

MAP® SCK5N: WHITE ANTISTATIC COATING

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ABSTRACT

SCK5 is a low outgassing white silicone antistatic coating developed in the 1990s by CNES and MAP using a liquid-liquid purification process and a specific antistatic pigment developed by CNES.

A new white silicone antistatic coating, MAP® SCK5N has been developed to improve the very short shelf-life of the current coating (24 h). The production process has been updated to eliminate the use of organic solvents in the purification process. A new white antistatic pigment has also been developed.

This paper summarizes the validation tests which have been carried out so far to characterize the MAP® SCK5N coating.

1. INTRODUCTION

Since its creation in 1986, MAP has developed numerous products for the space industry. Most of these products are silicone-based greases, adhesives, varnishes or coatings.

SCK5 is a low outgassing white silicone antistatic coating obtained via a liquid-liquid purification process (patented by CNES) that makes it possible to obtain degassing values compatible with space applications [1]. The antistatic properties of the coating are provided thanks to a specific pigment. In order to end the use of organic solvents that do not comply with new European environmental regulations (REACH), the purification process has been modified. For environmental regulation compliance, the white antistatic pigment used until now has been changed.

Moreover, to develop the use of white antistatic coating, the shelf-life of the product has been significantly increased from 24 h to 6 months¹.

The following qualification plan has been defined to evaluate the properties of MAP® SCK5N,

1. Control of the product in the initial stage and comparison of the properties of MAP® SCK5N with the current version SCK5;
2. Ageing tests;

3. Environmental tests.

This paper first presents the properties of MAP® SCK5N in its initial state. These properties are compared to those of the current version SCK5. Secondly, the results after ageing tests are presented and finally, the results after environmental tests are described.

2. MATERIALS, PROCESSES AND TECHNIQUES

2.1. Materials and processes

MAP® SCK5N is a two-component RTV² silicone coating. The base is a mix of silicone polymer, pigments and several additives which give it its electrical, rheological and mechanical properties. The hardener is composed of a mix of silicone cross-linkers. The base has a solids content of 51.2 %, whereas the hardener contains 64.9 % solids content products. To reach the low outgassing rates as defined by the ECSS [1], a solvent-free purification process (CNES-MAP patented process) was used instead of the solvent-based purification process used until now.

To obtain the final material, the base and the hardener are mixed in weight proportions of 91 to 9, respectively. It is mandatory to dilute the mix using MAP® SCK5N thinner to get the right viscosity. Depending on the size and on the shape of the parts, the thinning ratio can be varied 20 ± 5 %.

The mix is then applied using spray gun pulverization. For instance, a Kremlin S3 spray gun [2] with an AM head and a No. 12 nozzle can be used with the following parameters (Table 1). To reach the final thickness (60 to 100 µm), 2 to 3 crossed coats are necessary depending upon the complexity, shape and size of the parts. Sufficient dry must be allowed between coats to achieve a flat appearance.

¹ As of March 2nd, 2020, the shelf-life of MAP® SCK5N is 6 months. The target is a shelf-life of 12 months.

² RTV: Room-temperature-vulcanizing silicone

Table 1. Spray gun parameters

Parameter	Value
Below output	1.5 turns
Output	3 turns
Pressure	2.0 bar
Vector gas	Oil free, compressed air

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

Table 2. Nominal application conditions

Parameter	Nominal conditions
Temperature	$18\text{ °C} \leq T \leq 25\text{ °C}$
Relative	$40\% \leq RH^3 \leq 60\%$

The standard curing process corresponds to (1) Minimum 4 h at 23 °C and 55 % RH to allow the solvents to evaporate, then 15h minimum at 70 °C (Fast-curing process) whereas an alternative process is (2) 8 days minimum at 23 °C and 55 % relative hygrometry.

2.2. Techniques

All the characteristics were measured in-house by MAP in accordance with ISO standards which are presented in the reference section below:

- Solids content [3];
- Density using a pycnometer [4];
- Viscosity [5] and pot-life using an AFNOR cup [6];
- Solar absorptance [7];
- Infrared emissivity [7];
- Electrical measurements [8];
- Adhesion [9].

