cross contaminate the component as well. This study focuses on the optimisation of conditioning methods, in order to propose some recommendations for the choice of packaging materials, according to the device sensitivity towards particulate and/or molecular contaminations. A methodology has been set up, including the exposure modes of substrate samples in various flexible and rigid packaging materials, and the development of the associated measurement protocols. A benchmark of the packaging materials tested is available, in terms of particulate and molecular contaminations: it will help the user to select the most appropriate materials for his dedicated applications.

1. INTRODUCTION

The presence of contaminants, organic residues or particles can be very harmful for space components such as optics, mechanisms, detectors, printed circuit boards or thermal control surfaces and alter their nominal functioning. Most of the time those components cannot be cleaned. Therefore, it is essential to protect them along the whole integration chain of each satellite sub-system, in particular during the critical steps of prolonged exposure in the clean rooms, transport and storage outside controlled environment that could last up to 7 years or more.

The specific cautions in terms of packaging are mainly mentioned in two space standards applicable at European level: ECSS-Q-ST-70-01C, reference standard for the contamination control, and ECSS-Q-ST-20-08C, more focused on the handling, transport and storage of equipment. In general, any type of packaging must provide the expected protection according to the technical and environmental specifications of the considered equipment and, if necessary, protect against electrostatic discharge. In addition, it should not induce any risk of contamination, degradation or loss of protection.

Packaging constraints are highly dependent on the cleanliness requirements of the sensitive surfaces to be protected. In general, small items are double packed in bags, custom cut, sealed in air or dry nitrogen. This ensures the level of cleanliness if one of the two packages is damaged but also to keep, after removal of the first envelope, a second clean envelope before entering the controlled environment again. Packaging in which desiccants and humidity indicators are often introduced must be transparent to facilitate the inspection of equipment at key points.

Besides, some guidelines are given for example for the design of containers, used in case of long-term storage: they must be easily cleaned, non-particle generator, pressurised in high purity dry nitrogen. Contamination witnesses may follow the equipment for the duration of storage. Finally, it is specified that packaging materials must be approved against contamination risks. However, some experience feedback has shown that contamination issues related to poor packaging may persist. Despite the recommendations described in the standards, it still happens that some packaging materials are poorly known and therefore misused.

2. STATE OF THE ART

2.1. Defect issues linked to packaging

Several cases of cleanliness anomalies related to materials or packaging methods have been highlighted during CNES surveys or have been the subject of an alert to the attention of the user community. Below are three examples of different origins:

a) Contamination by contact

Membrane boxes are particularly targeted. Depending on the chemical nature of the membrane, residues may be left on optics sometimes inadvertently provided in this type of packaging. Most of the time, the technical datasheet of the box gives poor details about it.

b) Contamination by the evaporation products

A few contamination cases have been the result of outgassing of polymer paints or foams inside containers, often custom made according to the equipment. In fact, any newly polymerized material continues to release, even at atmospheric pressure, more or less volatile chemical species which, in the confined space, can condense on sensitive surfaces. In this case, it is recommended to wait for a "drying" time beyond which the contamination risks are minimized.

b) "Pink poly" alert

In order to provide an optimum protection for static sensitive products such as electronic components, there are specific packaging made antistatic either by surface treatment or by the addition of conductive antistatic agents or surfactants in the current material formulation (polyethylene, nylon or polyester). They are commonly called "pink poly" because often colored pink. Even if the percentage of additives is low, the presence of common antistatic agents of amine or ethoxylated amide types can generate various degradations: contamination, corrosion, incompatibility with other materials (e.g. polycarbonate). An alert by Lockheed Martin has then been published on pink poly. It should be noted that other dangerous products can also be identified with similar consequences, such as corrosive octanoic acid, which may be part of the additives in low density polyethylene.

2.2. Preliminary study

The contamination risk of sensitive elements due to packaging is a big concern and requires a detailed knowledge of the materials used. Therefore, an inventory of the main flexible and rigid packaging used for the storage or transportation of space equipment (by the prime contractors or by the main suppliers of equipment and parts sensitive to contamination) has been made. Some preliminary assessments of the transfer of chemical contaminants were carried out thanks to the technical resources of the CNES Laboratories & Expertise department to evaluate different methodologies of analytical chemistry (infrared analysis after contact and conditioning test, chromatographic analysis coupled with mass spectrometry or GCMS to identify species desorbed at temperatures representative of the use ...). At the end of this upstream study, reservations had already been made regarding certain materials.

The objective of the new study is to deepen the results by exploring other methodologies and control techniques for both molecular and particulate contaminations, taking into account a wide variety of packaging materials.

3. SELECTION OF THE SUBSTRATE SAMPLES

The samples tested in this study were selected so that they are representative of the sensitive materials of the space (optical instruments for example), and easily exploitable in terms of contamination characterization (see figure 1). We have chosen:

- silica (SiO_2) substrates, 25 and 30-mm diameters
- germanium (Ge) substrates, 30-mm diameter
- silicon (Si) substrates, 25 and 30-mm diameters

