MAP® EPOX BLACK ESD: LOW OUTGASSING ELECTRICALLY CONDUCTIVE EPOXY PRIMER AND COATING

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ABSTRACT

MAP® EPOX BLACK ESD is a low outgassing black electrically conductive epoxy-based coating validated for use in space. This product can be used as a bonding primer on the aluminum panel skins of structural panels or for any bonding purposes. This product can also be used as a thermo-control coating as its solar absorptance, and emissivity can be as high as 0.95 and 0.90, respectively.

This paper summarizes the validation tests which have been done so far to characterize the MAP® EPOX BLACK ESD coating.

1. INTRODUCTION

Since its creation in 1986, MAP SPACE COATINGS has developed numerous products for the space industry. Most of these products are adhesives, varnishes, coatings, or greases based on silicone, polyurethane, or epoxy technologies.

A new bonding primer called MAP® EPOX BLACK ESD has been developed. This product is a low outgassing black electrically conductive epoxy-based coating. This primer can be used on the aluminum panel skin, for any bonding purposes or on parts which need specific thermo-optical properties.

MAP® EPOX BLACK ESD has been characterized according to the following qualification plan:
1. Control of the product at initial stage.
2. Ageing tests.

This paper first presents the properties of MAP® EPOX BLACK ESD at initial state. Finally, the results after ageing tests are presented.

2. MATERIALS, PROCESSES AND TECHNIQUES

2.1. Materials and processes

MAP® EPOX BLACK ESD is a two-component epoxy primer. The base is a mix of epoxy resin, pigments and several additives which give it its electrical, rheological, and mechanical properties. The hardener is composed of specific reagents. Base is 21.6% solids contents, whereas hardener is 9.2% solids content products.

To obtain the final material, it is necessary to mix the base and the hardener in the respective weight proportions of 58 to 42.

After mixing the base and the hardener, the mix is then filtered on a 25 µm sieve before application.

The mix is then applied using a spray gun pulverization. For instance, a Kremlin S3 spraying gun [1] with an AM head and a 12 nozzle can be used with the following parameters (Table 1). To reach the final thickness (5 to 15 µm), 2 to 3 crossed coats are necessary depending upon the complexity, shape and size of the parts.

Table 1. Spray gun parameters.

Parameter	Value
Below output	1.5 turns
Output	2.5 turns
Pressure	2.0 bars
Vector gas	Oil free, compressed air

Regarding the application conditions, the nominal values are listed in Table 2.

Table 2. Nominal application conditions.

Parameter	Nominal conditions
Temperature	18°C ≤ T ≤ 25°C
Relative hygrometry (RH)	40 % ≤ RH ≤ 60 %

The standard curing process corresponds to a minimum of 4 h at 23°C and 55% RH to allow the solvents to evaporate, then a further minimum time of 1h at 125°C.

Before application, the following surface preparation according to a MAP SPACE COATINGS internal procedure [2] has been used:

- Degreasing with acetone
- Abrasion using scotch brite pad
- Degreasing with acetone.

In order to evaluate the adhesion properties of MAP® EPOX BLACK ESD when used as a primer for bonding application, lap-shear tests were carried out using EC 9323-2 [3] and Redux® 312/5 [4] adhesives. The bonding process with EC-9323 B/A adhesive was done following a MAP SPACE COATINGS internal procedure [5] based on NF-EN 1465 standard [6]. 2024-T3 aluminum samples of 1.6 mm thick were used. The average thickness of the adhesive used was around 155 µm. The adhesive was cured with the following curing cycle: 24h at 23°C + 2h at 65°C.

The bonding process with Redux® 312/5 adhesive was carried out by HEMERIA. The bonding operation is performed at 125 °C. The average thickness of the adhesive used was between 100 and 140 µm. In this case, the surface preparation was done according to a specific procedure used by Hemeria [7]. The adhesive was cured with the following curing cycle: 1h at 120°C further to the TDS of the product [4].

2.2. Techniques

Most of the characteristics were measured in-house by MAP according to the following ISO standards included in the reference section:

- Solids content [8];
- Density using a pycnometer [9];
- Viscosity [10] and pot-life using an Afnor cup [11];
- Solar absorptance [12];
- Infrared emissivity [12];
- Electrical measurements [13];
- Adhesion [14] with Scapa 8705b tape [15];
- Tensile-shear stress [5,6]

The tensile-shear tests of the samples bonded with Redux® 312/5 adhesive was carried out at HEMERIA at initial state and after ageing tests. The tensile-shear tests of the samples bonded with EC9323 B/A were carried out by MAP SPACE COATINGS.

