# Ongoing activities on materials substitution in the European Space Agency

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# Agenda of the Presentation

## *CrVI alternatives development*

- Status of the R&D funded program dealing with CrVI alternatives development and testing
- REACH regulation does limit/restrict use of hexavalent chromium-> The Sunset date for chromium trioxide was September 2017
- An application for authorization has been submitted, space applications are covered by CTACSub, EC decision is pending

## *Lead free electronics development*

- ECHA RAC has been requested by EC DG EMPL in January 2019 to update the existing health-based bOEL (air) and biological limit value (blood) for lead and its compounds under the Chemical Agents Directive (CAD). RAC is expected to issue an opinion by the end of 2019 (latest by mid-2020).
- Status of the activities performed in the frame of Joint Working Group on Pb-Free transition

REACH

# Programs currently running (CrVI alternatives development)

| | |
|---|---|
| *ESA-NASA joint activity concerning* | *"Evaluation of alternative surface treatments and coating systems as future replacements for chromate-based conversion coatings".* |
| *ISQ Frame Contract* | *"Evaluate the suitability and availability of alternative surface treatments to hexavalent chromium"* |
| *ECD Project I and II and III* | *"3 R&D project for the development and space qualification of Surtec CC on various aluminum alloys and large parts"* |
| *IRUCOAT4SPACE* | *"Environmentally friendly corrosion protective hybrid coating as substitute of chromium based conversion coatings for space applications"* |

# ISQ Frame Contract

## *Main achievements and current status*

# Selected Alloys and Pretreatments

| Aluminium Alloy | Temper | Specifications | Major alloying elements |
|---|---|---|---|
| AA2024 | T3 | A | |
| AA2024 | T81 | | |
| AA6061 | T6 | A | |
| AA7075 | T73 | A | |
| AA5083 | H111 | AMS | |

| Conversion Coating | Chemistry | Application Methods | Supplier | Visual Appearance |
|---|---|---|---|---|
| Alodine 1200 (reference) | Chromic acid, complex fluorides | Immersion, spray, wipe | Henkel, Germany | Light golden |
| PreCoat 32 | Trivalent Chromium | Immersion, spray | AD International B.V, Netherlands | Clear blue/green |
| Socosurf TCS/PACS | Trivalent Chromium | Immersion, spray | Socomore, France | Green/Colorless |
| Surtec 650V | Trivalent Chromium | Immersion, spray | Surtec, France | Iridescent, faintly blue to tan and visible layer |
| Bonderite M-NT 65000 | Trivalent Chromium | Immersion, spray | Henkel, Germany | Green |

# Test Campaign - Organization

**Phase 1 Pre-Screening**

- Pre-screening of selected pre-tretments on five different substrates with special attention to AA2024
- Salt Spray Testing (ASTM B117) for 168 hour was the baseline test

**Phase 2 Extended Testing**

- Metallurgical coating evaluation
- Salt spray testing (ASTM B 117)
- Resistivity check (MIL-DTL-81706)
- Humidity testing (ASTM D 2247):
- Thermal Cycling Test (ECSS-Q-ST-70-04C)

| | Step 1 | Step 2 | Step 3 | Step 4 |
|---|---|---|---|---|
| | 10 cycles in vacuum | 80 cycles in ambient | 10 cycles in vacuum | |
| $T_{max}$ | 100 (+5/-10) ºC | 100 (+5/-5) ºC | 100 (+5/-10) ºC | Isothermal step 150ºC in high vacuum for 145 hrs |
| $T_{min}$ | -100 (+5/-5) ºC | -100 (+5/-5) ºC | -100 (+5/-5) ºC | |
| $T_{CR}$ | 1 ºC/min | 5 ºC/min | 1 ºC/min | |
| $T_{dwell}$ | >5 min | >5 min | >5 min | |

**Phase 3 Long Term Env. Test Programme**

- Based on the results of Phase 2, three surface treatments (**Precoat A32, Socosurf TCS/PACS and Surtec 650V**) and three substrates (**AA2024-T81, AA6061-T6 and AA7075-T73**)have been be exposed, alone and with primer/topcoat in ESA facilty in Kourou, French Guiana

# Test Campaign - Main Achievements

**Phase 1 Pre-Screening**

- Precoat A32 and Socosurf TCS/PACS are the most promising chemical conversion alternatives for all the aluminum alloys, including AA2024.
- Surtec 650V and Bonderite M-NT 65000 could be also candidates for replacement if submitted to optimization for the AA2024
- All the 4 treatment selected for the extended test campaign

**Phase 2 Extended Testing**

- Salt Spray Testing: two pre-tretments, PreCoat A32 and Socosurf TCS/PACS, passed the minimum requirement for all alloys excepted AA2024 T81 (for Precoat A32). Surtec 650V and Bonderite M-NT 65000 passed the minimum requirements for AA7075-T73, AA6061-T6 and AA5083-H111 and failed for both AA2024 T3 and T81.
- Humidity Testing: all the pretreatments performed well for exposure time around 3000 h
- Thermal cycles : PreCoat A32 had good results for all the alloys, after the 4 steps and Socosurf TCS/ PACS, Surtec 650V and Bonderite M-NT 65000 showed corrosion and discoloration after step 3 and 4 for all the alloys

**Phase 3 Long Term Env. Test Programme**

- The results of the phase 3 test campaign have been concluded and the results presented in the following

# Phase 3_Just finished

| Aluminium Alloys | Chemical Conversion Coating | Pre-treatment Supplier | Primer + Topcoat Application |
|---|---|---|---|
| AA2024-T81 (2) | Alodine 1200 S (A) (reference) | ISQ - Henkel | MAP Coatings |
| AA6061-T6 (3) | Precoat A32 (B) | AD International | |
| AA7075-T73 (4) | Socosurf TCS/ PACS (E) | Socomore | |
| | F - Surtec 650 V (F) | Surtec | |

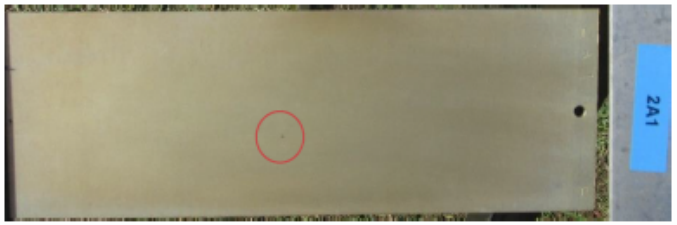
- 60 sample exposed in ESA facility in Kourou (French Guyana) for 7 months
- Panels have been inspected daily during the first 7 days, weekly until 4 months of exposure and then monthly until the end of exposure. The inspection includes visual analysis and photographic report.
- Parameters such as solar global radiation, ambient temperature (daily maximum and minimum), relative humidity (daily maximum and minimum) and total rainfall have been measured

# Phase 3 – Results (conversion coating)

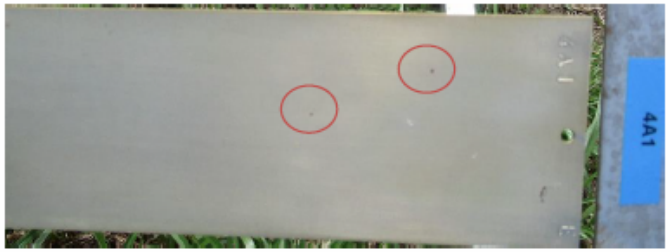
| CCC | Aluminium Alloys | Inspection/ Exposure Time | | | | | |
|---|---|---|---|---|---|---|---|
| | | After 1st Day | After 7th Day | After 1st Month | After 2nd Month | After 5th Month | After 7th Month |
| **Alodine 1200 S** | AA2024-T81 | No evidences of surface defects (blistering, cracking, flaking, delamination and corrosion around a scribe) | No evidences of surface defects (blistering, cracking, flaking, delamination and corrosion around a scribe) | No/ little degradation of the protective coating (some blistering and corrosion spots); few corrosion products build up in scribe, but no blisters around | More evidence of blistering and corrosion spots on the surface of panels; corrosion products build up in scribes; no blisters and delamination/ corrosion around a scribe | More evidence of degradation of the protective coating (blistering and corrosion spots); corrosion products build up in scribes; some blisters around a scribe; no delamination/ corrosion around a scribe | Blistering and corrosion spots, namely on the AA7075-T73; few corrosion products and blisters around a scribe |
| | AA6061-T6 | | | | | | |
| | AA7075-T73 | | | | | | |
| **Precoat A32** | AA2024-T81 | | | Few degradation of the protective coating (blistering and corrosion spots); few corrosion products build up in scribe, but no blisters around | | | |
| | AA6061-T6 | | | | | | |
| | AA7075-T73 | | | | | | |
| **Socosurf TCS/PACS** | AA2024-T81 | | | Few degradation of the protective coating (blistering and corrosion spots on the surface of panels); few corrosion products build up in scribe, but no blisters around | More evidence of degradation of the protective coating (blistering and corrosion spots); corrosion products build up in scribes; no blisters and delamination/ corrosion around a scribe | | |
| | AA6061-T6 | | | | | | |
| | AA7075-T73 | | | | | | |
| **Surtec 650 V** | AA2024-T81 | | | | | | More degradation of the protective coating (blistering and corrosion spots); few corrosion products and blisters around a scribe |
| | AA6061-T6 | | | | | | |
| | AA7075-T73 | | | | | | |

# Phase 3 –Results (conversion coating)

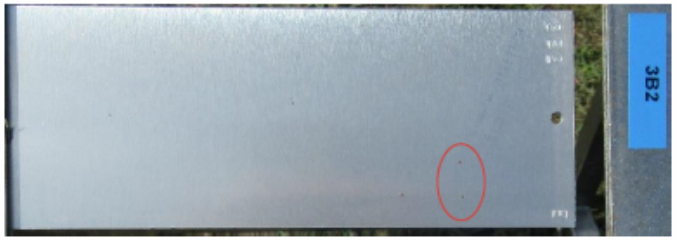
Evidence of first signs of corrosion (after 2 months)