For the ageing and the environmental tests, the CNES and ONERA also carried out some characterization tests: Adhesion tests were conducted at the CNES and solar absorptance and infrared emissivity measurements were performed at the ONERA.

Outgassing rates were measured in accordance with standard ECSS-Q-ST-70-02C [1]. The measurements were taken at Airbus Toulouse.

³ RH: Relative hygrometry

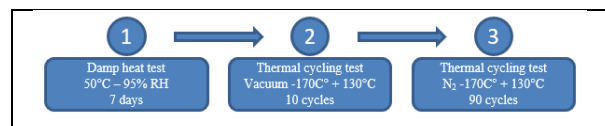
Theoretical calculations were used to define the coating consumption, the dry film weight and the volatile organic content (V.O.C.) [10].

2.3. Ageing tests

Ageing tests were carried out at the CNES facility. Ageing tests are composed of three cumulative steps [11]:

- 1) A damp heat test was conducted at 50 °C and 95 % RH for 7 days;
- 2) Thermal cycling tests were performed under vacuum. 10 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 in an N₂ atmosphere. 90 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



Samples were composed of (1) 2024-T3 alloy plates which size was 80 mm x 80 mm with a thickness of 2 mm and (2) 50 µm thick Kapton[®] HN bonded on metallic plates.

2.4. Environmental tests

2.4.1. GEO tests

The geostationary orbit (GEO) environmental tests were carried out at the ONERA lab in Toulouse. The space environment at GEO is simulated using the SEMIRAMIS irradiation medium (Fig. 2) to reproduce the combined effects of UV photons, electrons and protons in a spatial environment [12].

Figure 2 - SEMIRAMIS irradiation chamber [12]



The proton beam is produced by a 2.5 MeV Van de Graaff accelerator. Protons are emitted from a pure hydrogen plasma and separated from the other charged species by a post-acceleration analysis of their mass. The protons thus accelerated scan the surface of the sample holder, irradiating its contents.

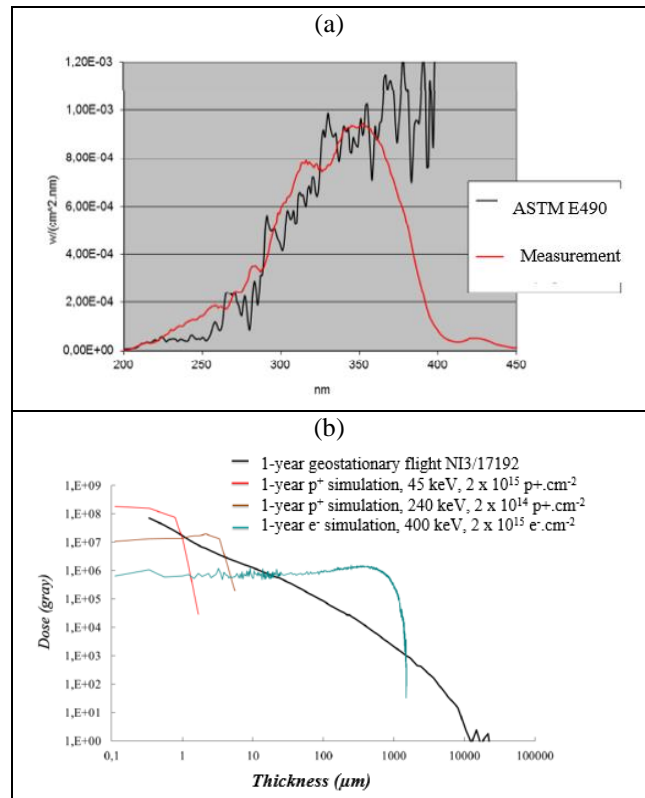
The sample holder is thermo-regulated, which allows the temperature of materials to be maintained at 40 °C throughout the duration of irradiation. The pressure is maintained at 1.6×10^{-6} mbar.

The tests carried out were used to simulate 1 year of exposure in geostationary orbit. The simulation was conducted in the following steps:

1. UV: 1133 equivalent sun hours (ESH);
2. Electrons: 400 keV at 1×10^{15} at/cm²;
3. Protons: 240 keV at 2×10^{14} at/cm²;
4. Electrons: 45 keV at 2×10^{15} at/cm².