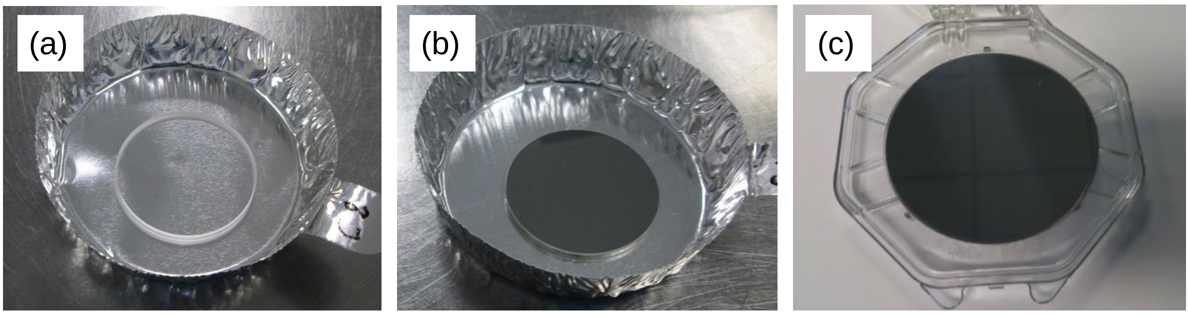


Figure 1: Samples of silica (a), germanium (b) and silicon (c) substrates

Type of bag	Designation	Materials	Features
	LDPE	Low Density PolyEthylene	- CL100 or 1000 (according to IEST-STD-CC1246E) - presence of slip agent likely
	HDPE	High Density PolyEthylene	- CL100 (according to IEST-STD-CC1246E) - no slip agent
	EAE	PolyEthylene-polyAmide-polyEthylene	- CL 1000 (according to IEST-STD-CC1246E) - presence of slip agent likely
	ULO-poly	Ultra Low Outgassing (polyethylene)	- CL100 – 50A/10 (according to IEST-STD-CC1246E) - low outgassing, no additive
	ULO-nylon	Ultra Low Outgassing (nylon)	
	Pink poly	Low Density PolyEthylene	- ESD S20.20 and EN 61340-5-1 - static dissipative - presence of additive (color)
	Poly-alu	Polyethylene-aluminium (standard reference)	- ESD S20.20 and EN 61340-5-1 - static dissipative - good barrier to humidity
	JPL film	Polyethylene-aluminium (JPL reference)	

Table 1: List of bags and films tested in the framework of the study

4. SELECTION OF THE PACKAGING SAMPLES

Regarding packaging, we wanted to test those that are commonly used in the various high-tech industries (Microelectronics, Medical, Space), as well as some references that may have promising features.

4.1. Bags and films tested in the framework of the study

Seven different materials were selected, including two separate references for the polyethylene-aluminium film: a standard reference (poly-aluminium) and a reference approved by the JPL (Jet Propulsion Laboratory). Two ULO (Ultra Low Outgassing) references were integrated into the study (ULO-poly and ULO-nylon), since they are supposed to have a low outgassing in terms of molecular contamination. All references of bags and films tested are presented in the table 1.

4.2. Boxes tested in the framework of the study

Three types of boxes were considered in this study, each having a predominant use by activities (Space, Microelectronics and Medical). All of the box references tested are presented in the table 2.

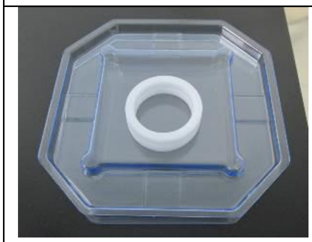
Type of bag	Designation	Materials	Features
	Membrane box	- Polycarbonate (lid and base) - Phthalate (membranes)	- intended to transport fragile and/or shock-sensitive parts, by maintaining with the membranes
	Wafer box	Polypropylene	- intended to transport silicon wafers for Microelectronics
	PETG blister	PolyEthylene Terephthalate Glycol	- manufactured in ISO 7 environment minimum - intended to transport medical parts (implants, devices)

Table 2: List of boxes tested in the framework of the study

5. METHODOLOGY AND ASSOCIATED ANALYTICAL TECHNIQUES

5.1. Methodology

The main objective of this study is to check the potential impact of a packaging on the sensitive device placed inside this packaging. Therefore, it will be necessary to check the initial cleanliness state of the part and the packaging individually, then the final contaminations after storage of the substrate in the packaging, according to the chosen conditions (see figure 2). This will enable to evaluate the potential transfer of contamination from the packaging to the substrate.

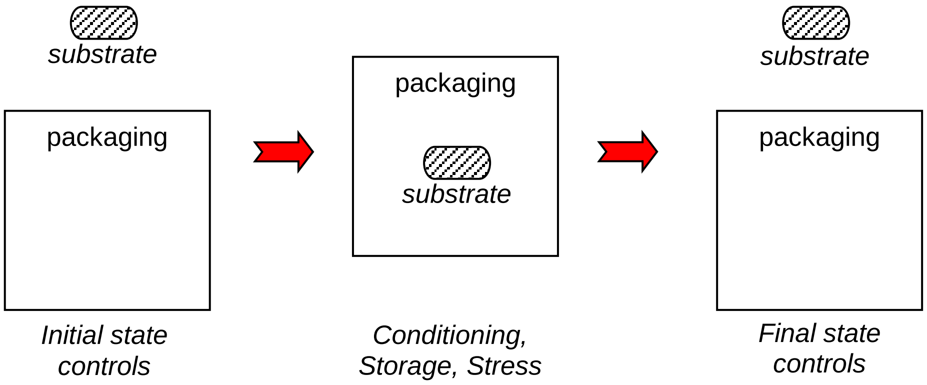


Figure 2: Protocol scheme for the exposure of substrate in the packaging and the associated controls (before and after exposure)

5.2. Accelerated ageing tests

In order to accelerate the ageing process of the materials of the various selected packages, and to demonstrate whether or not a contamination can be transferred (particulate or molecular) from the packaging to the part, we have tested three different modes that stressed the packaging materials and allowed a maximum release of particulate and/or molecular contaminants:

a) heating in oven: exposure at a temperature of 60°C, during a period of 2 days

b) hot-cold cycles: alternation of exposures at 60°C for 30 min and at 5°C for 30 min

c) shaking back and forth: use of a back and forth table, at 120 strokes/min for 30 min

