DEVELOPMENT OF A HIGH TEMPERATURE BLACK COATING FOR SPACE APPLICATION

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**ABSTRACT**

In the last years, several missions close to the sun have highlighted the low number of thermal control coatings that can withstand temperatures of around 400 °C and a large flow of charged particles. Based upon this observation, CNES and MAP have therefore decided to develop a coating that can withstand this extreme environment while remaining compatible with "paint" technology.

These coatings have to withstand high temperatures, the adhesion has to be compliant on the following specific metallic substrates used for high temperatures needs: TA6V, INOX 304L or INCONEL 600 for instance. Moreover to these specificities, the following ones common for all the coatings dedicated to space application have to be validated: compliance versus ECSS-Q-ST-70-02C, resistance under thermal vacuum cycling tests, adhesion versus ISO 2409 standard, high solar absorptivity ($\alpha_S$) and infrared emissivity ($\varepsilon$). In order to avoid any electrostatic risk, it has been decided to develop an antistatic coating which is defined by an electrical surface resistance $R_s < 1 \text{ M}\Omega/\square$ (ESD compliance versus ECSS-E-ST-20-06).

Moreover, GEO environmental exposure simulation was performed at ONERA (UV + electrons + protons). A specific attention was dedicated to the choice of raw materials and products which are not identified as risk versus REACH regulation. On the other hand, ITAR free products were selected. Finally, sustainable production process that limits the risk of vagaries has been selected to also improve product-process combination to save time on the critical path stages.

**2 TECHNICAL SPECIFICATIONS**

High temperature resistant coatings have to withstand temperatures up to 400°C, the adhesion has to be compliant on the following specific metallic substrates used for high temperatures needs: TA6V, Stainless Steel 304L or INCONEL 600 for instance. Moreover to these specificities, the following ones common for all the coatings dedicated to space application have to be validated: compliance versus ECSS-Q-ST-70-02C [2], resistance under thermal vacuum cycling tests, adhesion versus ISO 2409 standard, high solar absorptivity ($\alpha_S$) and infrared emissivity ($\varepsilon$). In order to avoid any electrostatic risk, it has been decided to develop an antistatic coating which is defined by an electrical surface resistance $R_s < 1 \text{ M}\Omega/\square$ (ESD compliance versus ECSS-E-ST-20-06 [3]).

Moreover, GEO environmental exposure simulation was performed at ONERA (UV + electrons + protons). A specific attention was dedicated to the choice of raw materials and products which are not identified as risk versus REACH regulation. On the other hand, ITAR free products were selected. Finally, sustainable production process that limits the risk of vagaries has been selected to also improve product-process combination to save time on the critical path stages.

**3 RESULTS**

**3.1 General properties**

We selected a silicone polymer for its resistance at high temperature. A specific T resin has been developed in order to fulfil the criteria of high temperature, low thickness and adhesion on metallic substrates. The coating developed is a 2K polycondensation silicone formulation.

Due to its specific adhesion properties no primer is needed on metallic alloys such as TA6V, 304L, Inconel 600, 2017-T4 aluminium.

The coating has to be cured at room temperature first during 48 hours and second 1 hour at 300°C. Main properties of the coating are listed in Table 1.

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![Fig. 1. Artist view of Solar Orbiter satellite [1]](image-url)
3.2 Outgassing results

Outgassing tests were carried out at Intespace further to the ECSS-Q-ST-70-02C standard. The coating was cured first at room temperature during 48 hours and second 1 hour at 300°C. The results are presented in Table 2. The outgassing results of the coating are very low, the RML = 0.1% and no CVCM was collected.

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
<th>Standard</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base / Hardener mixing ratio</td>
<td>99.5/0.5</td>
<td>-</td>
</tr>
<tr>
<td>Solid contents mix (%)</td>
<td>38</td>
<td>ISO 3251</td>
</tr>
<tr>
<td>VOC rate (g/L)</td>
<td>466</td>
<td>ASTM D3960</td>
</tr>
<tr>
<td>Thickness (µm)</td>
<td>20 - 40</td>
<td>NF EN ISO 2360</td>
</tr>
<tr>
<td>Dry film weight</td>
<td>1.52 g dry /m² /µm</td>
<td>Theoretical calculation</td>
</tr>
<tr>
<td>Solar absorptance (αs)</td>
<td>0.96</td>
<td>ECSS-Q-ST-70-09C</td>
</tr>
<tr>
<td>Infrared emittance (εi)</td>
<td>0.89</td>
<td>ECSS-Q-ST-70-09C</td>
</tr>
<tr>
<td>Total Integrated Scatter</td>
<td>4.5%</td>
<td>CNES method</td>
</tr>
<tr>
<td>Electrical surface resistance</td>
<td>&lt; 10⁶</td>
<td>MAP 006/AQ/92/NI</td>
</tr>
</tbody>
</table>

Tab. 1. Technical data of MAP® HT1607 coating [4]

3.3 Ageing tests: damp heat test + thermal cycling test

In order to evaluate the ability of MAP® HT1607 to withstand environmental stresses, damp heat test (50°C – 95% HR – 8 days) and thermal vacuum cycles test (-170°C – 400°C) were carried out. Damp heat tests were performed on ESPEC 2KTH chamber whereas thermal vacuum cycles were performed at AAC Aerospace &Advanced Composites.