The spectral distribution of the UV flux is described in Fig.3a [12] and compared to the solar flux conventionally used and detailed in ASTM E490 [13]. A simulation of the dose versus the thickness of the coating was performed using the GEANT4 calculation for the electron and proton irradiations (Fig.3b) [14].

Figure 3 – (a) UV flux spectral distribution measurement and from ASTM E490 and (b) Simulation of the dose versus the coating thickness



Solar absorptance is measured in a vacuum at the initial state and after each step of irradiation.

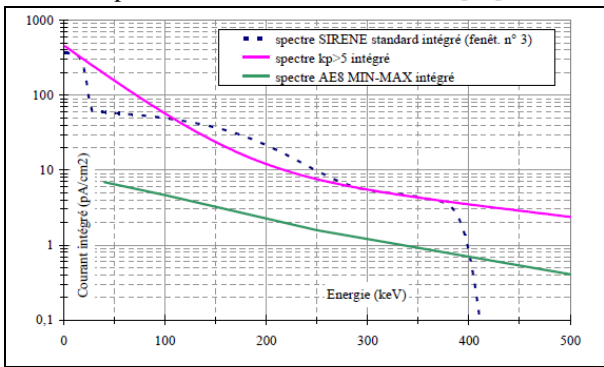
2.4.2. ESD measurements after GEO tests

Electrostatic discharges (ESD) were implemented on MAP® SCK5N samples before and after the GEO irradiation tests [15]. The electrical characterization consists in subjecting the materials in the SIRENE enclosure to a spectrum of standard GEO electrons (called “standard SIRENE”) and in measuring changes in surface potential on the different samples as a function of time during the irradiation and relaxation phases. The samples aged in SEMIRAMIS were transferred to the SIRENE enclosure without a vacuum break.

Within the framework of an experimental simulation of the flux of critical charge electrons encountered on the GEO orbit, the beam used for the tests is composed of the association of the two sources of radiation: Van de Graaff accelerator and electron gun in order to reproduce the spectrum of reference Kp > 5.

The integrated energy spectrum of the SIRENE electron beam supplied by the two sources of radiation is given in Fig.4; the reference spectrum Kp > 5 and the integrated spectrum AE8MAX are also shown in this figure for comparison [15].

Figure 4: Integrated spectrum of the SIRENE electron beam composed of the 2 radiation sources [15]



The integrated flux level (for $E > 20$ keV) being in the order of $300 \text{ pA} / \text{cm}^2$, this value is adopted as the flux level for irradiations representative of a critical orbit charge environment in GEO. This beam, the composition of which is $E = 20 \text{ keV}$, $F = 250 \text{ pA} / \text{cm}^2$ + E from 0 to 400 keV, $F = 50 \text{ pA} / \text{cm}^2$, characterizes the spectrum designated by the term "standard SIRENE spectrum".

3. QUALIFICATION PLAN

In order to qualify the MAP[®] SCK5N, its characteristics must meet the requirements listed in Table 3. These requirements are derived from the characteristics of an antistatic coating and from the ECSS-Q-ST-70-02C outgassing standard [1].

Table 3. Requirements for MAP[®] SCK5N antistatic coating

Properties	Requirements
Solar absorptance	< 0.34
Infrared emissivity	> 0.90
Electrical surface resistance (Ω/\square)	$10^6 - 10^9$
RML ⁴ (%)	≤ 1
CVCM ⁵ (%)	< 0.1

Moreover, the coating must be applied mainly on Kapton[®] HN. Other substrates such as the following are also used and will be evaluated by late June 2020: Polyimide-based plastic, Titanium alloys, Aluminum alloys, Be-Cu alloys, Kevlar, Glass epoxy composite...

The adhesion on such substrates is generally obtained using adhesion primers such as PSX [16] or E' primers

⁴ RML: Recovered mass loss

⁵ CVCM: Collected volatile condensable material

⁶ TML: Total mass loss

[17]. These primers have been developed to promote adhesion on a large variety of substrates such as glass, metallic alloys, and composites. With the MAP[®] SCK5N coating, E' primer has been selected due to its adhesion properties on several substrates.