For the ageing tests, CNES carried out adhesion tests.

Outgassing rates are measured according to the ECSS-Q-ST-70-02C standard [16]. The measurements were taken at Airbus Stevenage.

Theoretical calculations were used to define the coating consumption, the weight of the dry film and the volatile organic content (V.O.C.) [17].

2.3 Ageing tests

Two kinds of ageing tests were carried out. Firstly, cumulative ageing tests were carried out in accordance with ECSS-Q-ST-70-17C [18] to validate the MAP® EPOX BLACK ESD as a coating: Damp heat test + Thermal cycling under vacuum + Thermal cycling at atmospheric pressure.

Secondly, cumulative ageing tests were carried out on MAP® EPOX BLACK ESD as a bonding primer: Damp heat test + Thermal cycling at atmospheric pressure. These tests were defined based on the industrial specifications.

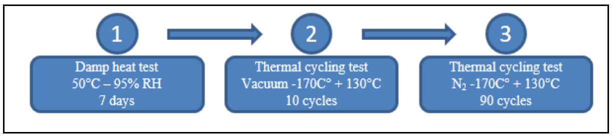
The description of both types of ageing tests is written below.

2.3.1. MAP® EPOX BLACK ESD as a coating

Ageing tests were carried out at the CNES facility. Ageing tests are composed of three cumulative steps [18]:

1) A damp heat test was done at 50°C and 95% RH for 7 days.
2) Thermal cycling tests were performed under vacuum. Ten cycles were performed between -170°C and 130°C with a 10-minute plateau at high and low temperatures (gradient = 5°C/min).
3) Thermal cycling tests were performed under N_2 atmosphere. Ninety cycles were performed between -170°C and 130°C with a 10-minute plateau at high and low temperatures (gradient = 5°C/min).

Figure 1. Schematic principle of cumulative ageing tests for coatings validation



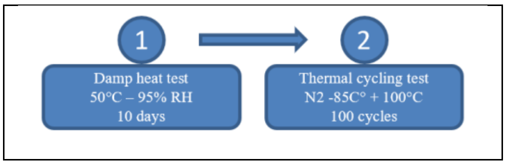
Samples were composed of (1) 2024-T3 alloy plates which were 80 mm x 80 mm in size, with a thickness of 2 mm.

2.3.2. MAP® EPOX BLACK ESD as a bonding primer

Ageing tests were carried out at the CNES facility. Ageing tests are composed of two cumulative steps:

1) A damp heat test was done at 50°C and 95% RH for 10 days.
2) Thermal cycling tests were performed under N_2 atmosphere. One hundred cycles were performed between -85°C and 100°C with a 10-minute plateau at high and low temperatures (gradient = 5°C/min).

Figure 2.Schematic principle of cumulative ageing tests for bonding primer validation.



Samples were composed of an assembly of two 2024-T3 alloy plates coated with MAP® EPOX BLACK ESD and bonded with Redux® 312/5 adhesive. The size of the samples was 105 mm x 25 mm with a thickness of 1.6 mm

2.4. Storage tests

To evaluate the ability of the MAP® EPOX BLACK ESD use as a bonding primer a long time after application, the following tests were carried out.

First, bonding with Redux® 312/5 was carried out 3 months after MAP® EPOX BLACK ESD application.

Second, MAP® EPOX BLACK ESD coated samples were exposed 10 days under damp heat at 50°C and 90% RH before bonding with Redux® 312/5. This test is used as a simulation of storage in clean rooms with the assumption based on several works that 7 days of damp heat test under the previous temperature and hygrometry conditions simulate 9 years of storage in clean rooms. [19].

3. QUALIFICATION PLAN

In order to qualify the MAP® EPOX BLACK ESD, its characteristics must meet the requirements listed in Table 3. These requirements come from the characteristics of an antistatic coating and from the ECSS-Q-ST-70-02C outgassing standard [16].

Table 3. Requirements for MAP® EPOX BLACK ESD.