AA2024-T81 pre-treated by the Alodine 1200 S – After 1st Month

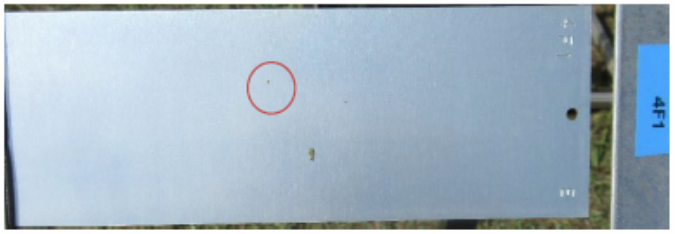



AA7075-T73 pre-treated by the Alodine 1200 S – After 2nd Month




AA6061-T6 pre-treated by the Precoat A32 – After 1st Month


AA6061-T6 pre-treated by the Surtec 650V – After 2nd Month

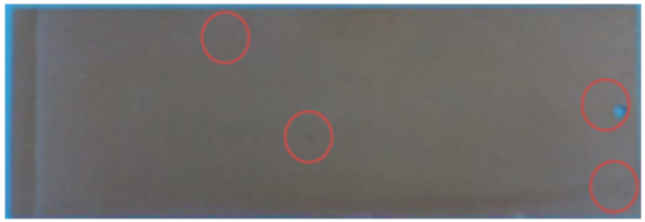



AA7075-T73 pre-treated by the Surtec 650V – After 1st Month

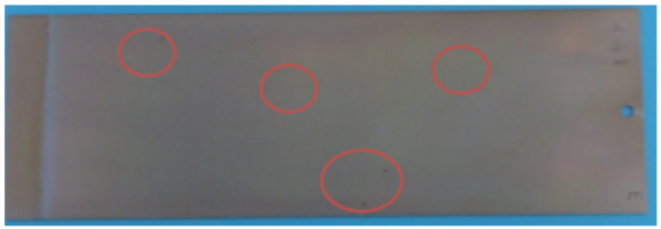

AA2024-T81 pre-treated by the Socosurf TCS/ PACS – After 2nd Month

# Phase 3 –Results (conversion coating)

Evidence of signs of corrosion (after 7 months)

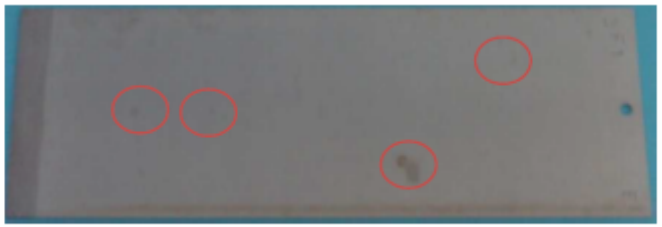


AA2024-T81 pre-treated by the Alodine 1200 S – After 7th Month



AA7075-T73 pre-treated by the Alodine 1200 S – After 7th Month

AA6061-T6 pre-treated by the Precoat A32 – After 7th Month



AA6061-T6 pre-treated by the Surtec 650V – After 7th Month

AA7075-T73 pre-treated by the Surtec 650V – After 7th Month

AA2024-T81 pre-treated by the Socosurf TCS/ PACS – After 7th Month

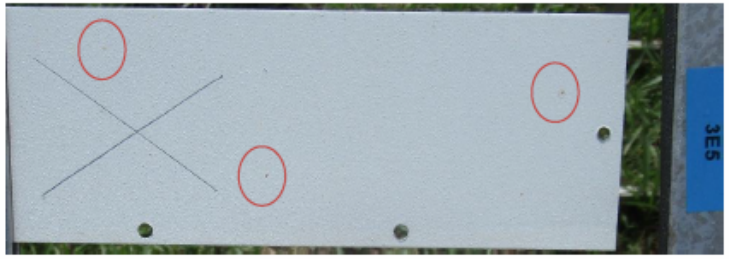
# Phase 3 –Results (primer/topcoat)

| CCC | Aluminium Alloys | Inspection/ Exposure Time | | | | | |
|---|---|---|---|---|---|---|---|
| | | After 1st Day | After 7th Day | After 1st Month | After 2nd Month | After 5th Month | After 7th Month |
| Alodine 1200 S | AA2024-T81 | No evidences of surface defects (blistering, cracking, flaking, delamination and corrosion around a scribe) | No evidences of surface defects (blistering, cracking, flaking, delamination and corrosion around a scribe) | No/ little degradation of the protective coating (some blistering and corrosion spots); few corrosion products build up in scribe, but no blisters around | More evidence of blistering and corrosion spots on the surface of panels; corrosion products build up in scribes; no blisters and delamination/ corrosion around a scribe | More evidence of degradation of the protective coating (blistering and corrosion spots); corrosion products build up in scribes; some blisters around a scribe; no delamination/ corrosion around a scribe | Blistering and corrosion spots, namely on the AA7075-T73; few corrosion products and blisters around a scribe |
| | AA6061-T6 | | | | | | |
| | AA7075-T73 | | | | | | |
| Precoat A32 | AA2024-T81 | | | Few degradation of the protective coating (blistering and corrosion spots); few corrosion products build up in scribe, but no blisters around | | | |
| | AA6061-T6 | | | | | | |
| | AA7075-T73 | | | | | | |
| Socosurf TCS/PACS | AA2024-T81 | | | Few degradation of the protective coating (blistering and corrosion spots on the surface of panels); few corrosion products build up in scribe, but no blisters around | More evidence of degradation of the protective coating (blistering and corrosion spots); corrosion products build up in scribes; no blisters and delamination/ corrosion around a scribe | | |
| | AA6061-T6 | | | | | | |
| | AA7075-T73 | | | | | | |
| Surtec 650 V | AA2024-T81 | | | | | | More degradation of the protective coating (blistering and corrosion spots); few corrosion products and blisters around a scribe |
| | AA6061-T6 | | | | | | |
| | AA7075-T73 | | | | | | |

# Phase 3 –Preliminary results (primer/topcoat)

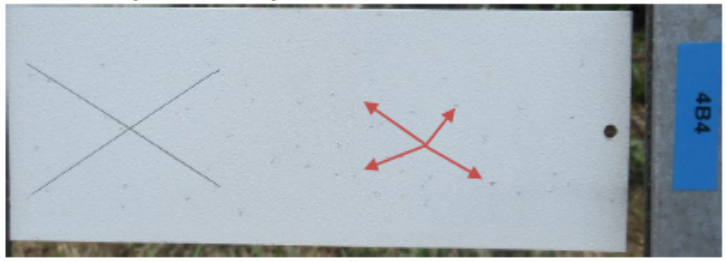
Evidence of first signs of corrosion


AA7075-T73 pre-treated by the Alodine 1200 S – After 1st Month

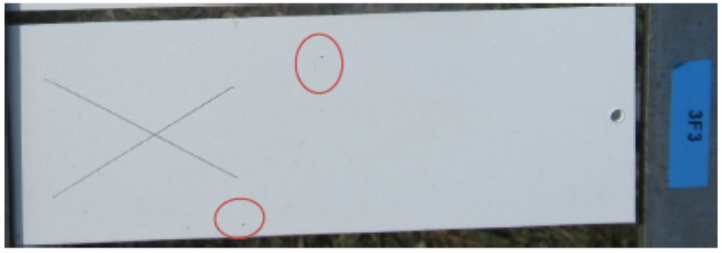



AA6061-T6 pre-treated by the Socosurf TCS/PACS – After 2nd Month

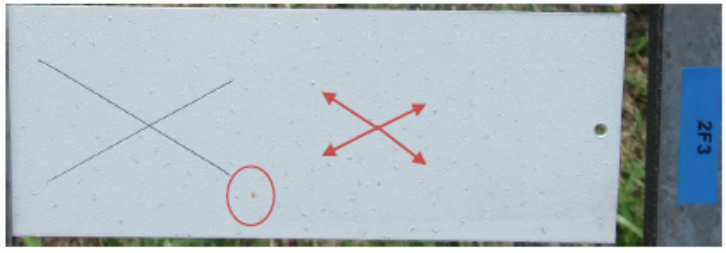

AA2024-T81 pre-treated by the Precoat A32 – After 1st Month




AA7075-T73 pre-treated by the Precoat A32 – After 2nd Month



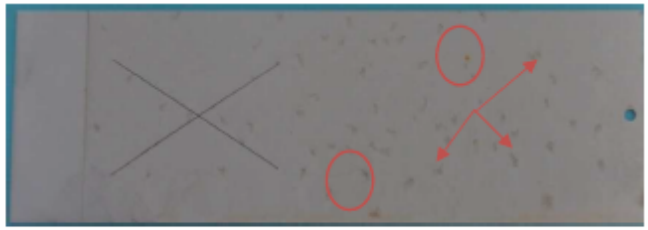

AA6061-T6 pre-treated by the Surtec 650V – After 1st Month



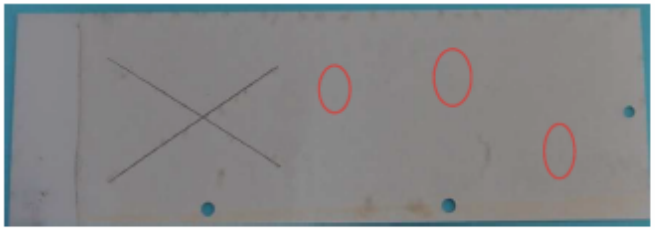

AA2024-T81 pre-treated by the Surtec 650V– After 2nd Month

# Phase 3 – Results (primer/topcoat)

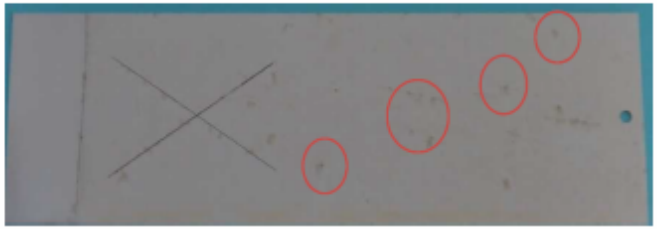
Evidence of signs of corrosion (after 7 months)



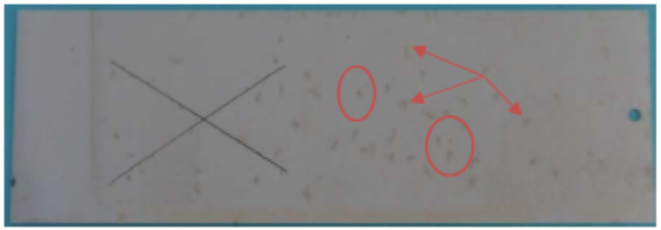
AA7075-T73 pre-treated by the Alodine 1200 S – After 7th Month



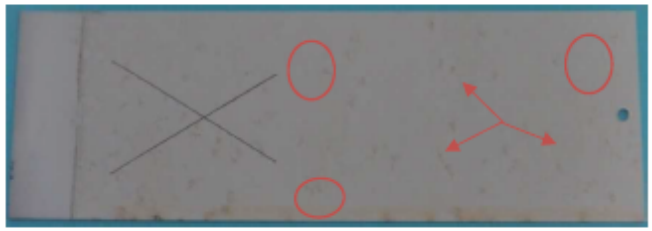
AA6061-T6 pre-treated by the Socosurf TCS/PACS – After 7th Month