5.3. Control techniques for particulate contamination

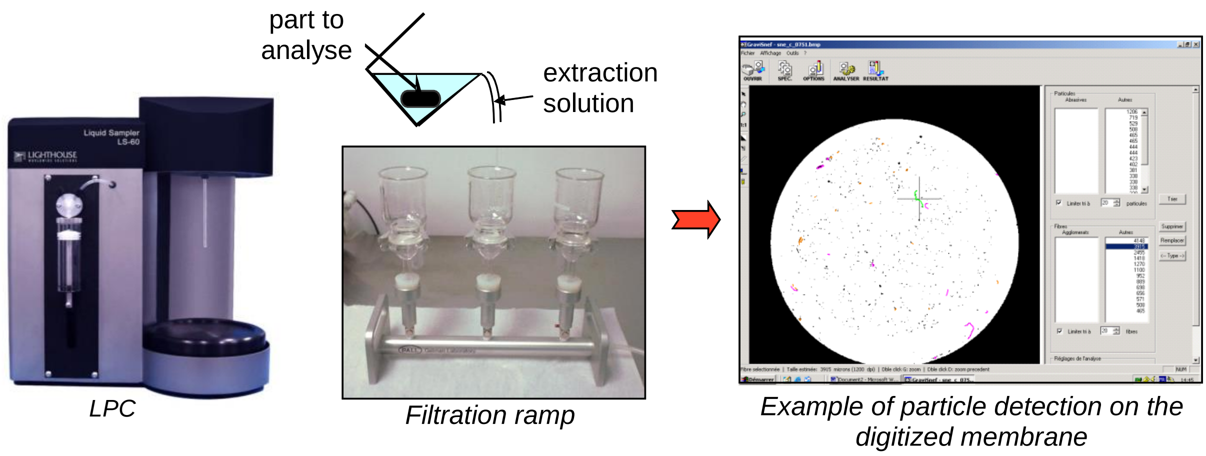


Figure 3: Protocol for the extraction and analysis of particulate contamination

Two complementary techniques were used to control the surface contamination of the parts, after immersion or spraying protocol, according to the type of part (see figure 3):

a) liquid particulate counting (LPC), using an optical counter detecting particles suspended in the extraction solution; the detection range is between 0.5 and 100 µm;

b) particle counting by membrane filtration, followed by particle size analysis, using automatic counting software, which allows a complete mapping of all particles recovered on the membrane; the minimum detection threshold is 10 µm.

For the results of particulate contamination on substrates and packaging, the obscuration rates (in ppm) are reported.

5.4. Control techniques for molecular contamination

The molecular contamination can be detected in two forms: the surface part and the volatile part that can be released via gaseous way. In both cases, the analytical equipment used will be the same, namely GCMS (Gas Chromatography coupled with Mass Spectrometer). The main difference will lie in the sampling protocol, before the analysis (see figure 4):

a) control of the surface organic contamination, via leaching or swabbing of the part with an appropriate solvent

b) control of the volatile organic contamination, via static sampling (exposure during several days) or via dynamic sampling (in an outgassing micro-chamber, with a gaseous flow)

For the results of molecular contamination on substrates and packaging, the concentrations per sampled surface (in µg/cm^2) are reported.

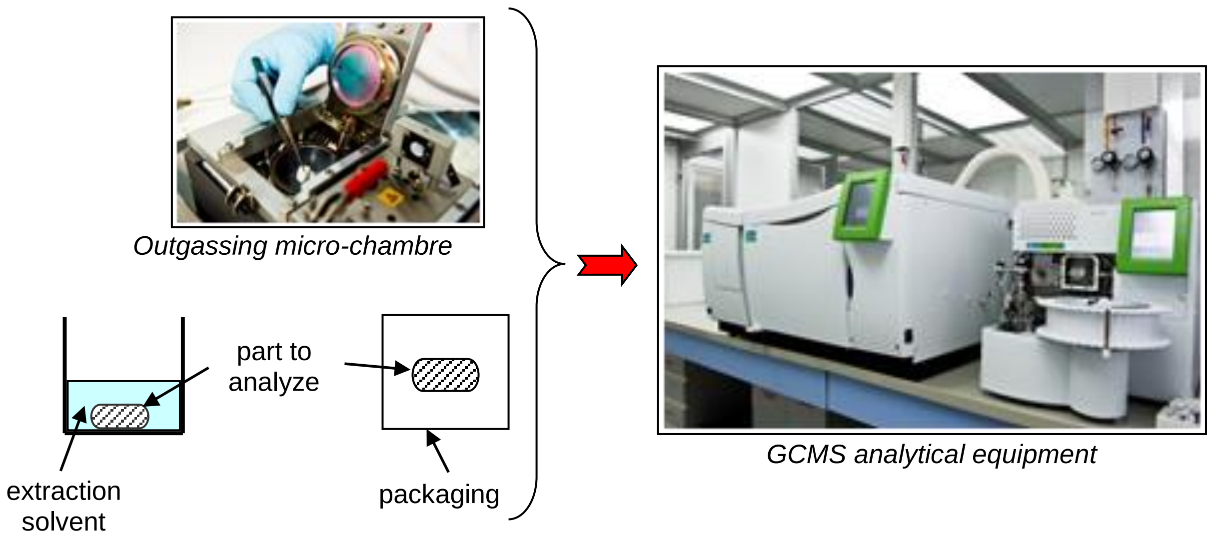


Figure 4: Protocol for the extraction of molecular contamination and analysis via GCMS

6. RESULTS

6.1. Results from the particulate contamination tests

a) <u>Results from the packaging samples tested in terms of particulate contamination</u>

On all the packages tested during the study, we carried out controls on a minimum of 3 samples, before (witness) and after exposure (exposed) of substrates, in order to find an average, in terms of particulate contamination. From all the results obtained, we can see a trend in the measured particulate contamination, or even propose a cleanliness ranking among the tested packages, both on the witness samples (see figure 5) and on the packages that have been exposed with the substrates (see figure 6).