The conditions of the cycles are listed hereunder:
- Pressure: 10⁻³ hPa;
- Temperature: -170°C/+400°C ± 5°C;
- Number of cycles : 100;
- Cycle duration: 182.5 minutes (cold step = hot step = 20 min, heating and cooling slope 8°C/min)

Results of adhesion further to ISO 2409 standard [6], thermo-optical properties and electrical surface resistance at initial state, after damp heat tests and after damp heat tests + thermal vacuum cycling are presented in Tables 3, 4, 5 and 6.

<table>
<thead>
<tr>
<th>Substrate</th>
<th>Adhesion</th>
<th>Solar absorptivity (αp ± 0.03)</th>
<th>Infrared emissivity (εi ± 0.03)</th>
<th>Electrical surface resistance (kΩ/□)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>t₀</td>
<td>After DH</td>
<td>After DH + TVC</td>
<td>t₀</td>
</tr>
<tr>
<td>304L</td>
<td>0/5</td>
<td>0/5</td>
<td>0/5</td>
<td>0.96</td>
</tr>
<tr>
<td>Inconel 600</td>
<td>0/5</td>
<td>0/5</td>
<td>0/5</td>
<td>0.96</td>
</tr>
<tr>
<td>TA6V</td>
<td>0/5</td>
<td>0/5</td>
<td>0/5</td>
<td>0.96</td>
</tr>
</tbody>
</table>

Tab. 3. Adhesion tests results at initial state, after damp heat test and thermal vacuum cycling

Tab. 4. Solar absorptivity values at initial state, after damp heat test and thermal vacuum cycling

Tab. 5. Infrared emissivity values at initial state, after damp heat test and thermal vacuum cycling

Tab. 6. Electrical surface resistance values at initial state, after damp heat test and thermal vacuum cycling

No change of thermo-optical properties, adhesion or electrical surface resistance has been observed after damp heat test exposure and thermal vacuum cycling.

3.4 Environmental simulation at ONERA lab

3.4.1 Experimental conditions of the test

Coatings used on geostationary satellites receive a UV solar flux of about 118 W/m². The dose of UV radiation received by the satellite is expressed in equivalent solar hours (ESH). This unit corresponds to an exposure of one hour under a flow of 118 W/m² in the UV, that is, 1 ESH = 118 X 3600 = 4.25 x 10⁴ J/m².

Over a period of 1 year in geostationary orbit the coatings on the North / South faces of the satellite receive a dose of 1112 ESH. For this test, a total of 3336 ESH was applied following 3 steps of 1112 ESH. Performing irradiation for 3336 hours with a 118 W/m² flux being too long, an average acceleration coefficient of 7.64 (7.64 X 118 W/m²) is applied. The irradiation is carried out day and night until reaching 1112 ESH, which is equivalent to duration of about 6 days.

For particles, the dose profile of the space is simulated...
with electron energy (in sample thickness) and two proton energies (at the surface of the sample). Note: the uniformity of the irradiated area is ± 9% for UV, ± 10% for electrons, ± 11% to ± 18% for protons. The simulation of a 3 years exposure in geostationary orbit is divided into three identical phases that each represent 1 year in this orbit. During a test phase the irradiations are carried out in the following order:

1) UV radiation of 1112 ESH;
2) 400 keV energy electrons (beam current 10nA, density 1×10^{15} electrons/cm^2)
3) 240 keV energy protons (beam current: 20nA, density: 2×10^{14} protons/cm^2)
4) 45 keV energy protons (beam current: 20nA, density: 2×10^{15} protons/cm^2)

The test has been carried out at DESP department of ONERA at Toulouse using SEMIRAMIS facility, Fig.2.

For the total GEO simulation, total dose measured during 1 year flight experimented is plotted on the graph of Fig.3 and compared to each component of the total irradiation: protons of 45 keV, protons of 240 keV and electrons of 400 keV. Finally these hypotheses are used to calculate the theoretical depth penetration of each component inside the coating assuming a composition of pure silicone.

Fig. 2. Picture of SEMIRAMIS facility at ONERA

Fig. 3. Particles simulation depth penetration for a silicone (d = 1.2) – calculation [8]

3.4.2 Results

Thermo-optical properties are measured under vacuum at the initial state and after each year of GEO simulation exposure.