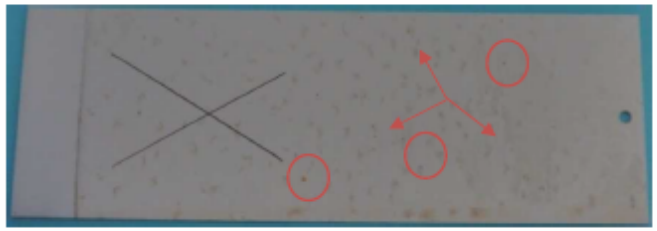
AA2024-T81 pre-treated by the Precoat A32 – After 7th Month



AA7075-T73 pre-treated by the Precoat A32 – After 7th Month



AA6061-T6 pre-treated by the Surtec 650V – After 7th Month



AA2024-T81 pre-treated by the Surtec 650V– After 7th Month

# ECD Program

## Main achievements and current status

# Company Introduction

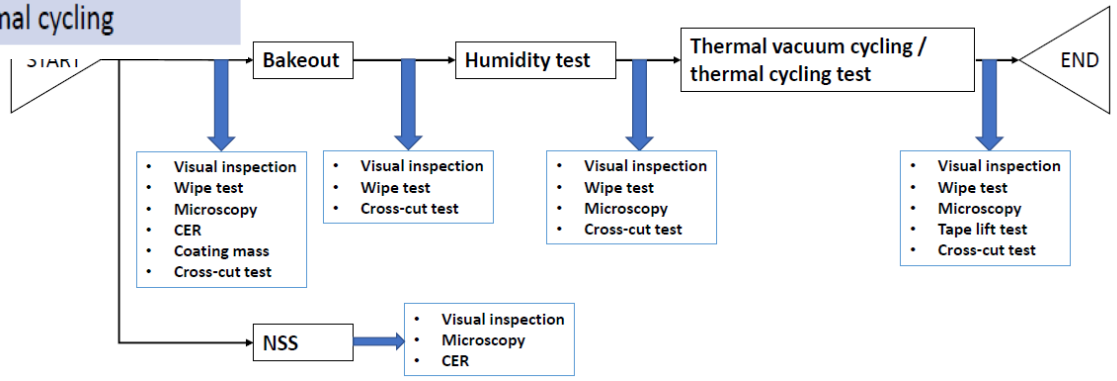
- **MATMOD Ltd.** Was founded in 2007 primarily for the management of Hungarian Cluster (http://hunspace.org/)
- HUNSPACE is a cluster organization for the advocacy of industrial partners in the aerospace segment
- **MATMOD Ltd. (**http://www.matmod.eu): has significant contribution to R&D activities for the support of satellite industry in Hungary in the field of materials science
- **Tenders:**

| Hungarian | Europe (ESA) |
|---|---|
| ÉMOP-1.2.1-2008-0029 | ESA PECS - Environment Friendly Conversion Coating Development I. (ECD-1) |
| ÉMOP-1.2.1-11-2011-0002 | ESA HIS - Environment Friendly Conversion Coating Development II. (ECD-2) |
| GOP-2.1.1M-11/M-2011-0219 | ESA HIS - Environment Friendly Conversion Coating Development III. (ECD-3) |
| GOP-2.1.1-11/M-2012-3841 | |
| GINOP-2.1.8-17-2017-00017 | |

# ECD 1: Organization of the project

- **Prequalification phase**: several destructive and reduced environmental test on chromated and painted test samples
- **Qualification phase**: destructive and environmental test on qualification test samples and flight representative hardware

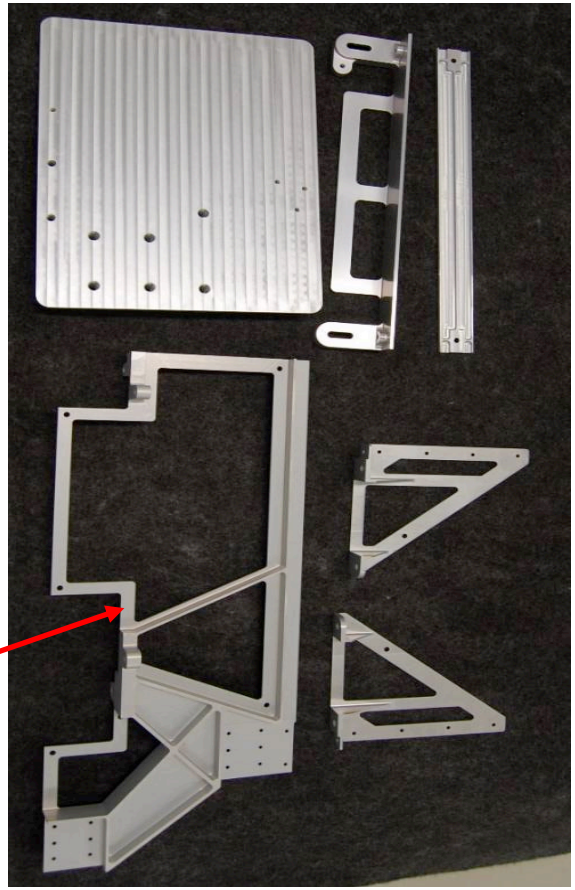
| Non-destructive tests / inspection | Destructive tests | Environmental tests |
|---|---|---|
| Visual inspection | Contact electrical resistance test | Neutral salt spray test |
| Dry/wet wipe test | Coating mass measurement | Bakeout |
| Optical microscopy | Cross-cut test | Humidity |
| Paint thickness measurement | Tape lift test | Thermal vacuum cycling |
| | | Thermal cycling |



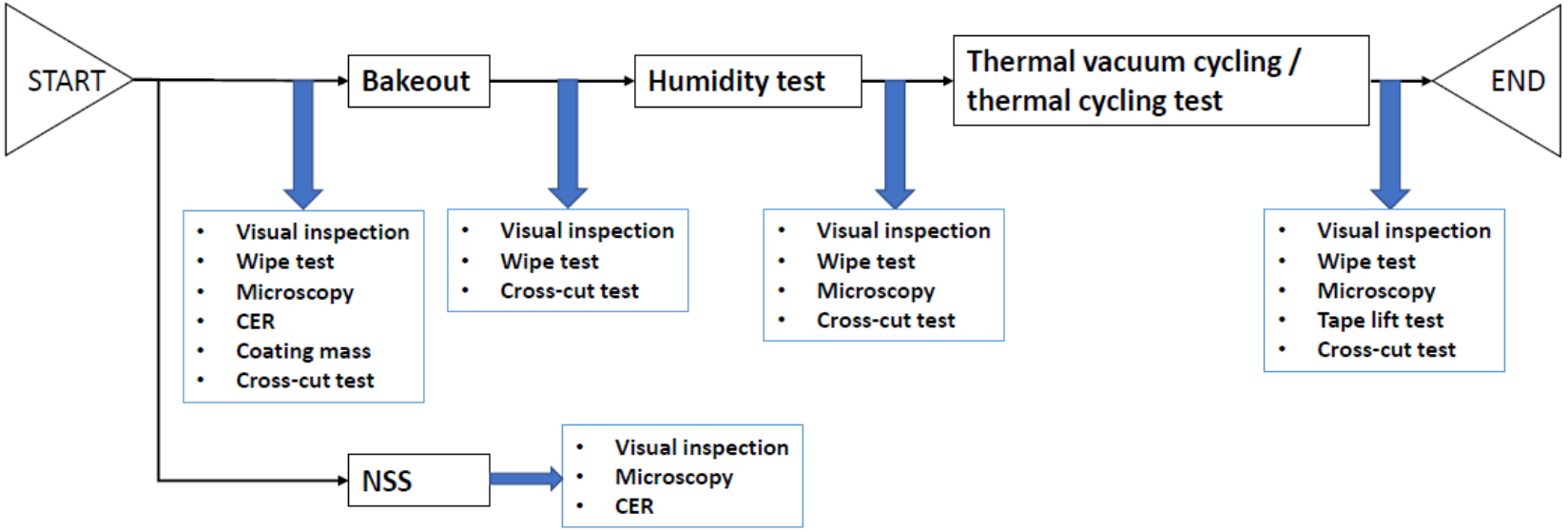
# ECD1: Materials and surface treatment selected

- Alloys selected:
  - EN AW 6063 T651
  - EN AW 6082 T651
  - EN AW 7075 T7351
- All substrate have been treated with Surtec 650 RTU
- Flight representatives samples were used

| Name | Alloy | Coating | Topcoat |
|---|---|---|---|
| Sentinel-2 SWIR Harness Radiator | 6082 T651 | SURTEC 650RTU | E' + SG121FD |
| Sentinel-2 SWIR Harness Radiator Compression Plate | 6082 T651 | SURTEC 650RTU | None. |
| CHEOPS FEE Radiator | 6063 T651 | SURTEC 650RTU | SG121FD |
| Sentinel-2 Telescope Baffle 1 | 7075 T7351 | SURTEC 650RTU | E' + PU1 |
| Sentinel-2 Baffle Vane PZ (+Y) | 6082 | SURTEC 650RTU | PU1 |
| Sentinel-2 VCU Connector Bracket | 6082 | SURTEC 650RTU | None. |
| Sentinel-2 VCU Connector Bracket | 6082 | SURTEC 650RTU | None. |



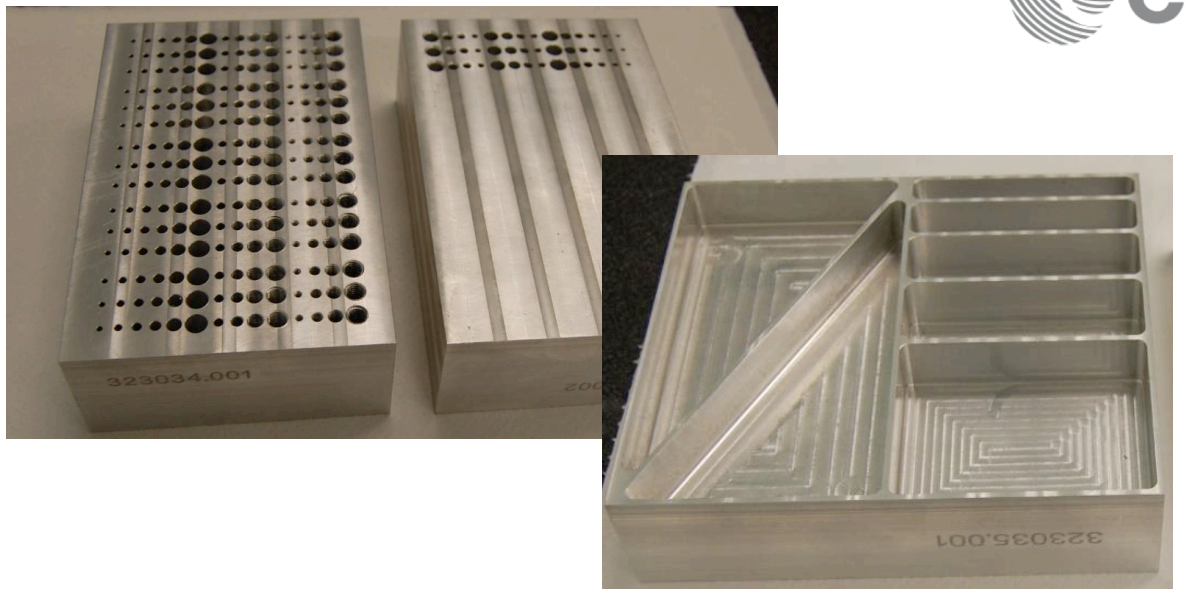
# ECD 1:Results of the qualification campaign