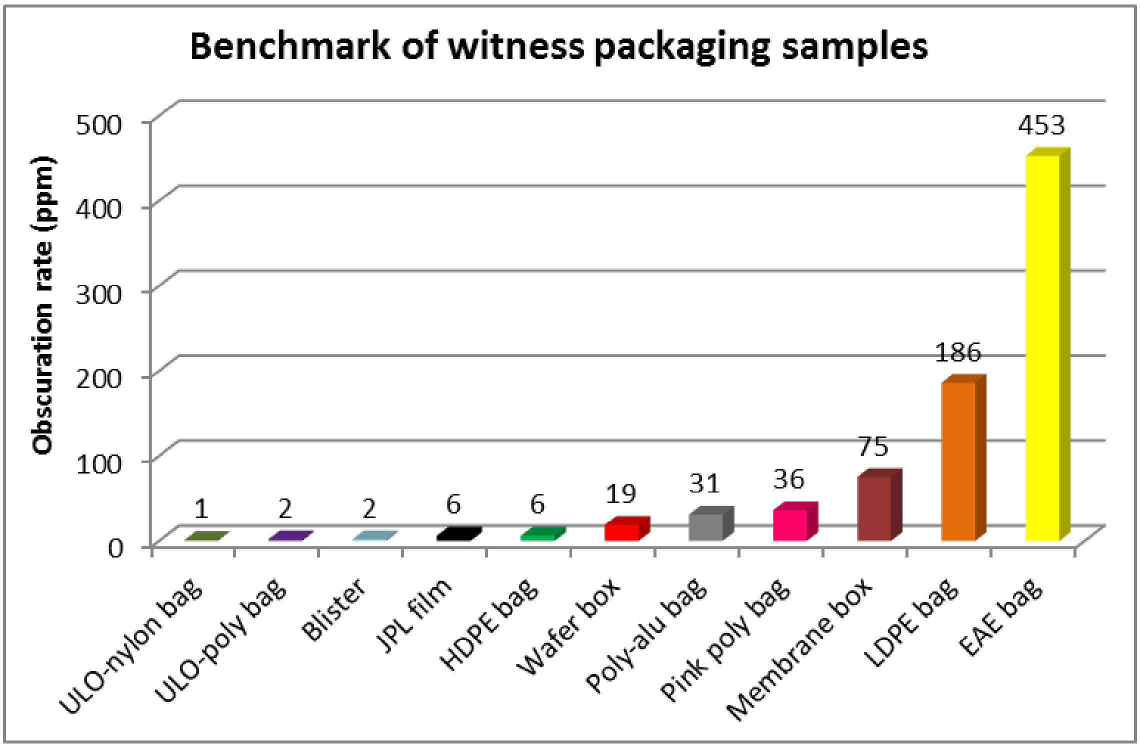


Figure 5: Particulate contamination results from the witness packaging samples

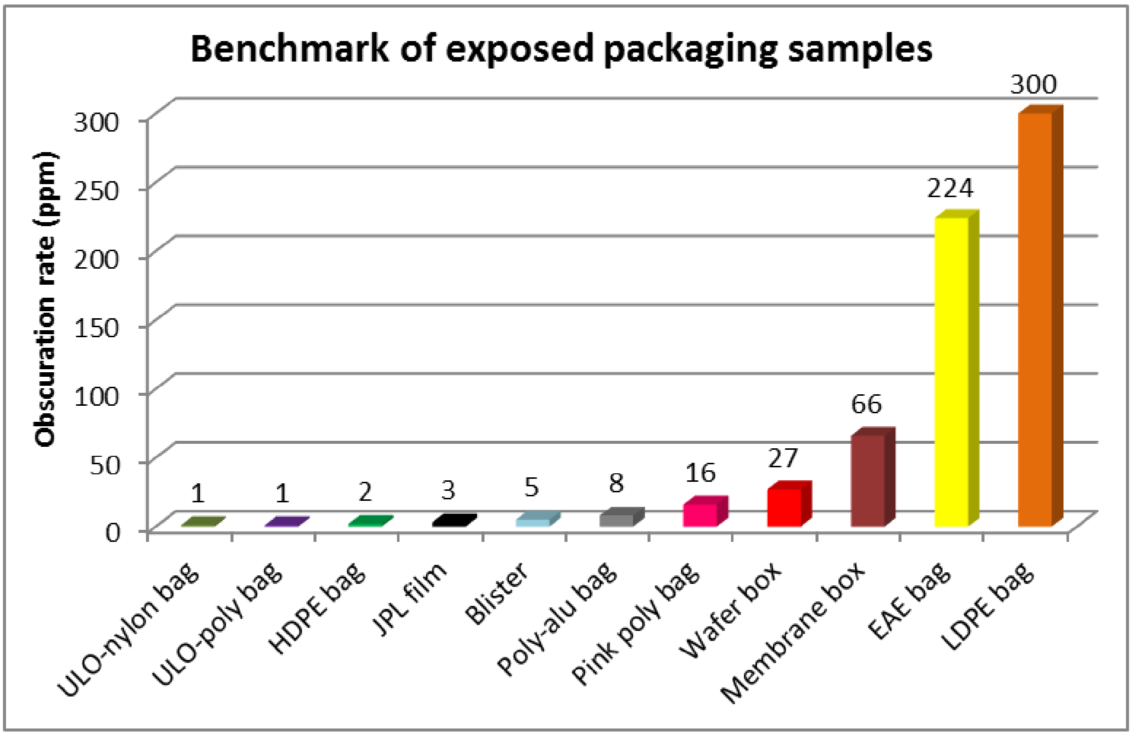


Figure 6: Particulate contamination results from the exposed packaging samples

b) Results from the exposure tests on silica substrates

The results from the exposure tests on the silica substrates in the various packages are calculated from the difference average of obscuration rates (amount of ppm added) between the values measured on exposed substrates and the witness values (see figure 7). Although the values obtained remain relatively low, compared to the particulate contamination measured on the packaging, there is quite a similar trend in the classification of packages by level of cleanliness, the three most contaminant ones being: EAE, LDPE and membrane box.