Previously, PS [18] and PSW [19] primers were used with the SCK5 coating on Kapton[®] and composites [20, 21]. Due to a production stoppage for compliance with environmental regulations, PS and PSW primers have been substituted.

Product characterization was performed at the initial state for all 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 $50 \text{ }^\circ\text{C}$ and 95 % RH) and after a cumulative damp heat test + thermal cycling (10 cycles between $-170 \text{ }^\circ\text{C}$ and $130 \text{ }^\circ\text{C}$ in a vacuum + 90 cycles between $-170 \text{ }^\circ\text{C}$ and $130 \text{ }^\circ\text{C}$ in an N_2 atmosphere).

The main characteristics of the current SCK5 coating [22, 23] are listed in Table 4 and Table 5.

Table 4. Functional properties of SCK5 coating cured at $23 \text{ }^\circ\text{C}$ for 8 days minimum

SCK5	Properties
Solar absorptance	0.27 ± 0.04
Infrared emittance	0.89 ± 0.04
Electrical surface resistance (Ω/\square)	$10^6 - 10^9$
TML ⁶ (%)	0.99
RML (%)	0.89
CVCM (%)	0.12

Table 5. General properties of SCK5 cured at $23 \text{ }^\circ\text{C}$ for 8 days minimum

SCK5	Properties
Typical thickness	$25 - 50 \text{ }\mu\text{m}$
Density	1.60 ± 0.05
Solids content	$57 \pm 2 \text{ } \%$
Viscosity ⁷	40 ± 3
V.O.C.	720 g.L^{-1}
Theoretical consumption	$4.3 \text{ m}^2. \text{Kg}^{-1}$
Theoretical dry film weight	$3.7 \text{ g.m}^{-2}. \mu\text{m}^{-1}$

⁷ AFNOR cup 2.5 at $20 \text{ }^\circ\text{C}$

4. RESULTS

4.1. INITIAL STATE

4.1.1. GENERAL PROPERTIES

The density of MAP[®] SCK5N was measured using a pycnometer in accordance with standard ISO 2811-1 [4]. The value was measured at 1.15 for the Base/Hardener mix. The solids content value was measured according to standard ISO 3251 [3] and is equal to 47 % (Base/Hardener mix).

The values of the viscosity measurements and pot-life are listed in Table 6. The viscosity was measured at 23 °C with a thinning ratio of 20 % using MAP[®] SCK5N thinner.

Table 6. Rheological properties measured at 23 °C for MAP[®] SCK5N

Properties	Value
Viscosity AFNOR cup No. 4 after dilution (Base + Hardener + Thinner)	13 ± 5 s
Pot-life	> 1 h

All the properties and characteristics are listed in Table 7 and compared to those of the SCK5 coating.

Table 7. General properties of SCK5 and MAP[®] SCK5N – measurement at 23 °C

Properties	SCK5	MAP [®] SCK5N
Typical thickness	25 – 50 µm	60 – 100 µm
Density	1.60 ± 0.05	1.15 ± 0.05
Solids content	57 ± 2 %	47 ± 2 %
Viscosity AFNOR cup No. 2.5 at 20 °C	40 ± 3 s	-
Viscosity AFNOR cup No. 4 at 23 °C	-	13 ± 5 s
V.O.C.	720 g.L ⁻¹	683 g.L ⁻¹
Theoretical consumption	4.3 m ² . Kg ⁻¹ (40 µm thick)	2.4 m ² . Kg ⁻¹ (80 µm thick)
Theoretical dry film weight	3.7 g.m ⁻² . µm ⁻¹	2.4 g.m ⁻² . µm ⁻¹

4.1.2. FUNCTIONAL PROPERTIES

Solar absorptance of MAP[®] SCK5N was measured between 0.28 and 0.33, whereas infrared emissivity was around 0.91. The measurements were carried out in accordance with ECSS-Q-ST-70-09C [7] on aluminum and Kapton[®] HN substrates.