- More than 80 samples together with flight representative hardware were investigated using visual inspection, dry/wet wipe test, microscopy, layer weight measurement, contact electrical resistance and tape lift test
- Test items were subjected to environmental test like bake out, humidity, thermal vacuum cycling and thermal cycling. NSST were also performed.
- All test have been passed successfully

# ECD2 (continuation of ECDI)

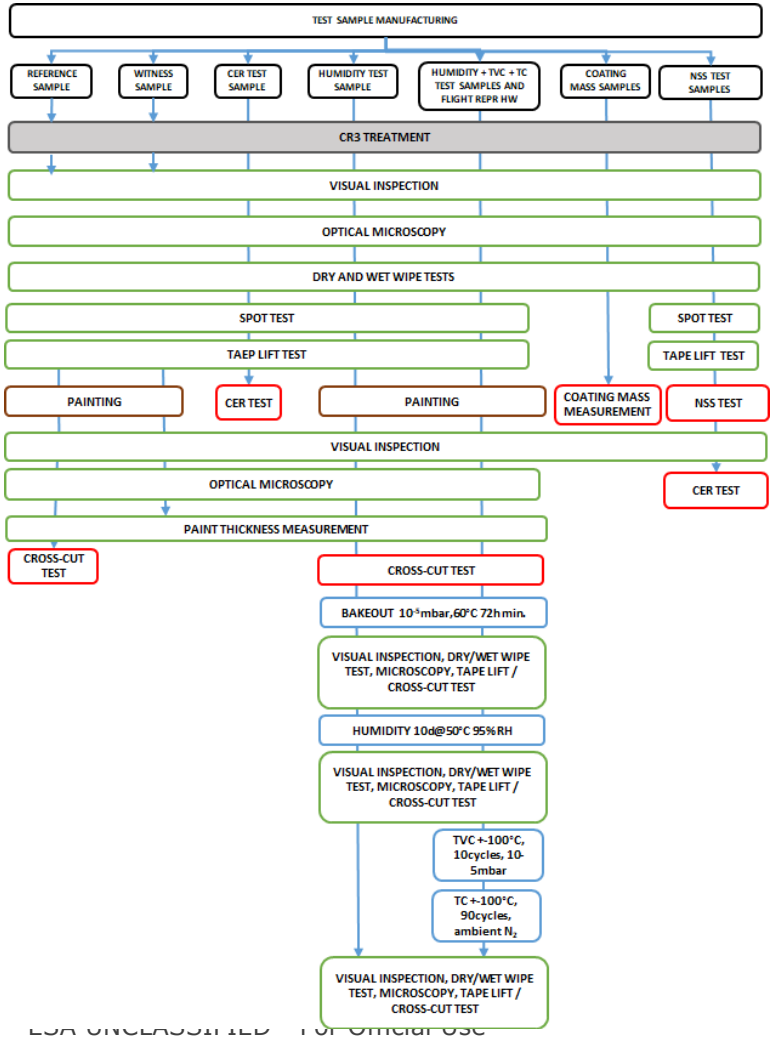
Selected Alloys:
- EN AW 1050 H0 sheet metal
- EN AW 2024 T351
- EN AW 2024 T851
- EN AW 5052 H22
- EN AW 6061 T651
- EN AW 6061 T6 sheet metal
- EN AW 6063 T651
- EN AW 6082 T651
- EN AW 6082 T6 sheet metal
- EN AW 7075 T7351




Conversion coatings:
- Surtec 650
- Surtec 650 RTU
- Surtec 650V

Topcoat:
- MAP PUK (black paint)
- MAP SG121FD (white paint)
- MAP E‘ (primer)
- LORD (Socomore)Aeroglaze Z306
- LORD (Socomore)Aeroglaze 9741
- LORD (Socomore) Aeroglaze 9947

# ECD2 (continuation of ECDI) – Test Program




**150 samples!**

- 1050 sheet metal + SURTEC 650 RTU
- 2024 T351 + SURTEC 650V
- 2024 T851 + SURTEC 650V
- 2024 T851 + SURTEC 650
- EN AW 5052 H22 + SURTEC 650 RTU
- EN AW 5052 H22 + SURTEC 650 RTU + PUK
- EN AW 5052 H22 + SURTEC 650 RTU + E’ + PUK
- EN AW 6061 T651
- EN AW 6061 T6 sheet metal
- EN AW 6082 T651 + SURTEC 650 RTU (brush application)
- EN AW 6082 T651 + SURTEC 650 RTU (brush application) + PUK
- EN AW 6082 T651 + SURTEC 650 RTU (brush application) + E’ + PUK
- EN AW 6082 T651 + SURTEC 650 RTU (brush application) + SG121FD
- EN AW 6082 T651 + SURTEC 650 RTU (brush application) + E’ + SG121FD
- EN AW 6082 T651 + SURTEC 650 RTU (brush application) + Z306
- EN AW 6082 T651 + SURTEC 650 RTU (brush application) + 9741 + Z306
- EN AW 6082 T651 + 9947 + Z306
- EN AW 6082 T6 sheet metal

# ECD2 (continuation of ECDI) – TCP results

| | | Alloy/ coating type | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Alloy type | 1050 H0 sheet metal | 2024 T351 | 2024 T851 | 2024 T851 | 5052 H22 | 6061 T651 | 6061 T6 sheet metal | 6082 T651 | 6082 T651 | 6082 T6 sheet metal |
| Conversion coating | SURTEC 650 RTU | SURTEC 650V | SURTEC 650 | SURTEC 650V | SURTEC 650 RTU | SURTEC 650 RTU | SURTEC 650 RTU | SURTEC 650 | SURTEC 650 RTU (brush method) | SURTEC 650 RTU |
| Optical microscopy | PASS | PASS | PASS | PASS | PASS | PASS | PASS | NA | PASS | PASS |
| Tape lift test | PASS | PASS | PASS | PASS | PASS | PASS | PASS | NA | PASS | PASS |
| | AFTER HUMIDITY, 240h, 50°C, 95% RH | | | | | | | | | |
| Visual inspection | PASS | PASS | PASS | PASS | PASS | PASS | PASS | NA | PASS | PASS |
| Dry wipe test | PASS | PASS | PASS | PASS | PASS | PASS | PASS | NA | PASS | PASS |
| Wet wipe test | PASS | PASS | PASS | PASS | PASS | PASS | PASS | NA | PASS | PASS |
| Optical microscopy | PASS | PASS | PASS | PASS | PASS | PASS | PASS | NA | PASS | PASS |
| Tape lift test | PASS | PASS | PASS | PASS | PASS | PASS | PASS | NA | PASS | PASS |
| | AFTER TVC + TC TESTS, 10 cycles in ambient atmosphere, 90 cycles in $10^{-5}$mbar, ±100°C, 15min plateau | | | | | | | | | |
| Visual inspection | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS |
| Dry wipe test | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS |
| Wet wipe test | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS |

Test Campaign Passed Successfully!

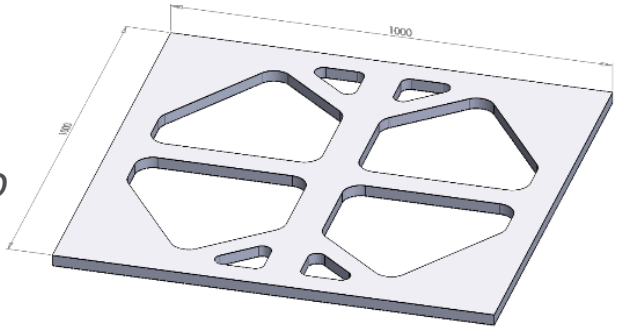
# ECD2 (continuation of ECDI) – TCP + topcoat results

| | Alloy/ coating type | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| Alloy type | 5052 H22 | 5052 H22 | 6082 T651 | 6082 T651 | 6082 T651 | 6082 T651 | 6082 T651 | 6082 T651 | 6082 T651 |
| Conversion coating | SURTEC 650 RTU | SURTEC 650 RTU | SURTEC 650 RTU (brush method) | | | | | | N/A |
| Topcoat | PUK | E' + PUK | PUK | E' + PUK | SG121FD | E' + SG121FD | Aeroglaze Z306 | Aeroglaze 9741+Z306 | Aeroglaze 9947+Z306 |
| Optical microscopy | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS |
| AFTER HUMIDITY, 240h, 50°C, 95% RH | | | | | | | | | |
| Visual inspection | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS |
| Average cross – cut test classification | 0 | 0 | 0,3 | 0 | 0 | 0 | 0,4 | 0 | 0 |
| Optical microscopy | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS |
| AFTER TVC + TC TESTS, 10 cycles in ambient atmosphere, 90 cycles in $10^{-5}$mbar, ±100°C, 15min plateau | | | | | | | | | |
| Visual inspection | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS |
| Average cross – cut test classification | 0 | 0 | 0,8 | 0 | 0 | 0 | 0 | 0 | 0 |
| Optical microscopy | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS | PASS |

Test Campaign Passed Successfully!

# ECD 3: Environment friendly conversion coating development for large parts

*Technical objective is to develop a conversion coating process for large parts made of aluminum alloys using SURTEC650 chemical family. The process is based on spray and/or brush method instead of immersion to be able to treat large satellite parts like sandwich panels and adaptor rings.*



Selected Alloys:

- EN AW 5052 H22
- EN AW 5005 H24
- EN AW 6082 T651
- EN AW 6082 sheet metal
- EN AW 7075 T7351

+ SurTec +

Selected topcoat:
MAP PU1
MAP SG121FD
Aeroglaze Z306.