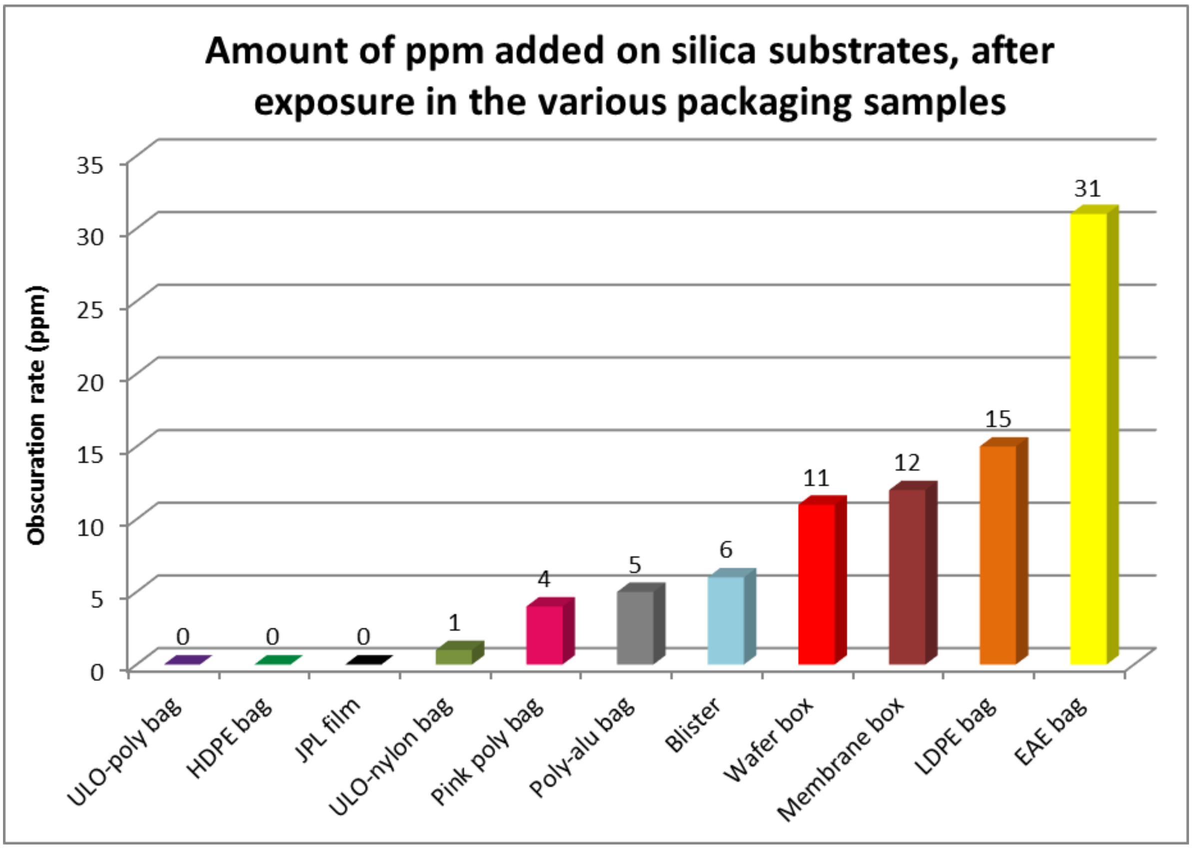


Figure 7: Particulate contamination added on the silica substrates, after exposure in the various packaging samples

c) Results from the exposure tests on silicon substrates

In the same way as for the silica substrates, the results from the exposure tests on the silicon substrates in the various packages are calculated from the difference average of obscuration rates (amount of ppm added) between the values measured on exposed substrates and the witness values (see figure 8). In the case of silicon wafers, the trend appears to be slightly different in the ranking. However, there was only one sample tested per packaging reference.

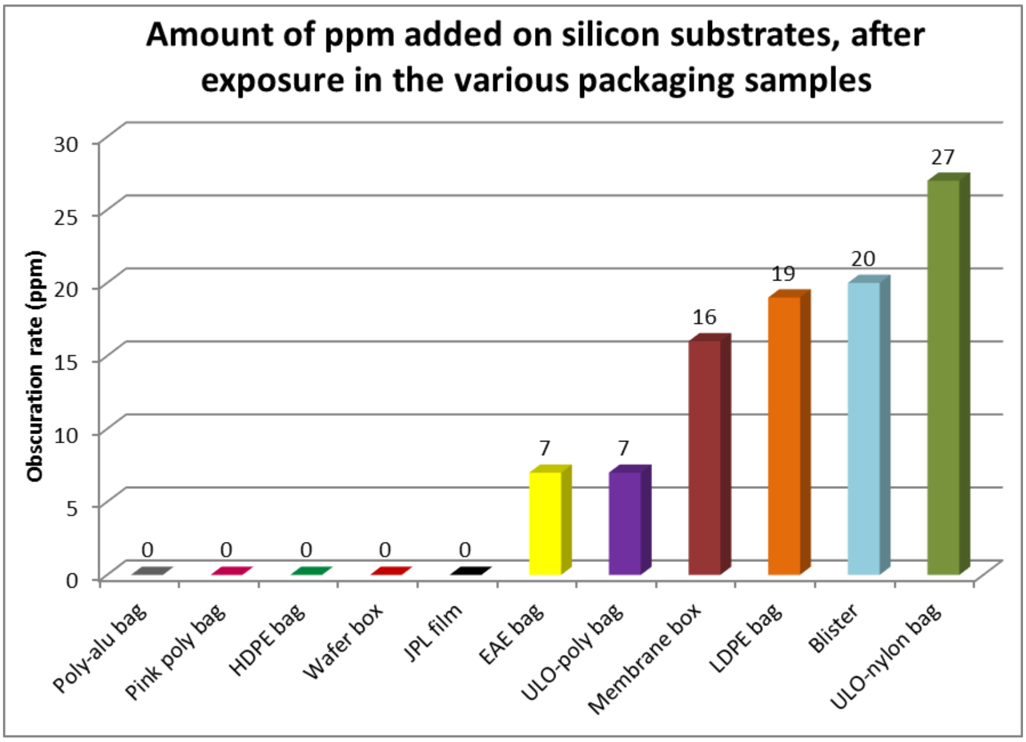


Figure 8: Particulate contamination added on the silicon substrates, after exposure in the various packaging samples