Properties	**Requirements**
Thickness (µm)	5 - 15
Electrical surface resistance on metallic substrate (Ω/☐)	$< 10^6$
RML[1] (%)	≤ 1
CVCM[2] (%)	< 0.1

Characterization of the products was performed at the initial state for all the characteristics: rheological, mechanical, outgassing, and electrical properties.
Some of the characteristics (adhesion, solar absorptance and electrical surface resistance) were characterized after a damp heat test (7 days at 50°C and 95% RH) and after a cumulative damp heat test + thermal cycling (10 cycles between -170°C and 130°C under vacuum + 90 cycles between -170°C and 130°C under N_2 atmosphere).

4. RESULTS

4.1. Initial state

4.1.1. General properties

The density of MAP® EPOX BLACK ESD was measured using a pycnometer further to ISO 2811-1 standard [9]. The value was measured at 0.95 for the base / hardener mix. The solids content value was measured according to ISO 3251 [8] standard and is equal to 16.4 % (Base/Hardener mix).

[1] RML: Recovered mass loss

[2] CVCM: Collected volatile condensable material

The values of the viscosity measurements and pot-life are listed in Table 4. The viscosity has been measured at 23°C.

Table 4. Rheological properties measured at 23°C for MAP® EPOX BLACK ESD.

Properties	**Value**
Viscosity Afnor cup n°4 (Base + Hardener)	10.5 ± 3.0 s
Pot-life	> 1 h

All the properties and characteristics are listed in Table 5.

Table 5. General properties of MAP® EPOX BLACK ESD – measurement at 23°C.

Properties	**MAP® EPOX BLACK ESD**
Typical thickness	5 – 15 µm
Density	0.95 ± 0.05
Solids content	16 ± 2 %
Viscosity Afnor cup 2.5 at 23°C	27.5 ± 5.0 s
Viscosity Afnor cup 4 at 23°C	10.5 ± 3.0 s
V.O.C.	794 $g.L^{-1}$
Theoretical consumption	11 $m^2.Kg^{-1}$ (8 µm thick)
Theoretical dry film weight	1.9 $g.m^{-2}.\mu m^{-1}$

4.1.2. Functional properties

Solar absorptance of MAP® EPOX BLACK ESD was measured around 0.94, whereas infrared emissivity was around 0.83 for a 7 µm thick coating and 0.90 for a 14 µm thick coating. The measurements were carried out according to ECSS-Q-ST-70-09C [12] on aluminum substrates.

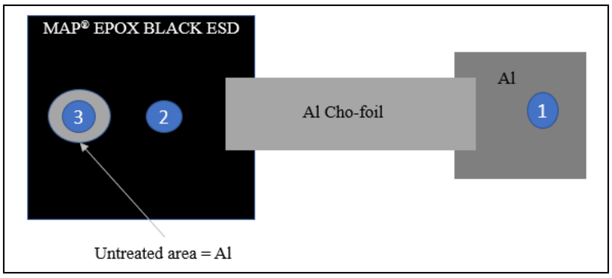
The electrical surface resistance was measured around 5 x 10^3 Ω/□ on aluminum substrate for a 7 µm thick coating and around 28 x 10^3 Ω/□ on aluminum substrate for a 14 µm thick coating.

Table 6. Electrical surface resistance for MAP® EPOX BLACK ESD on metallic substrate (2024-T3 alloy).

Thickness (µm)	**Rs (x 10^3 Ω/•)**
7	5
14	28

Additional measurements were done in the following configuration. The measurements were performed using a megohmmeter with a voltage under 50V.

Fig 3. Configuration used for the electrical measurement.



Two coupons were used for each thickness samples of MAP® EPOX BLACK ESD: 7 µm and 14 µm. The measurements were carried out between #1 and #2 areas (“Measure 1–2” in Table 5) and between #1 and #3 areas (“Measure 1-3” in Table 5). For each coupon, three measurements were done. The average measurements are listed in the table below.

Table 7. Electrical measurements in configuration described in Fig. 2.

Coupon	**Thickness (µm)**	**Measure 1 – 2**	**Measure 1 – 3**
#1	7	19 ± 8 Ω	17 ± 6 Ω
#2	7	13 ± 3 Ω	12 ± 4 Ω
#1	14	15 ± 2 Ω	13 ± 1 Ω
#2	14	22 ± 3 Ω	16 ± 2 Ω

The outgassing properties were measured at the Airbus Stevenage facility on a product after (1) 4 h at 23°C and 55% relative hygrometry to allow solvents to evaporate and then cured for 1 h at 125°C. The results are listed in Table 8 [20].