# Current Status

| Alloy type | Conversion coating | TRL |
|---|---|---|
| 1050 | SURTEC 650 | 5 |
| 2024 T351 /T851 | SURTEC 650 | 5 |
| 5052 H22 | SURTEC 650 | 5 |
| 6061 | SURTEC 650 | 5 |
| 6082 T651 | SURTEC 650 | 5 |
| 6082 T6 | SURTEC 650 | 8 |
| 7075 | SURTEC 650 | 5 |

**Topcoats (TRL5):**

- MAP PU1
- MAP E'+PU1
- MAP PUK
- MAP E'+PUK
- MAP SG121FD
- MAP E'+SG121FD
- Aeroglaze Z306

# Main Achievements

- Parts coated with hexavalent chromate conversion coating were produced until 2017 for **SENTINEL-2 A&B** and **CHEOPS** mission in collaboration with ADMATIS Ltd.
- R&D work on trivalent chromium conversion coating (SURTEC 650) was started in 2013 in the frame of a national program called MAF.
- Application of TCP coating on space metallic HW was implemented in 2017 in **SENTINEL 2 C&D**

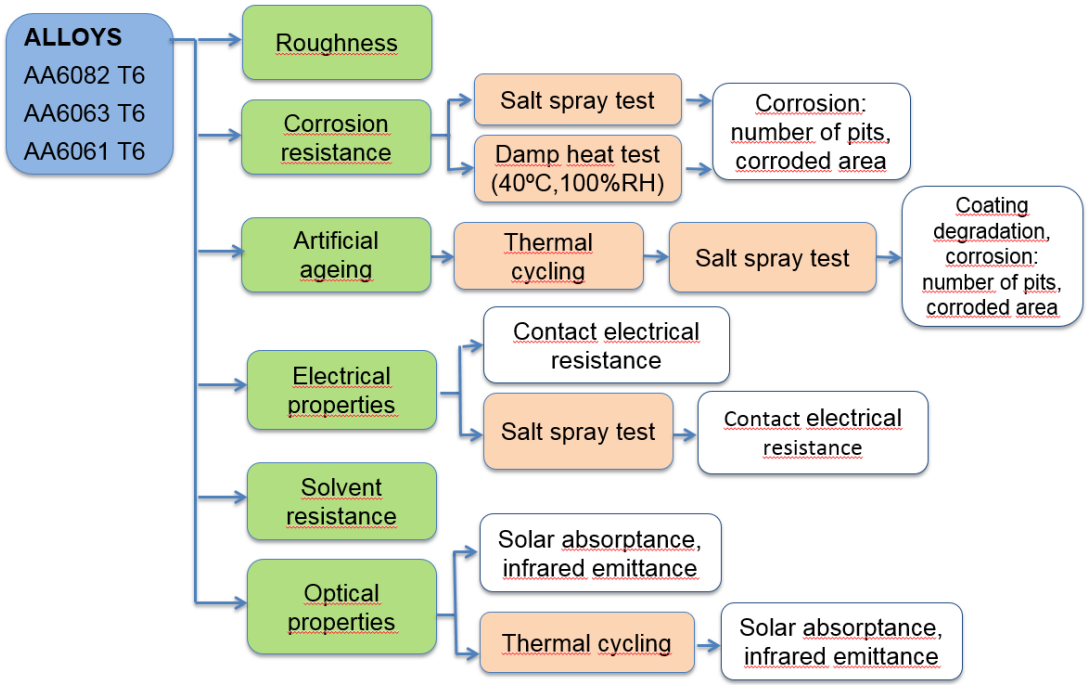
- A full characterization and testing of the conversion coating in combination with other well known paints was conducted in the frame of the ECD projects supported by ESA.
- ECD is continuously widening the applicability of TCP on different aluminum alloys.
- Touch up and application by brush performed on large parts (1 m2) is the main focus of the ECD 3 activity currently running.

# *IRUCOAT4SPACE Program*

## *Current status*

# Main Objective and preliminary test program

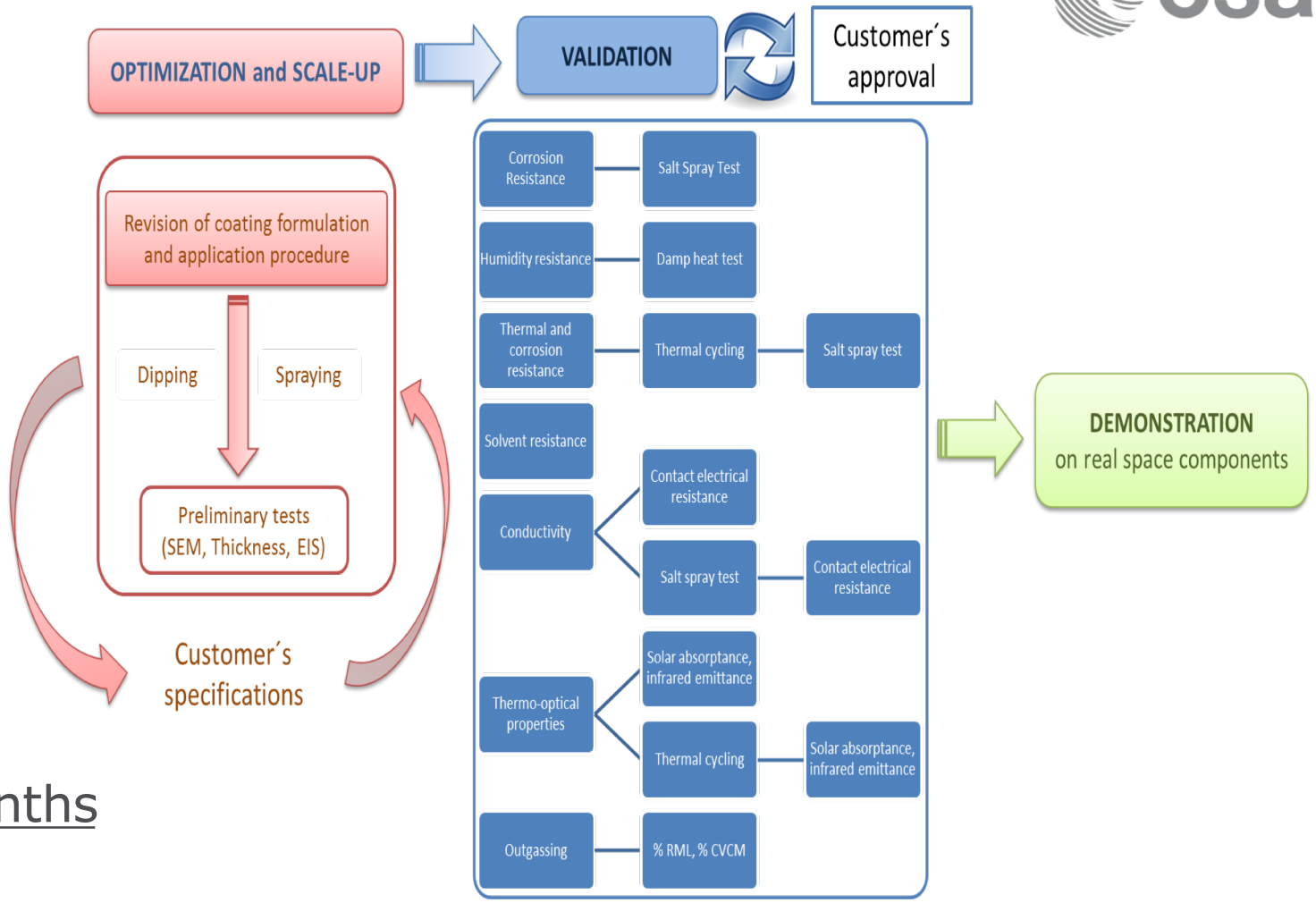
- Hybrid Material Based on SiO2-ZrO2-Organic Interconnected Network
- Synthesized by sol-gel technique and dip-coating deposition (spraying in process)
- Protection on Zn-coated Steel (delay of Zn and Steel corrosión)
- Alkaline, wear and UV resistant
- Excellent matrix for incorporation of other functional components: corrosion inhibitor, dyes, nanoparticles, conductive polymers
- Thickness range: 1.5 - 4 microns
- IRUCOAT patent application: EP16382471 Dec 2016 and PCT/EP2017/076375 October 2017



# Current Project (ESA funded) – Summary

The main objective of this 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.

Basic characterization performed on AA2024




## Project duration: 24 months

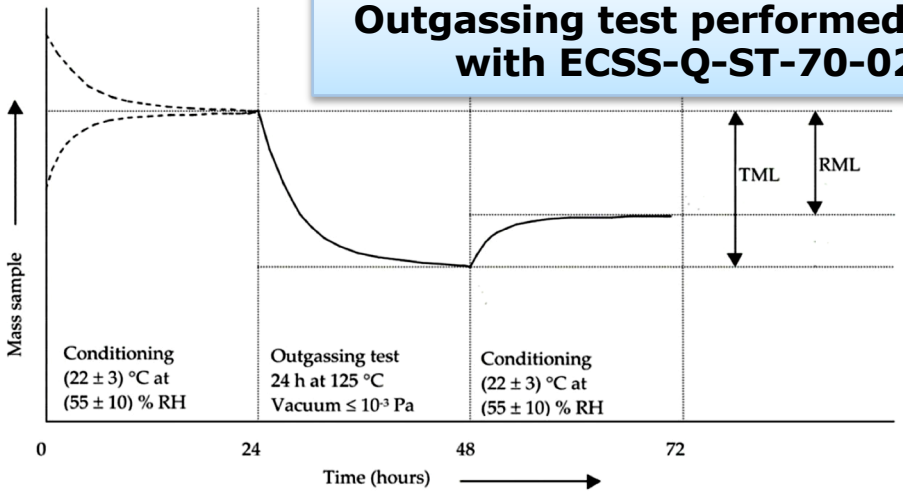
# WP 2: Optimization and scale-up - Dipping

- Modification of Irucoat formulation:
  - Substitution of one precursor to make it methanol free (BB3E)
  - Optimization of curing temperature and time
  - Optimization of organic/inorganic ratio
- Basic characterization of dip-coated samples (AA2024)
  - Coating thickness: 2.5 – 3 µm
  - Electrochemical Impedence Spectroscopy (EIS)
  - SEM analysis
  - Salt Spray Test (SST)
  - Outgassing

Priority has been given to Task 2.2, as deposition by spraying is more challenging and time consuming