The electrical surface resistance was measured between $1 \times 10^6 \Omega/\square$ to $1 \times 10^9 \Omega/\square$. These values are kept in this range for the defined thickness range of 60 µm to 100 µm. MAP[®] SCK5N is then an antistatic coating. Measurements were carried out on insulating substrates such as glass or Kapton[®] HN.

The outgassing properties were measured at the Airbus Toulouse facility on a product after (1) 4 h at 23 °C and 55 % relative hygrometry to allow the solvent to evaporate, and then cured for 15 h at 70 °C and (2) 28 days at 23 °C. The results are listed in Table 6 [24].

Table 8. Outgassing results for MAP[®] SCK5N

MAP [®] SCK5N	TML (%)	RML (%)	CVCM (%)
4h at 23 °C + 15h at 70 °C	0.16	0.11	0.01
28 days at 23 °C	0.18	0.13	0.01

All the functional properties are listed in Table 9 and compared to those of the SCK5 coating.

A significant improvement in outgassing data was obtained with the MAP[®] SCK5N coating compared to the previous SCK5 coating. Thermo-optical properties (α s and ϵ) were in the same range.

Table 9. Functional properties of SCK5 and MAP[®] SCK5N

Properties	SCK5	MAP [®] SCK5N
Solar absorptance	0.27 ± 0.04	0.30 ± 0.04
Infrared emittance	0.89 ± 0.04	0.92 ± 0.03
Electrical surface resistance (Ω/\square)	$10^6 - 10^9$	$10^6 - 10^9$
TML (%)	0.99	0.16
RML (%)	0.89	0.11
CVCM (%)	0.12	0.01

4.2. POST-AGEING TESTS

Ageing tests were carried out at the CNES facility for the MAP[®] SCK5N. Minimum and maximum recorded temperatures during vacuum thermal cycling were as follows: -172 °C and 131 °C whereas for N₂ thermal cycling, the minimum and maximum temperatures were -176 °C and 135 °C.

MAP[®] SCK5N was applied at the initial state (1 week after production) and 6 months after production to evaluate storage behavior in order to define the shelf-life of the product.

MAP[®] SCK5N was applied on samples composed of (1) 2024-T3 alloy plates which size was 80 mm x 80 mm with a thickness of 2 mm and (2) 50 µm thick Kapton[®] HN bonded on metallic plates.

On aluminum alloy, electrical surface resistance is under $1 \times 10^6 \Omega/\square$ due to the electrical conductivity of the substrate. A slight increase is observed after thermal cycling: $0.03 \times 10^6 \Omega/\square$ to $5.80 \times 10^6 \Omega/\square$. This increase can be attributed to a post-curing during thermal cycling due to energy input.

Adhesion is class 0 in accordance with standard ISO 2409 [9]. Some very slight traces are observed on the tape at the initial state with 3M[™] 92 tape. These traces did not evolve after the damp heat test and thermal cycling test. Adhesion tests were also performed using Scapa 8705b tape [25], which is an acrylic-based tape with an adhesion strength on steel of 3.5 N/10 mm. On the other hand, 3M[™] 92 tape is a silicone-based tape which adhesion strength on steel is 2.8 N/10 mm [26]. Class 0 adhesion was also observed, and here again, almost no traces were observed at the initial state after ageing.

Solar absorptance and infrared emissivity did not evolve during these ageing tests.

Table 10. Functional properties of MAP[®] SCK5N at the initial state and after ageing for “fresh” MAP[®] SCK5N – R.15.17.219 sample applied on 2024-T3 alloy

	t ₀	Damp heat	Damp heat + Thermal cycling
Rs (x 10 ⁶ Ω/□)	0.01	0.03	5.80
Adhesion 3M [™] 92 tape	Class	Class 0	Class 0s
Infrared emissivity	0.92	-	0.92
Solar absorptance	0.31	-	0.32

The same results were obtained when using MAP[®] SCK5N that had been stored for 6 months (Tables 11 and 12).