6.2. Results from the molecular contamination tests

a) Results from the packaging samples tested in terms of molecular surface contamination

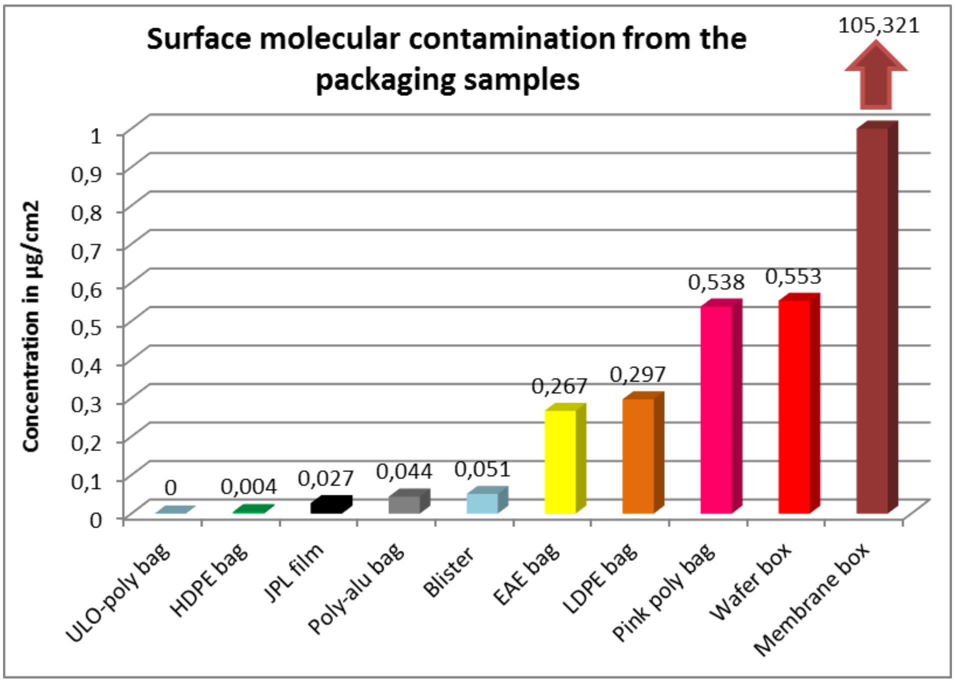


Figure 9: Surface molecular contamination results, after solvent leaching of the various packaging samples

The tests carried out for the evaluation of the surface molecular contamination of the packages were carried out by direct solvent (methanol) leaching of the various samples. All the results obtained are presented in the figure 9, allowing a classification of the tested samples in three categories:

- ULO-poly, HDPE, JPL film, poly-alu and blister -> low contaminating packaging
- EAE, LDPE, pink poly and wafer box -> moderately contaminating packaging
- Membrane box -> high contaminating packaging

Obviously, it is the membrane box that has the highest concentration in terms of surface contamination (mainly composed of phthalate compounds, as well as other relatively heavy compounds in significant concentration); which is logically due to the composition of the membranes.

b) <u>Results from the packaging samples tested in terms of outgassing</u>

The tests carried out for the evaluation of the volatile molecular contamination of the packages were carried out by outgassing cut samples inside a micro-chamber, combining heating and gaseous flow. Lower concentrations (about one order of magnitude) are observed compared to the values obtained by leaching. All the results are presented in the figure 10, allowing a classification of the tested samples in two categories:

- blister, poly-alu, JPL film and wafer box -> low contaminating packaging
- HDPE, ULO-poly, membrane box, EAE, LDPE and pink poly -> moderately contaminating packaging