# WP 2: Optimization and scale-up - Dipping

**Outgassing test performed in line with ECSS-Q-ST-70-02C**




**Test Requirement**

- RML < 1.00% and
- CVCM <0.10%

**TML:** Total Mass Loss
**RML:** Regained Mass Loss
**CVCM:** Collected Volatile Condensable Material

**5 different formulations tested with different curing parameters**

| Ref # | Reference material | Curing T (ºC) | Curing time | $d_{coat}$ (mg/cm$^2$) | TML% | RML% | CVCM% |
|---|---|---|---|---|---|---|---|
| 19183 | BB3Mc-180 | 180 | 1 h | 0.8 | 5.483 | 1.318 | 0.006 |
| 19182 | BB3M-180 | 180 | 1 h | 0.54 | 5.157 | 1.404 | 0.009 |
| 19154 | BB3E-180 | 180 | 1 h | ~0.86 | 4.398 | 1.831 | 0.028 |
| 19184 | BB3M_HC-180 | 180 | 1 h | 1.35 | 3.551 | 1.791 | 0.303 |
| 19156 | BB3E_HC-180 | 180 | 1 h | ~1.28 | 3.4 | 2.339 | 0.311 |
| 19106 | BB3E-120 | 120 | 8 h | 0.86 | 8.225 | 5.231 | 2.357 |
| 19155 | BB3E_HC-120 | 120 | 8 h | ~1.47 | 18.135 | 17.04 | 7.024 |

**Further optimization**

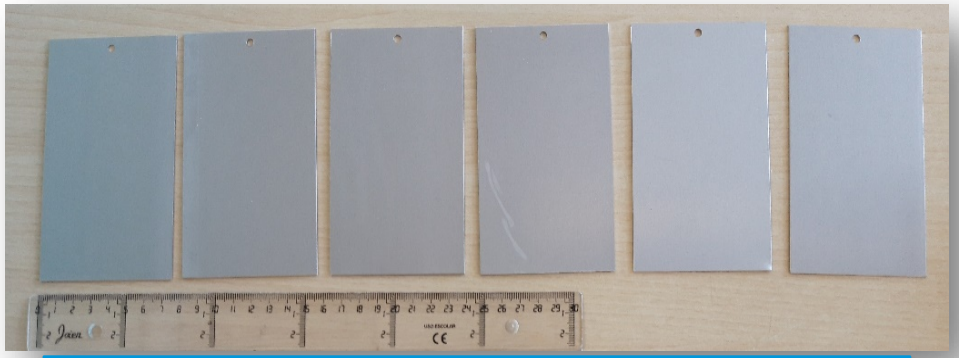
- Formulation(s) **BB3Mc**
- Curing conditions:
  - 180°C for 2 hours
  - 150°C for 5 hours

# WP 2: Optimization and scale-up - Spraying

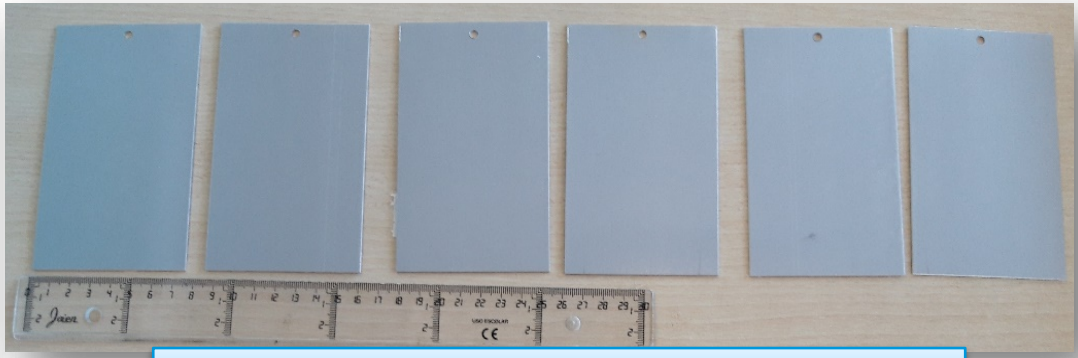
- Modification of IRUCOAT formulation (in collaboration with external services)

  Dry extract of IRUCOAT: 39,21% → dilution with 1-methoxy-2-propanol and filtration 1µm
- Optimization of Spraying and Deep Coating parameters (2 series of samples)
- All samples were cured for 8h @ 120°C

Preliminary Results - Visually, the coating appears quite homogeneous, although further improvement in the spraying technique is needed



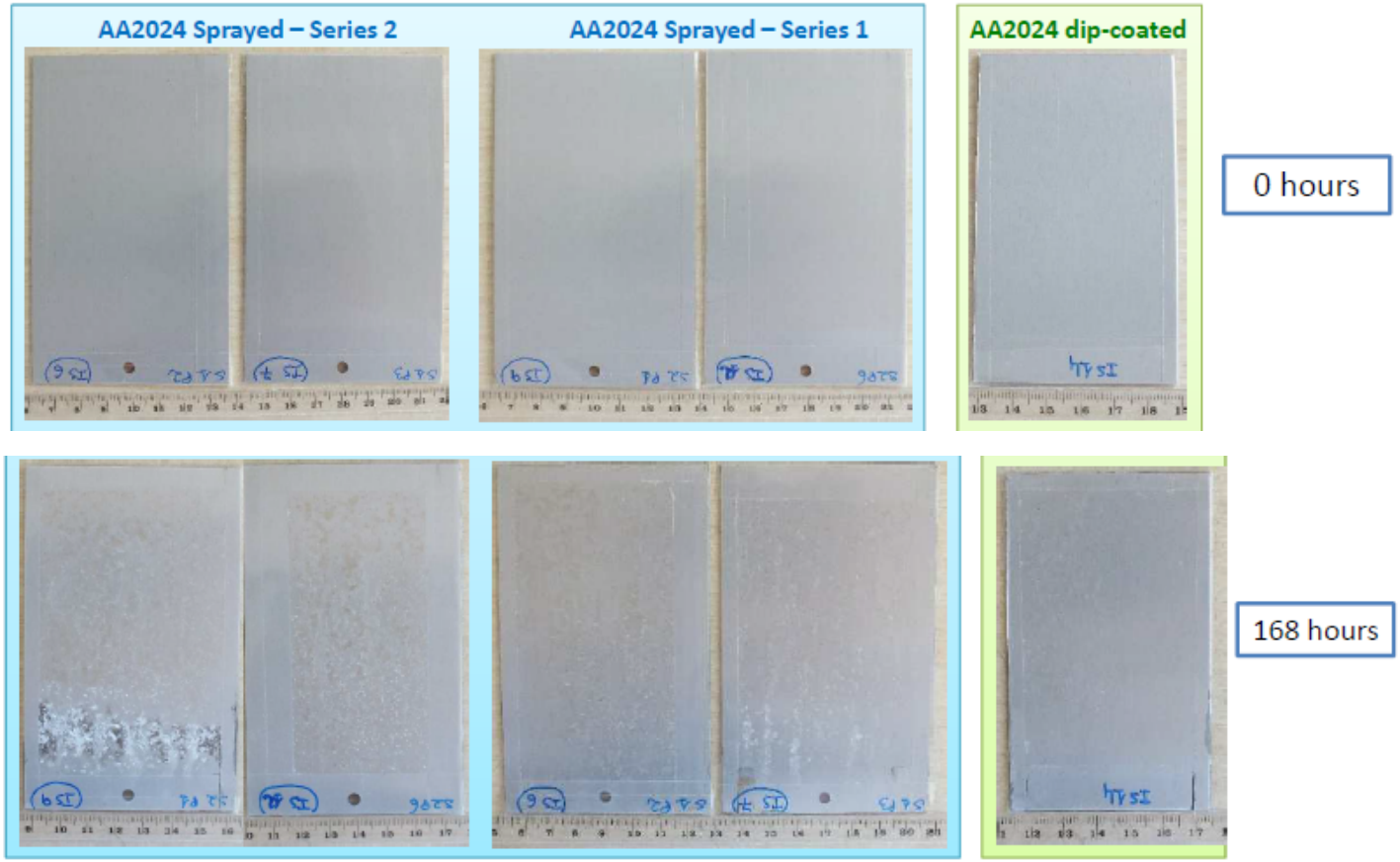
Series 1 : thickness around 3-3.3 µm



Series 2: thickness around 3.5 µm

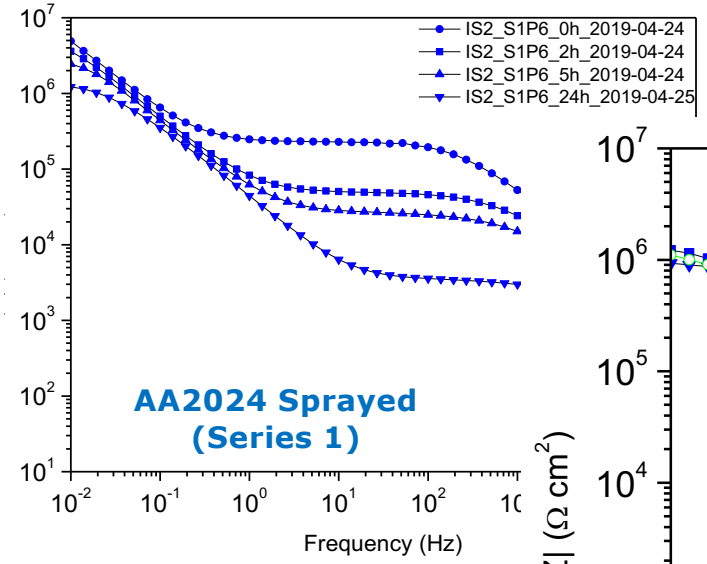
# WP 2: Optimization and scale-up - Spraying