Table 11. Functional properties of MAP[®] SCK5N at the initial state and after ageing i storage for “6 months” MAP[®] SCK5N – R.15.17.221B sample applied on 2024-T3 alloy

	t ₀	Damp heat	Damp heat + Thermal cycling
Rs (x 10 ⁶ Ω/□)	0.01	0.02	0.30
Adhesion 3M [™] 92 tape	Class 0	Class 0	Class 0
Infrared emissivity	0.92	-	0.91
Solar absorptance	0.30	-	0.31

On Kapton[®] HN samples, electrical surface resistance at the initial state is $27 \times 10^6 \Omega/\square$. A slight increase is observed after the damp heat test and after the thermal cycling test: $54 \times 10^6 \Omega/\square$ and $120 \times 10^6 \Omega/\square$, respectively. This increase can be attributed to post-curing during thermal cycling due to energy input.

The cross-cut test cannot be performed on soft substrate such as Kapton[®] HN. Consequently, the adhesion cannot be defined. The adhesion of the coating is compliant and very slight traces are observed using 3M[™] 92 tape. These traces do not evolve after the damp heat test and the thermal cycling test. Adhesion tests were also performed using Scapa 8705b tape. Again, no traces were observed at the initial state after ageing.

Solar absorptance and infrared emissivity do not evolve during these ageing tests.

Table 12. Functional properties of MAP® SCK5N at the initial state and after ageing for “fresh” MAP® SCK5N – R.15.17.219 sample applied on 50 µm thick Kapton® HN

	t ₀	Damp heat	Damp heat + Thermal cycling
Rs (x 10 ⁶ Ω/□)	27	54	120
Adhesion 3M™ 92 tape	Very slight traces	Slight traces	Slight traces
Infrared emissivity	0.92	-	0.92
Solar absorptance	0.31	-	0.32

The same results were obtained when using MAP® SCK5N that had been stored for 6 months (Tables 13 and 14).

Table 13. Functional properties of MAP® SCK5N at the initial state and after ageing in storage for “6 months” MAP® SCK5N – R.15.17.221B sample applied on 50 µm thick Kapton® HN

	t ₀	Damp heat	Damp heat + Thermal cycling
Rs (x 10 ⁶ Ω/□)	23	53	42
Adhesion 3M™ 92 tape	-	Slight traces	Slight traces
Infrared emissivity	0.92	-	0.91
Solar absorptance	0.30	-	0.31

4.3. POST ENVIRONMENTAL TESTS

Two GEO irradiation simulation campaigns were carried out on MAP® SCK5N coatings. The first one took place in 2017 for 1-year irradiation exposure [27, 28]. The second one began in January 2020 for 3 years of exposure⁸.

⁸ Final results will be available by April 2020

The results are presented in Fig. 5. One can see an increase of 0.13 in solar absorptance for the 2017 campaign, whereas an increase of 0.12 was observed in 2020 for the 1-year GEO exposure.

The evolution of the solar absorptance for each campaign at each irradiation step is presented in Table 14.

Table 14. MAP® SCK5N solar absorptance evolution versus irradiation step

Campaign	t ₀	UV	e- 400 keV	p+ 240 keV	p+ 45ke V
2017	0.29	0.34	0.36	0.37	0.42
2020	0.31	0.34	0.35	0.36	0.43

The solar absorptance evolution is presented in Fig. 6. Rather significant yellowing of the samples is observed corresponding to a final solar absorptance of 0.42.

Figure 5 – Solar absorptance evolution of MAP® SCK5N during GEO simulated exposure