Measurements have been carried out after each year of simulation. The value is equal to 0.96 at initial state under vacuum and then decreases to 0.95 after 1 year GEO. The value does not evolve when the duration of the exposure increases up to 3 years.

For the infrared emissivity, initial value measured at ambient pressure is 0.87 and does not evolve after 3 years GEO exposure.

No change has been observed on the coating after an exposition equivalent to 3 years in geostationary orbit (Figs.4 and 5).

MAP® HT1607 coating fulfilled the requirements of the GEO exposure simulation test. This coating is compliant with the use in space environment.

Fig. 4. Evolution of the solar absorptance versus duration of exposure to the GEO simulated environment

MAP® HT1607 coating fulfilled the requirements of the GEO exposure simulation test. This coating is compliant with the use in space environment.

Fig. 5. Pictures of MAP® HT1607 sample (a) before and (b) after GEO irradiation
3.5 ESD tests

3.5.1 Experimental conditions of the test

The purpose of this test is to validate the ability of the coating to dissipate electrostatic charges that reach its surface. The test was carried out in the SIRENE irradiation facility installed in the ONERA DESP in Toulouse. It breaks down into two tests: (1) An electron beam of 20 keV - 250 pA/cm² is sent on the samples during 3h. Surface potential is measured simultaneously with irradiation. This test is carried out at three different temperatures, using a sample holder thermostated at 25 °C, -80 °C and -150 °C. Between each configuration the samples remain 2h without irradiation (relaxation time) and are warmed up to ambient temperature (sample discharge). (2) A multienergetic flux called standard spectrum SIRENE (25 keV - 250pA/cm² + 0-400 keV - 50 pA/cm²), representative of the flow of the GEO orbit, is applied to the surface of the samples which are cooled to a temperature of -150 °C (the most critical case). The surface potential is measured simultaneously with irradiation for 3 hours.

The samples are 30mm x 50mm aluminum drilled (2.5mm diameter) at 2.5mm from the sides in the length direction of the plate. The paint is applied in a reduced area of 30mm x 40mm.

3.5.2 Results

Surface electrical potential is measured during the irradiation test. If it is less than 1000V, according to ECSS-E-ST-20-06 [3], then the paint does not present any risk of electrostatic discharge. Results are listed in Table 7.

<table>
<thead>
<tr>
<th>T (°C)</th>
<th>Surface potential (V)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Monoenergetic flux</td>
</tr>
<tr>
<td>25</td>
<td>Vₙ ≤ 2</td>
</tr>
<tr>
<td>-80</td>
<td>Vₙ ≤ 3</td>
</tr>
<tr>
<td>-150</td>
<td>Vₙ ≤ 2</td>
</tr>
</tbody>
</table>

Table 7. Surface potential of MAP® HT1607 coating under ESD tests

For the temperatures tested and the different flux, the surface electrical potential is much lower than 1000V which means that the coating does not present any risk of electrostatic discharge. Results are compliant with ECSS-E-ST-20-06. This test therefore validates that the use of the MAP® HT1607 coating will not induce electrical breakdowns on the satellite.

4 CONCLUSION

Finally, a new high temperature coating called MAP® HT 1607 has been successfully developed. This product is a 2K silicone coating which properties are the following:

- αₑ = 0.96 ± 0.02;
- ξₑ = 0.89 ± 0.03;
- Rₑ = 0.1 kΩ; 2o;
- T.I.S. = 4.51%;
- TML = 0.13 %;
- RML = 0.05 %;
- CVCM = 0.00 %.

At the initial state, the adhesion of the coating was defined as 0 or 1 class depending upon the substrates. All the thermal cycling tests performed under vacuum in the temperature range of -170°C to 400°C were compliant and did not show any change in the thermooptical properties nor electrical surface resistance nor adhesion. Cumulative damp heat test + thermal cycling tests were also performed and were compliant to the requirements. GEO simulated environmental tests were carried out at ONERA laboratory. After an exposition of three years simulated GEO the solar absorptivity of the coating was increased of 0.01 whereas infrared emissivity was not changed. ESD tests were also carried out at ONERA. For the temperatures used during this test and for the used fluxes, the electrical surface potential is under 1000 V which gives a complete compliance versus ESD risk.

REFERENCES

2. ECSS-Q-ST-70-02C, Thermal vacuum outgassing test for the screening of space materials, 2008
3. ECSS-E-ST-20-06, Spacecraft charging, 2008
4. Technical data sheet of MAP® HT1607 coating
5. Outgassing data of MAP® HT1607 coating – Intespace report N°M4558
6. ISO 2409 standard, Paints and varnishes, cross-cut tests, 2007