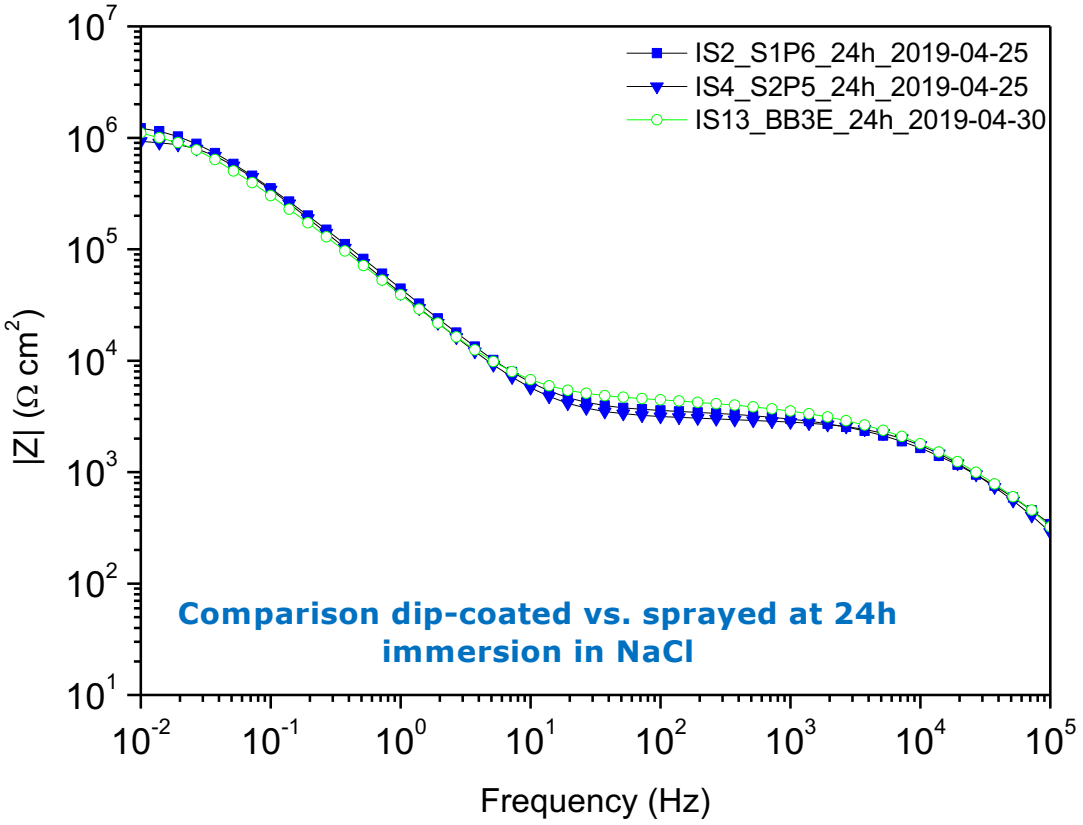
**SSF exposure**

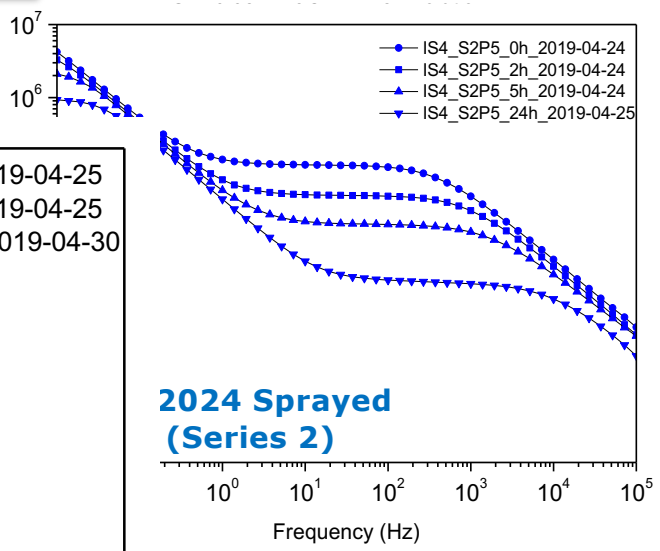


# Current Project (ESA funded) – Status

**EIS of samples in 0.05 M NaCl**



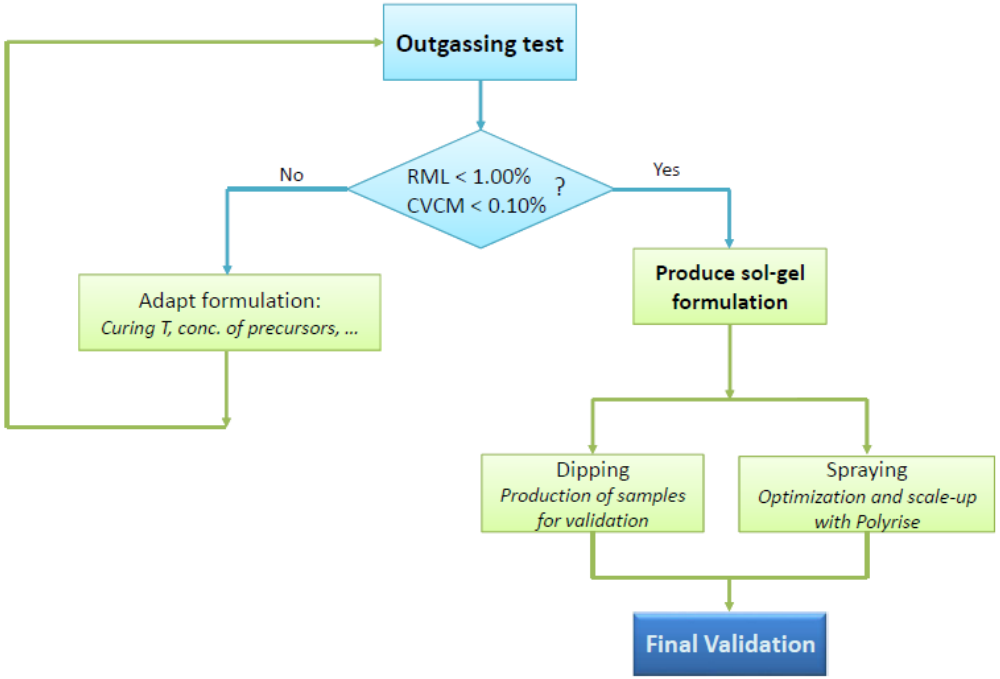
# On going/future steps

**Achievements**

- Visually, the sprayed coating is quite homogeneous, although further improvement in the spraying technique is needed
- Corrosion assessment (SST and EIS) gave satisfactory results: the performance of the sprayed coating is comparable to the dipped one.

**Next Steps**

- Scale-up of coating application (larger sample)
- Optimization of outgassing test results (the final formulation will depend on this test)



# Cr6-free CC qualifications through MPTB

## Qualification performed as part of Projects Activities

**Scope: to keep track of the qualification status performed by Industry as part of Project Activity**

A template has been provided to the MPTB members and shared in the common folder.


## Samples exposure in real cleanroom environment

**Scope: to collect data of the real exposure in cleanroom environment to verify the actual behavior of the coatings and the reliability of accelerated tests.**

5 different cleanroom have been identified, located in different places in Europe. A set of samples treated with CrVI alternative tratments will be delivered

| |
|---|
| ESTEC |
| Ruag Zurich |
| Thales Torino |
| Airbus Toulouse |
| Thales Cannes |

# Conclusion and next step

- Several activities are running in parallel, not limited to ESA funding
- Based on the results of the test campaigns some promising conversion treatments for specific alloys have been identified
- Qualification activities are held in parallel by Industry and some substrate/conversion coating combinations are already qualified successfully for future mission
- Based on the experience acquired from all the activities performed on this topic a guideline to be used as baseline for project qualification has been issued from Materials and Processes section (ESA-TECMSP-TN-007384 Rev2.)

*However some work remains still to be done concerning:*

- *Most test activities are performed on lab scale and require industrialization (challenge!)*
- *The possibility to identify reliable treatment especially for demanding aluminum alloys (e.g AA 2000 and 7000 series).*
- *The verification of the new system reliability in combination with topcoat/paint and bonding*

# *Lead Free Working Group – Achievements and Next Steps*

# Lead free electronics - Background

- The European Space Sector can no longer avoid the need to plan and implement a managed transition to lead-free electronics. The stimulus comes from:

| Substance CAS number(s) | CLP Annex VI (SVHC class.) | SIN List (ChemSec) | Candidate List (inclusion date) | ECHA Recommendation | Annex XIV | Comments |
|---|---|---|---|---|---|---|
| **Lead 7439-92-1** | Repr. 1A | YES | YES (27/06/2018) | NO, but ECHA draft recommendation may be expected in Sept 2019 for consultation (TBC by mid-June 2019 "MSC-65") | NO – No sunset date today: *worst case could be in mid-late 2024* | RoHS/EEE: Space-related exclusion from scope but commercial threat of obsolescence due to the abandon of lead in some other sectors |

**Substance identity**

**EC / List no.:** 231-100-4

**CAS no.:** 7439-92-1

**Mol. formula:** Pb

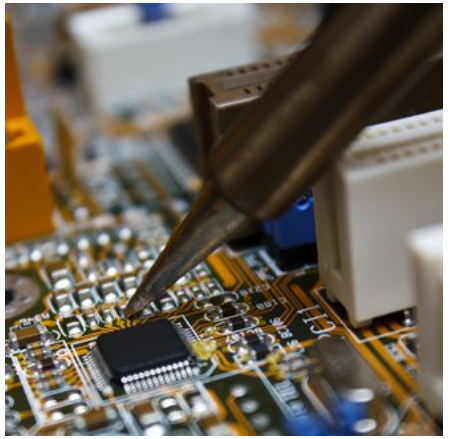
Pb
(v0)

**Hazard classification & labelling**

*Danger!* According to the classification provided by companies to ECHA in **REACH registrations** this substance may damage fertility or the unborn child, causes damage to organs through prolonged or repeated exposure, is very toxic to aquatic life with long lasting effects, may cause cancer, is very toxic to aquatic life and may cause harm to breast-fed children.

At least one company has indicated that the substance classification is affected by impurities or additives.

This substance is covered by several Harmonised Classifications and Labelling's (CLH) entries approved by the European Union. Differentiating between the different CLH's entries requires manual verification. To know more about the CLH please visit the C&L Inventory.

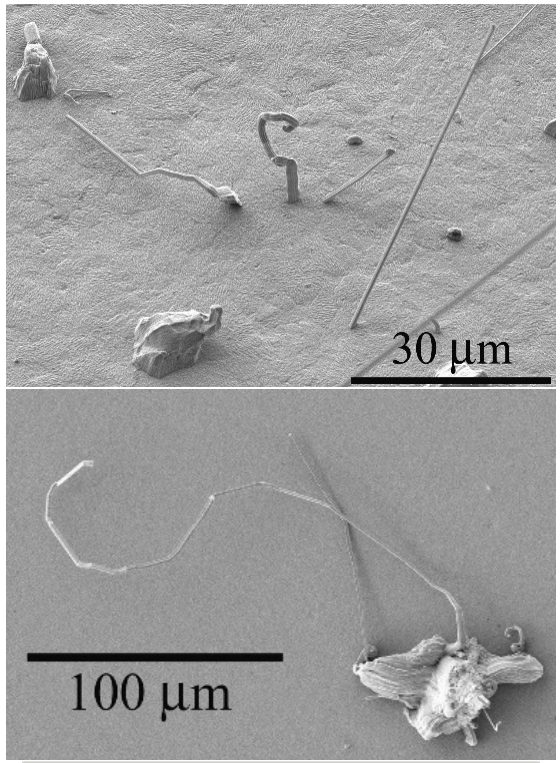
# Assembly of COTS

lead-free

RoHS 2002/95/EC

***Lead-free terminations in the EEE components not contemplated in current applicable ECSS standards***

***Managing the risk of Tin whiskers when pure tin is present***




*Courtesy of NIST (National Institute of Standards and Technology)*

# Lead-free terminations in the EEE components not contemplated in current applicable ECSS standards

The Standards that need to be modified to allow the assembly of those lead-free EEE components (mainly COTS) are:

- **ESCC 23500**, REQUIREMENTS FOR LEAD MATERIALS AND FINISHES FOR COMPONENTS FOR SPACE APPLICATION
- **ECSS-Q-ST-60-13C** Commercial electrical, electronic and electromechanical (EEE) components
- **ECSS-Q-ST-70-61C**, High reliability assembly for surface mount and through hole connections, says that Components lead materials and their finishes shall be selected in compliance with requirements from clause 3 of ESCC23500. However Section 7 (Preparations prior to mounting and soldering) would need to be reviewed.

