TRIVALENT CHROMIUM PASSIVATION
ENHANCEMENT OF THE LAYER FORMATION BY SURFACE ACTIVE SUBSTANCES

ASETS, Denver, Aug 2018
SURTEC 650 - TRIVALENT CHROMIUM PASSIVATION

Background

SurTec 650

Aluminium Bulk

Passivation Layer
approx. 50-100 nm thick

On QPL - Mil 81706 B , 1A, 3

Global availability

Widely used in

- Aviation
- Automotive Industry
- Electronic Industry
To fundamentally understand the layer formation process and the composition of the deposit, the trivalent chromium passivation SurTec 650 was applied on EN-AW 2024.

Topography
- Scanning Electron Microscopy (SEM)

Composition
- Energy Dispersive X-ray Spectroscopy (EDX)
- X-ray Photoelectron Spectroscopy (XPS)

Mechanism of the layer formation
- Electrochemical Potential in situ during the passivation
SURTEC 650 - TRIVALENT CHROMIUM PASSIVATION
Al EN -AW 2024 Metallographic Structure

Copper-rich intermetallic phases.

Cross section of EN-AW 2024, colour etched with Kroll’s reagent
The brownish areas indicate the copper precipitations in the material.
SURTEC 650 - TRIVALENT CHROMIUM PASSIVATION
Topography and composition - SEM/EDX

SurTec 650 - Morphology after different immersion times

<table>
<thead>
<tr>
<th>0 s</th>
<th>30 s</th>
<th>300 s</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="5%C2%B5m" alt="Image" /></td>
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SURTEC 650 - TRIVALENT CHROMIUM PASSIVATION
Composition - XPS Element Spectra

Aluminium

Zirconium

Chromium
SURTEC 650 - TRIVALENT CHROMIUM PASSIVATION
Layer formation - Based on SEM/EDX, XPS, Electrochemical Potential

- Cr\textsuperscript{3+}-Zr\textsuperscript{4+}-mixed oxides
- mixed oxides + aluminium fluoride
- Cr\textsuperscript{3+}-Zr\textsuperscript{4+}-mixed oxides
- CuO
- Cu-rich IMP
- etching pit
- 10 seconds

Cr\textsuperscript{0}
SURTEC 650 - TRIVALENT CHROMIUM PASSIVATION
Layer formation - Based on SEM/EDX, XPS, Electrochemical Potential

- 50 seconds
- Cu-rich IMP
- CrO
- Cr\textsuperscript{3+}-Zr\textsuperscript{4+}-mixed oxides
- micro crack
- Zr\textsuperscript{4+} -oxide
- mixed oxides + aluminium fluoride
- etching pit
Layer formation - Based on SEM/EDX, XPS, Electrochemical Potential

- 300 seconds

- Cu-rich IMP

- etching pit

- Zr$^{4+}$-oxide

- Cr$^{3+}$-Zr$^{4+}$-mixed oxides

- zirconium oxide + mixed oxides + aluminium fluoride
SURTEC 650 - TRIVALENT CHROMIUM PASSIVATION
Topography and composition - SEM/EDX

SurTec 650 - Morphology after 300 sec
SURTEC 650 - TRIVALENT CHROMIUM PASSIVATION

Electrochemical Potential - Measured in situ during the Passivation

Reaction time [s]

1. Acid reaction and formation of oxides (strong reaction at the surface)

2. Dense and complete layer (virtually no reaction)

3. Beginning of micro cracks (reaction re-starts)

4. Beginning of laminar macro cracks (constant reaction)

5. 

PASSIVATION SOLUTION

working electrode (aluminium sample)
reference electrode (Ag/AgCl)
counter electrode (Pt)
ADDITIVE SURTEC 650 A - SURFACTANT BLEND

Effect on the Passivation Process

Idea

Add surfactant blend

Less internal stress in the passivation layer

Enable more even layer deposition

Speed up wetting of the surface

Lower surface tension of the solution
ADDITIVE SURTEC 650 A - SURFACTANT BLEND
Effect on Morphology

With additive - Morphology after different immersion times

More uniform layer deposition - Cracks minimized throughout longer immersion
ADDITIVE SURTEC 650 A - SURFACTANT BLEND
Effect on Electrochemical Potential

In case of SurTec 650 + 650 A:
- Cracks minimized
- The curve appears more steady
ADDITIVE SURTEC 650 A - SURFACTANT BLEND

XPS - Element Spectra

Aluminium

Zirconium

Chromium
ADDITIVE SURTEC 650 A - SURFACTANT BLEND

Application

Process parameters for treating EN-AW 2024 T3

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<tr>
<th>SurTec 650 (20 %v/v)</th>
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<tr>
<td>Drying: 60 °C, 20 min</td>
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No Influence of the surfactant on
- Electrical contact resistance of the surface
- Coating weight
- Chemical composition of the layer (SEM-EDX and XRF hand-held)
Effect on Corrosion Protection

Bare corrosion protection after 168 h NSS; EN-AW 2024 T3

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ADDITIVE SURTEC 650 A - SURFACTANT BLEND
Effect on Temperature Resistance

Forced drying at elevated temperatures; 168 h NSS; EN-AW 6061 T6

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Without SurTec 650 A:
- Discoloration and first corrosion pits
ADDITIVE SURTEC 650 A - SURFACTANT BLEND
Analysis and Maintenance

Concentration Control

Surface Tension

Photometry

Test LCK 433 Nonionic Surfactants by company HACH
SurTec 650 A in SurTec 650
Dilution 1:20

\[ \gamma = 0.15x \\
R^2 = 0.9987 \]
ENHANCEMENT BY SURFACE ACTIVE SUBSTANCES

Summary

- Liquid additive directly added to the passivation bath, simple to control and maintain
- The surfactant is NOT deposited on the surface / in the layer
- It does NOT change the composition of the layer
- More uniform passivation layer
- No change in process parameters of currently used bath: pH, temperature, treatment time
- Drying at elevated temperatures is possible
- Larger process window for the treatment time
- Improved process stability and repeatability
- Only one process tank for the conversion coating
Protection upgraded

Thank you.
Cr at first is deposited metallic
ADDITIVE SURTEC 650 A - SURFACTANT BLEND
XPS - Element Spectra

Same composition as ST 650