Table 8. Outgassing results for MAP® EPOX BLACK ESD.

Curing cycle	TML (%)	RML (%)	CVCM (%)
4h at 23°C + 1h at 125°C	1.29	0.82	0.05

Adhesion of MAP® EPOX BLACK ESD on aluminum is 0 Class according to ISO 2409 using Scapa 8705b tape [15].

4.1.3. Tensile-shear stress

In order to evaluate the adhesion properties of the MAP® EPOX BLACK ESD, lap-shear tests were carried out using EC-9323 B/A [3] and REDUX® 312/5 adhesives [4] following MAP internal procedure [20] based on NF-EN 1465 standard [5,6]. 2024-T3 aluminum samples of 1.6 mm thick were used.

4.1.3.1. EC-9323 B/A adhesive

For EC-9323 B/A bonding, the average thickness of the adhesive used was 155 µm. The tensile-shear stress was in the range of 28.7 ± 0.8 MPa (Table 9) with cohesive type failures.

A difference in the tensile-shear stress value mentioned on the technical data sheet (40 MPa) has been observed [3]. Comparative tests have been done using BR 127 NC ESD instead of MAP® EPOX BLACK ESD. The same results of 25.9 Mpa were measured, confirming that the use of sulfochromic etch greatly improves the performances. Indeed, the surface preparation used in these tests were only a degreasing and a light sanding.

Table 9. Properties of tensile-lap shear samples of MAP® EPOX BLACK ESD bonded with EC-9323 B/A adhesive (Initial state) – measurement at 23°C.

Batch	**R.16.06.69**	**BR 127 NC ESD**
MAP® EPOX BLACK ESD Thickness (µm)	7	8
EC-9323 B/A Thickness (µm)	155	140
Tensile-shear stress (Mpa)	28.7 ± 0.8	25.9 ± 0.9
Failure mode	Cohesive	Cohesive

4.1.3.2. Redux® 312/5 adhesive

For Redux® 312/5 bonding, the average thickness of the adhesive used was 11 µm and 13 µm respectively, for 7 µm thick and 15 µm thick MAP® EPOX BLACK ESD. The tensile-shear strengths were 38.7 Mpa and 38.8 Mpa for7 µm and 15 µm, respectively (Table 10) with cohesive type failures for both thicknesses of MAP® EPOX BLACK ESD.
These values are in good accordance with the results obtained with BR 127 NC ESD [21] instead of MAP® EPOX BLACK ESD: 38 MPa.

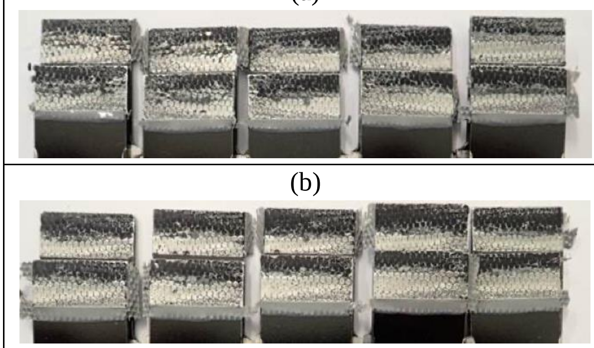
Table 10. Properties of tensile-lap shear samples of MAP® EPOX BLACK ESD bonded with Redux® 312/5 adhesive (Initial state) – measurement at 23°C.

Batch	**R.16.06.72**	**R.16.06.73**
MAP EPOX BLACK ESD Thickness (µm)	7	15
Redux® 312/5 Thickness (µm)	110	130
Tensile-shear stress (MPa)	38.7 ± 0.8	38.8 ± 0.6
Failure mode	Cohesive	Cohesive

Fig 4. Failure facies of 2024-T3 – MAP® EPOX BLACK ESD – REDUX® 312/5 assemblies. Cohesive failure for all the samples (a) Thickness of 7 µm of MAP® EPOX BLACK ESD, batch R.16.06.72 and (b) Thickness of 14 µm of MAP® EPOX BLACK ESD, batch R.16.06.73.