# Questions to be answered within the working group

## Standards for Lead free terminations

- *Are there any other standards that need to be updated in order to be able to mount components with lead-free terminations?*
- *Is there sufficient data publicly available to support PSWG work updating the relevant standards?*
- *Do we need to include data acquisition tasks in the roadmap?*

## Managing Whiskers Risk

- *Adopt North American documents?*
- *Develop European, ECSS, versions?*

# Introduction of lead free solder

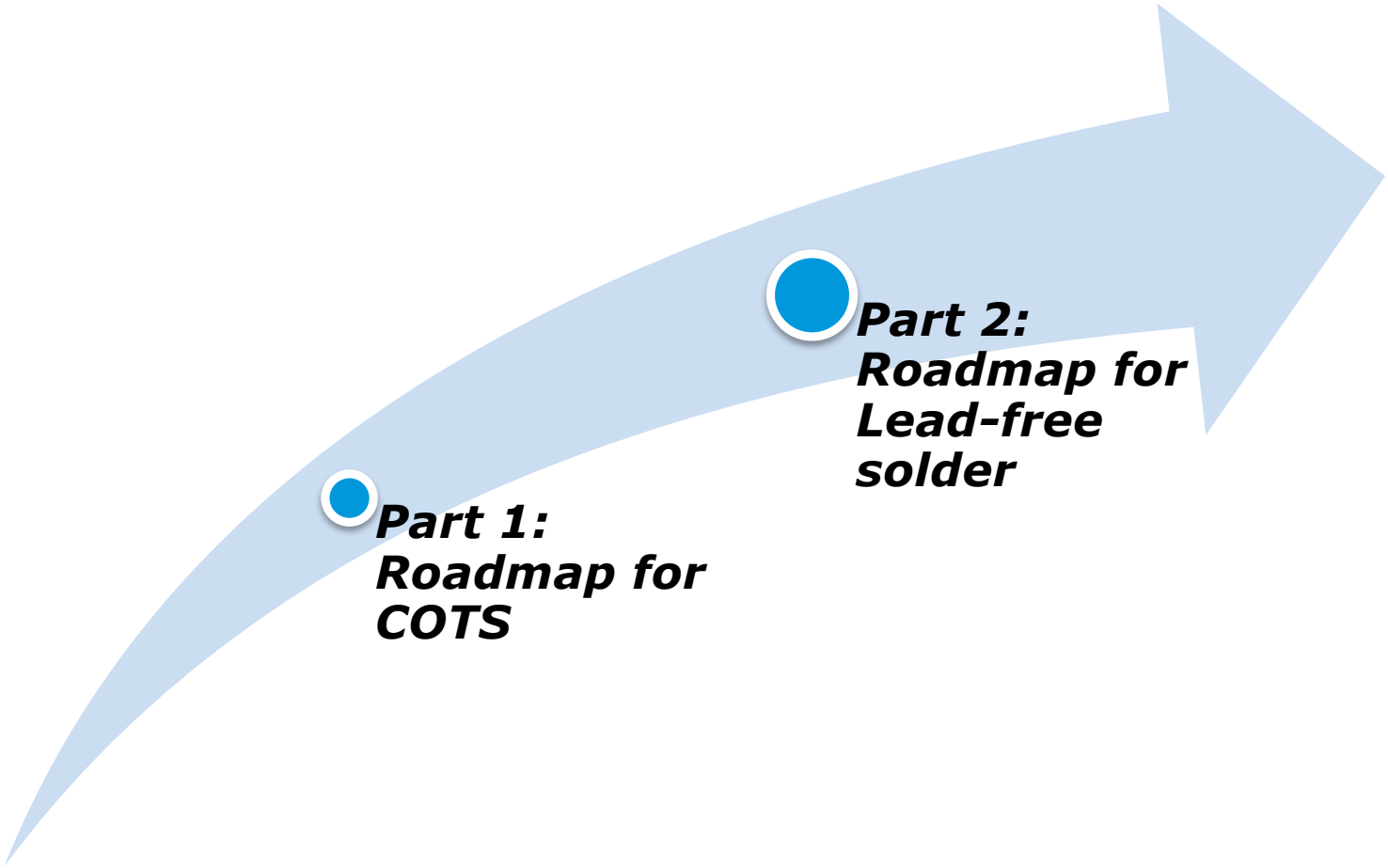
- The Space Sector is not constrained to abandon lead based solders in the short/medium term (< 5 years) due to regulation.
- In short/medium term the obsolescence risks for lead-tin solder paste is low.
- In the long term (>5 years) the Sector must transition to lead-free solders due to potential obsolescence risks.

Additional questions:

One or several lead-free solders?.

What R&D tasks should be included in the Roadmap in order to validate the choices?

# Lead Free Roadmap to be delivered by the end of 2019



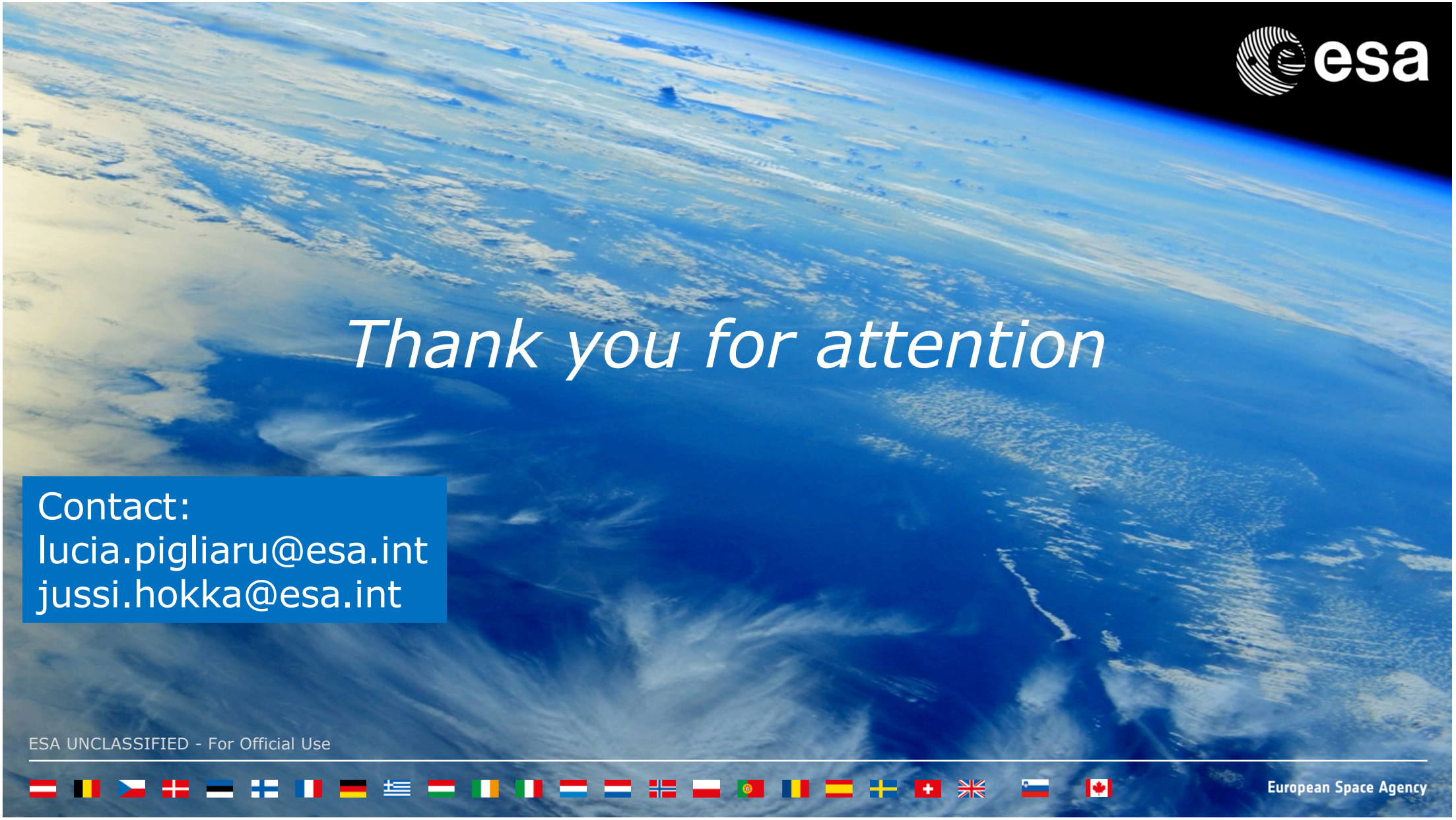
# Part 1: Roadmap for COTS Introduction

1. Lead-free terminations: SAC, Sn (mat or bright, with or without Ni), AuPd, SnCu, AgPd, Ag. SAC is the main choice for BGAs, Sn mat with Ni barrier the main choice for actives and AgPd/Ag the main choices for passives (COTS?).
2. Reliability (other than tin whiskers) of these terminations when assembled with standard qualified SnPb solder
3. Tin whisker mitigation as per GEIA-STD-0005-2. Do we need to propose R&D activities specific for conformal coating? Other mitigation actions to be investigated through R&D?
4. R&D to progress on the understanding of whisker growth ➔ leading to possible accelerated tests that would make it possible to qualify some of the mitigation techniques

# Part 2: Roadmap for Lead-free solder Introduction

R&D required to study the issues associated with lead-free solder:

- Reliability of space components subjected to higher reflow temperature
- Solder spreading and wetting and dissolution of copper metallization on the printed wiring board increase challenge in lead-free assembly and rework.
- Excessive part warpage and flux stability can lead to head on pillow solder interconnects in ball grid array packages. Higher reflow temperature associated with lead-free solders is a factor in head on pillow formation.
- Pad cratering failures with lead-free assemblies. Cratering has been observed in under temperature cycling, vibration and shock loading conditions
- PCBs: Lead-free Reflow Increases CFF (Conductive filament formation), that can produce unintended shorts
- PCB Creep Corrosion
- Durability and fatigue of Lead Free Solder


esa
Thank you for attention
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