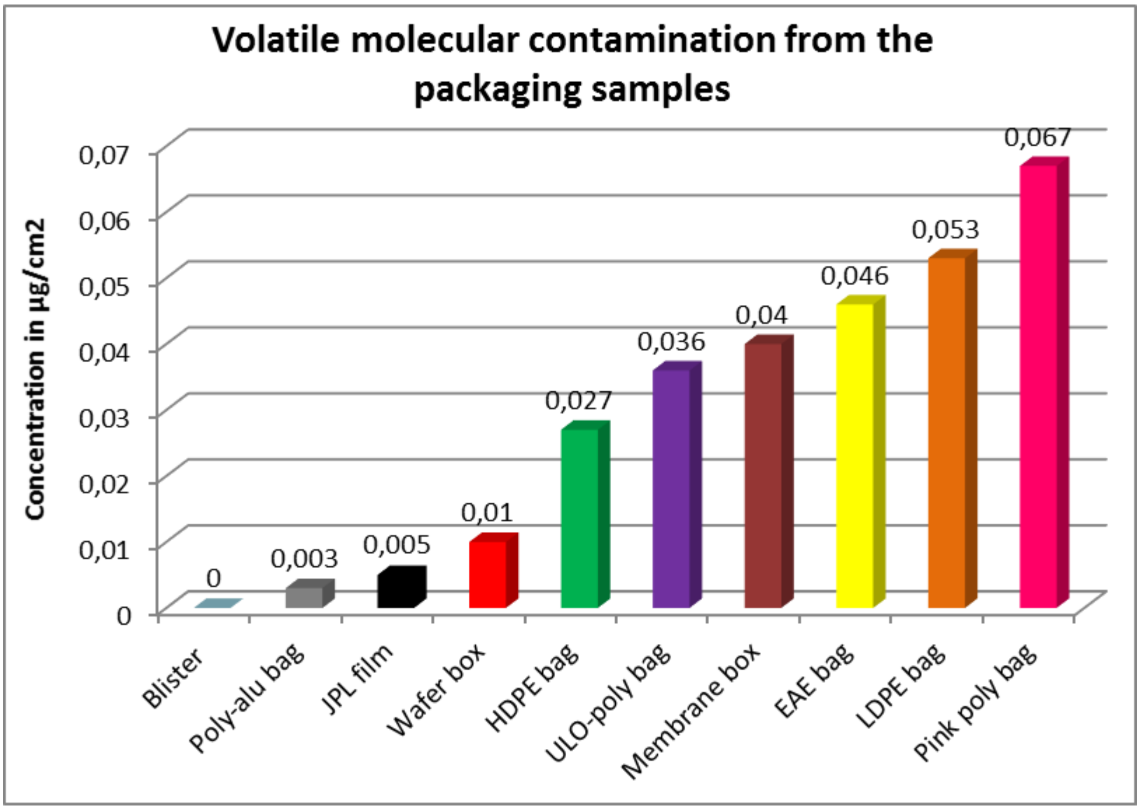


Figure 10: Volatile molecular contamination results, after outgassing of the various packaging samples

c) <u>Results from the exposure tests on silica substrates by heating</u>

The results from the exposure tests of the silica substrates in the various packages, by heating in an oven at 60°C for 2 days, are shown in the figure 11. Only the references EAE, membrane box and wafer box have revealed detectable compounds on the exposed substrates, enabling to say that those three references can be considered as most contaminating.

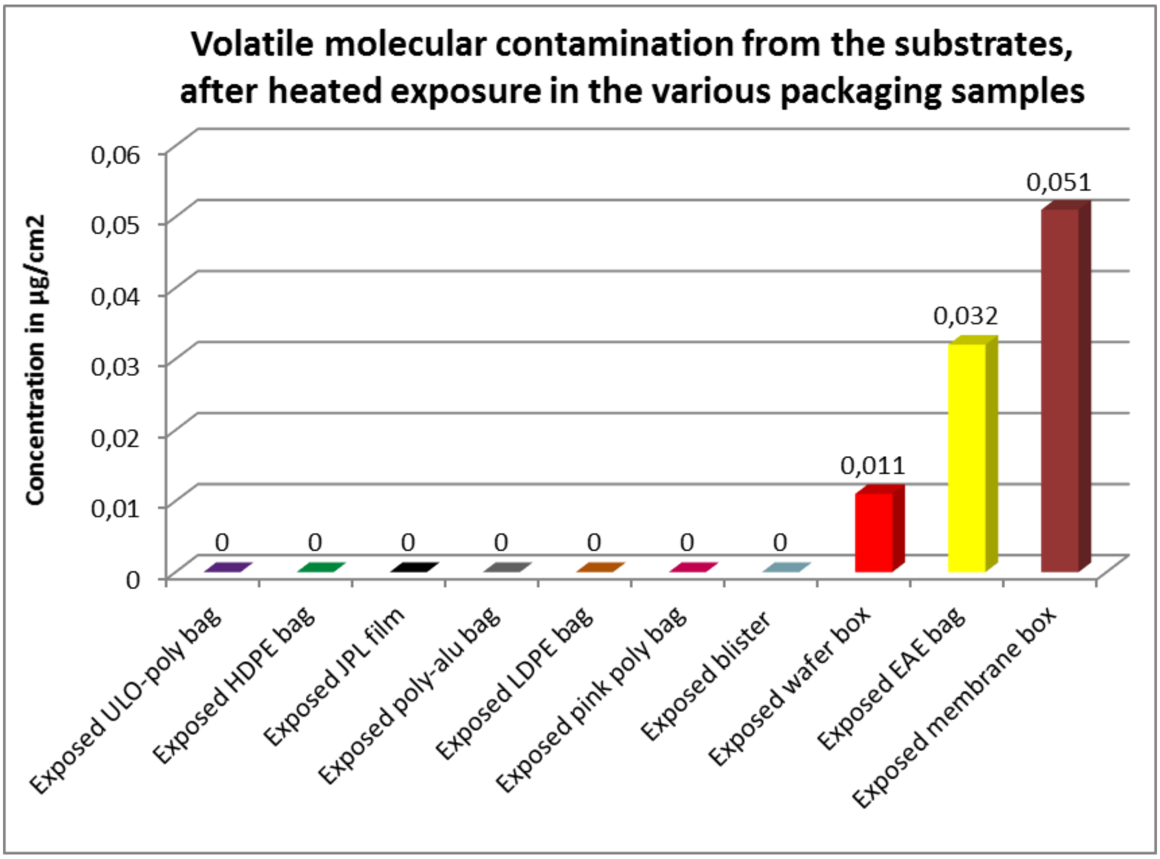


Figure 11: Volatile molecular contamination results from the outgassing of substrates exposed by heating in the various packaging samples

d) <u>Results from the long-term exposure tests on germanium and silicon substrates</u>

For this long-term substrate exposure experiment, we focused on a series of tests on the 3 study boxes, with germanium substrate exposure, and a series of tests on 3 selected packages (pink poly bag, JPL film and ULO-poly bag), with silicon substrate exposure. The substrates have been exposed in the various packages for a period of 2 months, at room temperature. All the results obtained are shown in the figure 12. Only the membrane box and wafer box references revealed compounds detectable on exposed substrates for 2 months.

