

# **IRUCOAT4SPACE**

## Environmentally friendly corrosion protective hybrid coating as substitute of chromium-based conversion coatings for space applications

(ESA AO71-8876/17/NL/CRS)

### **Executive summary**

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### **1** Executive Summary

The main objective of IRUCOAT4SPACE project is the optimization and scale up of a protective coating (IRUCOAT) for aluminium alloys components of space devices, as a substitute of Cr-based products. IRUCOAT is an eco-friendly hybrid sol-gel coating based on SiO<sub>2</sub>-ZrO<sub>2</sub>-organic epoxy interconnected network, developed by TECNALIA and already tested for corrosion protection of AA6xxx AI alloys and other performance requirements applicable to the aerospace sector.

The main goal of the project is having a coating with an outstanding performance, meeting the appropriate space requirements and ready for application to specific space components, as defined with end-user space companies.

The coating formulation and application technique (spraying and dipping) and procedure (application parameters and curing conditions) have been optimized considering outgassing, a property related to the release of gaseous species from the coated aluminium panels under high vacuum conditions. This is a quite crucial property for this sector as outgassed particles might be a serious source of contamination, especially for optical components.

Different coatings formulations and curing conditions were tested for both dipping and spraying applications, until the outgassing test proved successful. A specific coating formulation (variations from the IRUCOAT baseline) was developed for each application technique, each one with its specific curing time and temperature.

- Dipping: IRUCOAT-Mc, cured at 180°C for 3 h
- Spraying: IRUCOAT-M applied by ultrasonic gun, cured at 150°C, 4h

These coatings were then tested for corrosion properties through Electrochemical Impedance Spectroscopy (EIS) and Salt Spray Test (SST), besides morphological characterization. Figure 1 shows the EIS spectra at 2 and 24 hours of immersion in 0.05 M NaCl (left and right, respectively) of the best coatings optimized to pass outgassing test (as defined above), compared to their baseline (deposited by dipping and spraying and cured in both cases at 120 °C for 8 hours). For both deposition techniques, the coatings cured at higher temperature show a better corrosion resistance than their respective baselines, when immersed in a 0.05 M NaCl solution. At 2 hours of immersion, however, both coatings deposited by dipping have better barrier properties than the coatings applied by spraying, while after 24 hours of immersion, only the dipped coating cured at 180 °C maintains high barrier properties, while in the other cases some water uptake already happened. In all cases, the impedance at low frequency is comparable and above  $10^{+6} \Omega$  cm<sup>2</sup>.

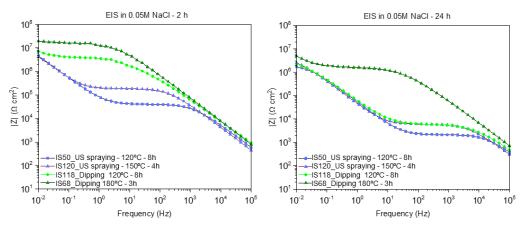


Figure 1. EIS spectra of IRUCOAT, applied on AA 2024 by dipping and spraying and cured at different times and temperatures (as indicated in the legend), after 2 hours (left) and 24 hours (right) of immersion in 0.05 M NaCl.



Overall, these results confirm the feasibility of the ultrasonic sprayed coating cured at 150 °C for 4 hours, which has proven to be successful for outgassing results. Although the coating applied by dipping and cured at 180 °C for 3 hours show better barrier properties than the others, it should be considered that this temperature is above the limit than the one suggested for treated this specific Al alloy (AA2024), so the specific substrate and its function as a component should be considered before proceeding to coating application. In addition, often the specific shape of a component is such that application by dipping is not possible and spraying is necessary.

Following optimization of coating formulation and application procedure, samples were produced on Al alloys AA2024-T3 and AA6061-T6 for validation. The coating IRUCOAT-Mc was applied by dipping and cured at 180°C for 3 hours. The coating was deposited on flat panels of size 250x75 mm. The coating thickness is  $3.5 \,\mu$ m on average.

Table 1 shows a summary of the results of the validation campaign. The main drawback is that the coating does not meet the conductivity requirements. However, it gave remarkable results in terms of thermo-optical properties, solvent resistance and humidity resistance on both AA2024 and AA6061 alloys, besides passing the outgassing requirements (following the indications of the ECSS-Q-ST-70-02C specification), with RML < 1.0 % and CVCM < 0.1 %.