(a)

(b)



4.2 After ageing tests

4.2.1. MAP® EPOX BLACK ESD as a coating

Ageing tests were carried out at the CNES facility for the MAP® EPOX BLACK ESD. Minimum and maximum recorded temperatures during vacuum thermal cycling were the following: -174°C and 132°C, whereas for N_2 thermal cycling, minimum and maximum temperatures were -174°C and 139°C.

MAP® EPOX BLACK ESD was applied at initial state. Samples were applied on 2024-T3 with a thickness of 7 µm (Table 11) and 14 µm (Table 12).

Solar absorptance of MAP® EPOX BLACK ESD is equal to 0.94 for 7 µm thick of MAP® EPOX BLACK ESD, whereas for 14 µm thick it is 0.95.

Emissivity of MAP® EPOX BLACK ESD is equal to 0.83 for 7 µm thick of MAP® EPOX BLACK ESD whereas for 14 µm thick it is 0.90.

Electrical surface resistance is around 5 x 10^3 Ω/☐ for a thickness of 7 µm whereas it increases to around 30 x 10^3 Ω/☐ for 14 µm thick.

Table 11. MAP® EPOX BLACK ESD (Thickness of 7 µm) properties at initial state, after damp heat test and after cumulative damp heat (DH) + thermal vacuum cycling (TVC) + thermal cycling at atmospheric pressure (TCAP) – measurement at 23°C

	t_0	**DH**	**DH + TVC + TCAP**
α_S	0.94	-	0.94
Infrared emissivity	0.83	-	0.84
Rs (x 10^3 Ω/□)	5	-	4
Adhesion	0 class	0 class	0 class

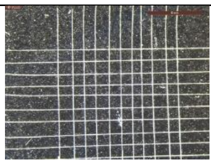
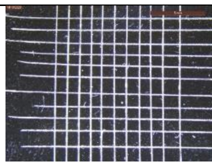
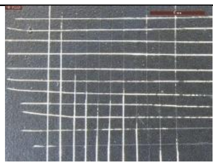
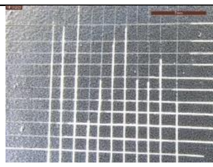
Table 12. MAP® EPOX BLACK ESD (Thickness of 14 µm) properties at initial state, after damp heat test and after cumulative damp heat (DH) + thermal vacuum cycling (TVC) + thermal cycling at atmospheric pressure (TCAP) – measurement at 23°C.

	t_0	**DH**	**DH + TVC + TCAP**
Solar absorptance	0.95	-	0.95
Infrared emissivity	0.90	-	0.90
Rs (x 10^3 Ω/□)	28	-	33
Adhesion	0 class	0 class	0 class

No change in optical properties (solar absorptance or emissivity), nor electrical resistance is observed when exposed to damp heat test and cumulative damp heat (DH) + thermal vacuum cycling (TVC) + thermal cycling at atmospheric pressure (TCAP).
Adhesion is class 0 on 5 at initial state and after damp heat test and cumulative damp heat test + TVC + TCAP.

Optical micrographs of the coating are presented at each step of the ageing test Fig. 5. No adhesion defect was observed at each ageing test step.

Fig 5. Surface micrographs of the MAP® EPOX BLACK ESD at each step of the ageing test showing results after an adhesion test. The distance between each line (cross-cut test) is 1 mm.

	7 µm	14 µm
Initial state		
After damp heat		
After DH + TVC + TCAP		

4.2.2. MAP® EPOX BLACK ESD as a bonding primer

When exposed to ageing test (cumulative DH + TVC + TCAP), tensile-shear stresses of assemblies composed of MAP® EPOX BLACK ESD and Redux® 312/5 adhesive were 37.5 MPa (Table 13) with a cohesive failure mode (Fig. 6). This value is close to those obtained at initial state: 38.7 MPa for 7 µm thick and 38.8 MPa for 14 µm thick MAP® EPOX BLACK ESD. No impact of the ageing test was observed on the tensile-shear stress nor the failure mode.

Table 13. General properties of MAP® EPOX BLACK ESD – measurement at 23°C.

Batch	**R.16.06.72**	**R.16.06.73**
MAP EPOX BLACK ESD Thickness (µm)	7	15
Redux® 312/5 Thickness (µm)	11	13
Tensile-shear stress (MPa)	37.5 ± 1.2	37.-5 ± 1.1
Failure mode	Cohesive	Cohesive

Fig 6. Failure facies of 2024-T3 - MAP® EPOX BLACK ESD – REDUX® 312/5 assemblies. Cohesive failure for all the samples (a) Thickness of 7 µm of MAP® EPOX BLACK ESD, batch R.16.06.72 and (b) Thickness of 14 µm of MAP® EPOX BLACK ESD, batch R.16.06.73.