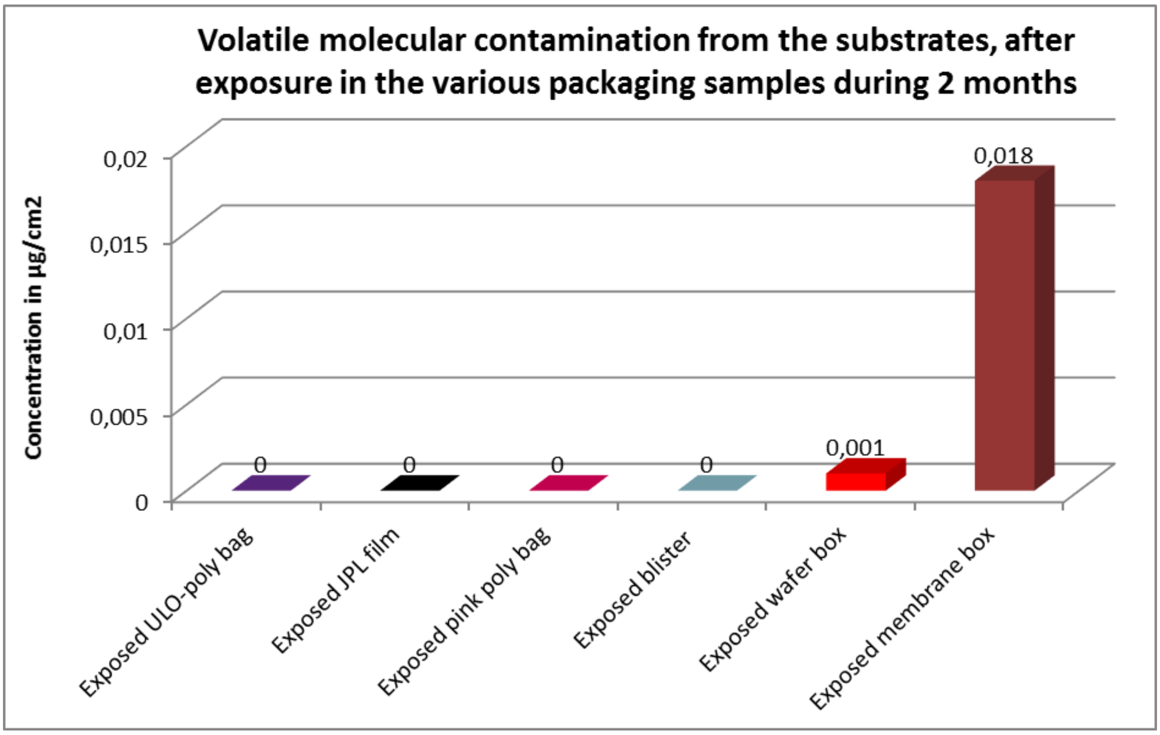


Figure 12: Volatile molecular contamination results from the outgassing of substrates exposed in the various packaging samples during 2 months

7. CONCLUSIONS

During this study, we have been able to highlight the need to use packaging with a high level of cleanliness, addressed to sensitive parts. In addition, it is also important to ensure that the packaging used guarantees the integrity of the device, in particular that it does not present any risk of contamination of the part by the packaging, during the transport and storage stages. Three types of substrates were considered for the tests to be conducted: silica, germanium and silicon. They are representative of the common sensitive materials on space equipment, and also easy to handle in terms of contamination control. We also selected a list of relevant packaging materials (bags, films and boxes) to be evaluated in this study. Among them, there are:

- those which are commonly used for space parts: pink poly, poly-alu, JPL film, membrane box;
- those which are used preferentially in the Microelectronics or Medical sectors: LDPE, HDPE, EAE, wafer box, blister pack;
- as well as two ULO (Ultra Low Outgassing) references: ULO-poly and ULO-nylon, with assumed low release of molecular contamination.

A methodology has been set up to expose the substrates in the selected packaging and to evaluate the contamination levels of the packaging and substrates, before and after exposure. Depending on the type of contamination concerned, the conditioning modes for the substrates have been defined, in order to promote a maximum release from the packaging. In parallel, methods for controlling particulate and molecular contaminations have been developed.

In terms of particulate contamination, an assessment was carried out on the selected packaging before and after exposure of substrates, as well as on exposed substrates. A ranking of all the tested packages is therefore proposed, based on the result of the cumulative particulate contaminations measured for each experiment. We can even find a classification in three categories (from the cleanest to the less clean), in terms of particulate cleanliness:

1) low contaminating packaging: HDPE < JPL film < ULO-poly
2) moderately contaminating packaging: ULO-nylon < blister < poly-alu < pink poly < wafer box
3) high contaminating packaging: membrane box < LDPE < EAE

Regarding the molecular contamination, an assessment was carried out on the selected packaging before exposure, as well as on the exposed substrates. A similar ranking can be proposed for the tested packages, based on the result of the cumulative molecular contaminations measured for each experiment. In the same way, we can find a classification in three categories (from the cleanest to the less clean), in terms of molecular cleanliness:

1) low contaminating packaging: HDPE < JPL film < ULO-poly < poly-alu < blister
2) moderately contaminating packaging: EAE < LDPE <wafer box < pink poly
3) high contaminating packaging: membrane box

Thus, there are three references of packaging bags that are best positioned in terms of cleanliness. These are: HDPE, JPL film and ULO-poly, with the lowest levels in terms of release, all contaminations combined. On the other hand, the references pink poly, LDPE and EAE are the least favourable in terms of use since they have a relatively significant level of release.

Regarding the boxes, it is the reference blister which is found the best positioned, with the lowest rate of release, all contaminations combined. The wafer box is at an intermediate level, while the membrane box is the most detrimental for a "clean" use, especially on molecular contamination.

In addition, a long-term exposure experiment of 2 months, among the tested packages, revealed that the following references did induce little or no transfer of molecular contamination on the exposed substrates: ULO-poly, JPL film, pink poly, blister, wafer box; unlike the membrane box which showed a significant transfer of molecular contamination.

From all these results, we are able to suggest some recommendations regarding the use of packaging for the most critical parts. The references HDPE, JPL film and ULO-poly are the most favourable ones, in order to guarantee the highest level of cleanliness of the parts, when storing or transporting sensitive materials. It is the same for the reference blister, which proves to be the most favourable material for boxes.

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