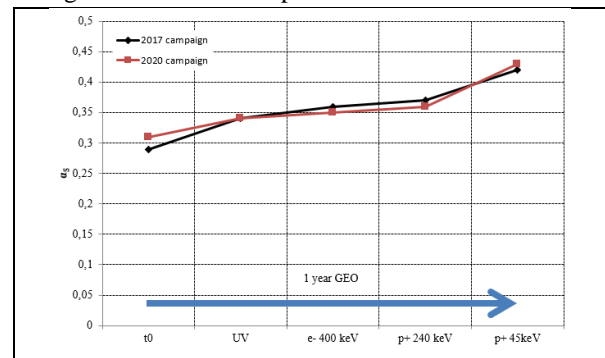
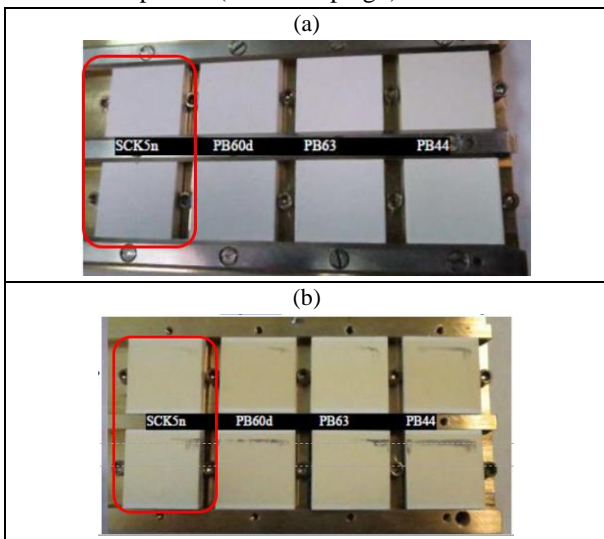


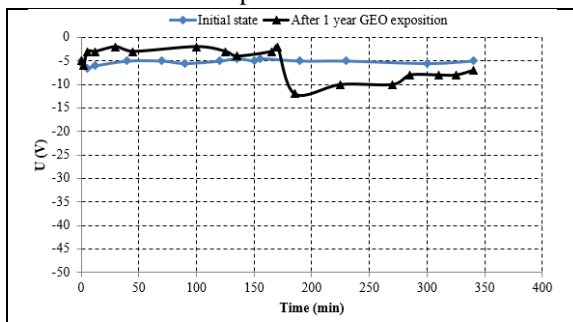
Figure 6 - MAP[®] SCK5N samples applied on 2024-T3 samples (a) at the initial state and (b) after 1-year GEO simulated exposure (2017 campaign)



When exposing MAP[®] SCK5N samples to SIRENE irradiation for \times h, one can observe very low surface potential around -5V for un-irradiated samples. For the 1-year GEO irradiated samples, the surface potential is slightly higher (-15V) but significantly under the threshold specified by standard ECSS-E-ST-20-06C: -500 V [29].

No significant impact of the GEO environment is foreseen on the MAP[®] SCK5N samples.

Figure 7 – Surface electrical potential of MAP[®] SCK5N samples applied on 2024-T3 samples during “SIRENE” irradiation for the coating at the initial state and after 1-year GEO simulated exposure



These results are compliant within the antistatic behavior of the coating and confirm the ability of MAP[®] SCK5N to be used on external parts of spacecraft without any risk of electrostatic charging.

5. CONCLUSION

MAP[®] SCK5N coating has been developed as a substitute for the SCK5 coating. This new product is an ultra-low outgassing silicone white antistatic coating. The product is composed of a specific silicone polymer purified without solvent and a specific white antistatic pigment. The functional properties are the solar absorptance of 0.29, infrared emissivity of 0.92 and a surface electrical resistance in the 10^6 to $10^9 \Omega/\square$ range. Outgassing properties are as low as 0.11 % for the RML and 0.01 % for CVCM. These properties are obtained using a fast-curing process (4h minimum at room temperature + 15h at 70 °C) or for a minimum of 8 days at room temperature. A thickness of 60 to 100 μm is necessary to obtain solar absorptance. Adhesion on aluminum alloys and Kapton[®] HN substrates is compliant provided that E' primer is used. Until now, a shelf-life of 6 months has been validated.

The ageing tests consist of three steps: damp heat test + vacuum thermal cycling and ambient pressure thermal cycling were applied to the coatings. Environmental tests: GEO and ESD were also carried out. Very low electrical surface potential was obtained qualifying the use of this product on external parts of the spacecraft. A limited increase in solar absorptance (0.12 to 0.13) was observed after 1-year GEO exposure.

The next steps will be the qualification of the coatings on most of the substrates used for space applications (Polyimide-based plastic, Titanium alloys, Aluminum alloys, Be-Cu alloys, Kevlar, Glass epoxy composite, etc.), 3-year GEO assessment, validation of 12 months shelf-life of the product and ESD tests at negative temperatures.

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