(a)

(b)

4.3. After storage tests

4.3.1. After DH before bonding

To simulate ageing of MAP® EPOX BLACK ESD, samples of aluminum covered with MAP® EPOX BLACK ESD were exposed to a damp heat test for 10 days at 50°C and 90% RH.

Bonding with Redux® 312/5 adhesive was carried out after damp heat tests. The results are shown in Table 14. Tensile-shear test was 37.2 MPa versus 38.7 MPa at initial state. Failure facies are also cohesive (Fig.5). No influence of the damp heat test of the primer MAP® EPOX BLACK ESD was observed on the tensile-shear stress.

Table 14. Properties of tensile-lap shear samples of MAP® EPOX BLACK ESD bonded with Redux® 312/5 adhesive (After DH exposure) – measurement at 23°C.

Batch	**R.16.06.74**
MAP EPOX BLACK ESD Thickness (µm)	10
Redux® 312/5 Thickness (µm)	130
Tensile-shear stress (MPa)	37.2 ± 1.2
Failure mode	Cohesive

Fig 7. Failure facies of 2024-T3 - MAP® EPOX BLACK ESD – REDUX® 312/5 assemblies. Cohesive failure for all the samples, Thickness of 10 µm of MAP® EPOX BLACK ESD, batch R.16.06.74.

4.3.2. Bonding after 3 months

In industrial conditions, bonding is often carried out several weeks or months after the application of primer. To simulate such industrial application, bonding with Redux® 312/5 was carried out 3 months after the application of MAP® EPOX BLACK ESD.

The results are shown in Table 15. Tensile-shear test was 38.4 MPa versus 38.7 MPa at initial state. Failure facies are also cohesive (Fig.8). No influence of the ageing of the primer MAP® EPOX BLACK ESD was observed.

Table 15. Properties of tensile-lap shear samples of MAP® EPOX BLACK ESD bonded with Redux® 312/5 adhesive (After DH exposure) – measurement at 23°C.

Batch	**R.16.06.74**
MAP EPOX BLACK ESD Thickness (µm)	10
Redux® 312/5 Thickness (µm)	130
Tensile-shear stress (MPa)	38.4 ± 0.8
Failure mode	Cohesive

Fig 8. Failure facies of 2024-T3 - MAP® EPOX BLACK ESD – REDUX® 312/5 assemblies. Cohesive failure for all the samples, Thickness of 10 µm of MAP® EPOX BLACK ESD, batch R.16.06.74.

5.CONCLUSION

MAP® EPOX BLACK ESD is a low outgassing black electrically conductive epoxy-based coating validated for space uses. This product can be used as a bonding primer on the aluminum panel skin or for any bonding purposes. This product can be used also as a thermo-control coating as its solar absorptance, and emissivity can be as high as 0.95 and 0.90, respectively, depending on the thickness. Electrical surface resistance is around 30 x 103 ohm/sq on metallic substrates which allows electrostatic charge flow.

No change in thermo-optical properties, adhesion and electrical surface resistance has been observed when submitted to ageing tests.

Regarding the bonding primer applications, tensile-shear stresses of 28 MPa were measured with cohesive failure mode using EC9323 B/A adhesive using a standard surface preparation. Using Redux® 312/5 adhesive tensile heat stress of 38 MPa were measured at initial state with cohesive failure mode. After ageing tests, no change was observed in tensile-shear stress values nor failure mode.

Storage of the coated coupons with MAP® EPOX BLACK ESD for 3 months before bonding does not show any change in the tensile-shear stress nor failure mode.
Finally, this product can be used as a thermo-control coating or as a bonding primer. MAP® EPOX BLACK ESD can be used as an alternative to BR 127 NC ESD.

6. ACKNOWLEDGEMENTS

The authors want to thank C. Tisseron, S. Lesecq and L. Lapaix from Airbus for their assistance in the evaluation of the product.
The authors wish to thank B. Gabillon, N. Turgné and E. Rondinaud from Hemeria for their assistance in the mechanical tests and characterization of the product.

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