IRUCOAT provided good corrosion protection to the AA6061 alloy after the samples were exposed to 168-hour salt spray test (as received or after being submitted to thermal cycles). On the other side, the alloy AA2024 still showed some signs of corrosion after the salt spray test, although the samples were not badly damaged: very small pits covered the entire area, but without overall damage. In addition, the samples passed the thermal and humidity cycles without any sign of damage and the application of thermal cycles before exposure to SST also improved the results (probably due to further curing of the coating). This makes IRUCOAT stand out compared to traditional conversion coatings such as Surtec 650 and Alodine 1200S, which have poor resistance to thermal cycles, including in corrosion resistant alloy such as AA6xxx<sup>1</sup>.

TEST/ MEASUREMENT	SUCCESS CRITERIA	AA2024-T3 w/ IRUCOAT	AA6061-T6 w/ IRUCOAT
<b>Corrosion resistance</b> (MIL DTL 81706 & MIL DTL 5541F) Salt spray test - 168 h	Evaluation according to MIL-DTL-5541 Section 3.6	x	٧
<b>Thermal and corrosion resistance</b> Salt spray test after thermal cycling – 100 cycles (-70 to 100°C), dwell time= 15min; heating rate= 6°C/min; cooling rate= 3.5°C/min	No corrosion after 100 thermal cycles + 168 h SST	√-	v
Humidity resistance Damp heat test (T= 40°C, RH: 100%) - 336 h	No coating degradation after 168 hours	V	v
<b>Conductivity</b> (MIL-DLT-81706B) Electrical contact resistance (before and after SST)	< 5,000 $\mu\Omega/in^2$ as received < 10,000 $\mu\Omega/in^2$ after 168-h SST	X	x
<b>Thermo-optical properties</b> (ECSS-Q-ST-70-09C) Infrared emittance ( $\epsilon$ ) and solar absorptance ( $\alpha$ ) before and after thermal cycling	$\epsilon$ and $\alpha$ not affected by thermal cycling	v	v
Solvent resistance (MEK) • 10 min immersion in ultrasonic bath with solvent at 70°C • repeated rubbing (50 passes) by a cloth soaked in solvent.	No coating degradation	v	v

Table 1. Summary of results of the validation campaign

<sup>&</sup>lt;sup>1</sup> Agustín-Sáenz, C.; Santa Coloma, P.; Fernández-Carretero, F.J.; Brusciotti, F.; Brizuela, M. Design of Corrosion Protective and Antistatic Hybrid Sol-Gel Coatings on 6XXX AlMgSi Alloys for Aerospace Application. Coatings 2020, 10, 441.



Here, a trade-off between different properties has been made and the coating conditions to meet the outgassing requirement have been chosen for validation. The coating formulation is such that the thickness and the composition have a negative effect on the conductivity requirements. Depending on the specific application and/or component where the coating needs to be applied, adjustment to the formulation and application procedure could be made to move more towards conductivity requirements or best outgassing results, keeping corrosion and thermal resistance within the limits.

As a final step in the project, the coating was applied on the actuator support provided by SENER (Al alloy AA7075) by means of a manual ultrasound spraying gun and cured at 150 °C for 4 hours, the spraying conditions to pass the outgassing test. The coated component was tested for damp heat test (40 °C and 95% RH), followed by thermal resistance (cycles between -70 °C and 100 °C) and it looked intact after both tests, without any visible signs of corrosion or coating damage (Figure 2). Table 2 summarizes all IRUCOAT properties, as validated in this project.

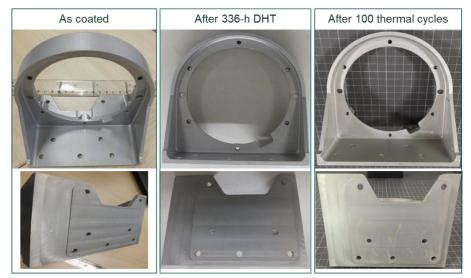


Figure 2. AA7075 actuator support (SENER) coated by spraying with IRUCOAT and cured at 150 °C for 4 hours: as received (left), after 336 hours of damp heat test (centre), and after 100 thermal cycles (right)

Table 2. Summary of IRUCOAT properties

Properties	Comply / Not comply	
Chromium free	V	
Deposited by dipping and spraying	V	
Salt spray test (ASTM B117): 168h	✓ on AA6061 × Pits on AA2024	
Damp heat test (40ºC, 100%RH): 168h	V	
Thermal cycling (-70ºC to 100ºC): 100 cycles + salt spray test: 168h	✓ on AA6061 × Pits on AA2024	
Solvent resistance (isopropanol, methyl ethyl ketone)	v	
Optical properties (solar absorptance, infrared emittance): not affected by thermal cycling	v	
Contact electrical resistance before and after 168h SST (on AA6061-T6, according to MIL-DTL-81706B)	x	
Humidity resistance (DHT) and thermal resistance on prototype (AA7075)	٧	