High Speed Anodizing of Aluminium Alloys 2024 and 2219

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Dirk Hüwelhans
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Content

- High Speed Anodizing - Idea
  Process / Alloys / Deposition rates / Electrolyte

- Characterization of aluminium oxide layers
  Useful Methods for characterization of oxide layers

- Results
  Test results
High Speed Anodizing of 2024 and 2219

Motivation & Challenge

- Motivation
Generate an anodizing process to produce oxide layers >15µm and optimize the layer quality of high copper and silicon alloyed aluminum. Comparison with standard alloy 6082 and 5754.

- Challenge
Development of a «high» speed anodizing process to produce oxide layers for functional application in machining industry, aerospace and injection molding industry.
Cost effective process with a combination of parameters, electrolyte and bath equipment.
High Speed Anodizing of 2024 and 2219 Process

Development of an efficient anodizing process:
- High deposition rate (1-6 µm/min)
- Current density 5-12 A/dm²
- Short process time
- Handling at room temperature
- Avoiding complex process bath composition
- Good cost management

Development of a manufacturing equipment concept
- Anodizing coating unit development
DH Technik - Floating Technology

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High Speed Anodizing of 2024 and 2219

General

Anodizing of medium and high strength material with copper amount > 2.5% and silicon amount >7% is critical in standard anodizing processes.

- Problematic: Intermetallic phases, electrical conductivity, partly overheating
- Trend of material “burning” at anodizing process
- Trend of dissolving during process
High Speed Anodizing of 2024 and 2219 Material

2000 alloy group: Al-Cu

- EN AW 2024 T351 – AlCu4Mg1

<table>
<thead>
<tr>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
<th>Cr</th>
<th>Ni</th>
<th>Zn</th>
<th>Ti</th>
<th>Pb</th>
</tr>
</thead>
<tbody>
<tr>
<td>Max. 0.5%</td>
<td>Max 0.5%</td>
<td>3.8–4.9%</td>
<td>0.3–0.9%</td>
<td>1.2–1.8%</td>
<td>Max. 0.1%</td>
<td>–</td>
<td>Max. 0.25%</td>
<td>Max. 0.15%</td>
<td>–</td>
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</tbody>
</table>

- Medium-High strength material
- Excellent shape stability
- Age-hardened and stress-relieved
- Low corrosion resistance
- High temperature applications
- Field of application: aerospace, machining industry, injection molding
- Technical Anodizing: Poor (limited oxide layer thickness)
2000 alloy group: Al-Cu

- EN AW 2219 Alumold® 350

<table>
<thead>
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<th>Si</th>
<th>Fe</th>
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<th>Mn</th>
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<th>Cr</th>
<th>V</th>
<th>Zn</th>
<th>Ti</th>
<th>Zr</th>
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</thead>
<tbody>
<tr>
<td>Max.</td>
<td>0.2%</td>
<td>Max</td>
<td>5.8–6.8%</td>
<td>0.2–0.4%</td>
<td>Max.</td>
<td>0.02%</td>
<td>0.05–0.15%</td>
<td>Max.</td>
<td>0.02–0.1%</td>
<td>0.1–0.25%</td>
</tr>
<tr>
<td>Max.</td>
<td>0.3%</td>
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- Good machinability
- No weldability
- Poor corrosion resistance
- High strength at high temperatures
- Rm 400-440MPa, A50 7-14% (depend on plate dimension)
- Application area: High dynamic and static stressed parts
- Machining industry, Aerospace

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High Speed Anodizing of 2024 and 2219 Material

Comparison material 6082 and 5754

- **EN AW 6082 (AlMgSi1)**
  - Good machinability, weldability, good anodizing properties

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- **EN AW 5754 (AlMg3)**
  - Good weldability, very good anodizing properties, high corrosion resistance (sea water resistance)

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High Speed Anodizing of 2024 and 2219

Development Project Process Parameter

- **Temperature**
  Temperature range 20-30°C.
  > 28°C powdered layers, burning of edges
- **Current density**
  Depending on alloy and flow of electrolyte
  2219: 8-10A/dm², deposition rate 2-3µm/min
  2024: 9-10A/dm², deposition rate 3-4µm/min
  5754: 9-12A/dm², deposition rate 2-4µm/min
  6082: 9-10A/dm², deposition rate 2-4µm/min
- **Flow of electrolyte**
  Laminar Flow / Rotational Flow / No Flow
- **Electrolyte SA + Oxalic Acid**
  180g/L Sulfuric Acid, 1g/L Oxalic Acid
Content

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- Characterization of aluminium oxide layers
  Useful Methods for characterization of oxide layers

- Results
  Test results
Characterization of Aluminium Oxide Layers

Methods Overview

Quick and easy methods for oxide layer observation.
Useful observation methods for first checks:

- Roughness – Profilometre
- 3D Topography / Roughness by Wyco
- SEM (Cross section and Fracture preparation)
- Corrosion resistance (NSS)
- Abrasion resistance by Taber Test
- Coating Thickness
Roughness measurement by feeler head sensor (profilometre)

Quick and easy measuring of the roughness profile.

Ra = (roughness average) is the arithmetic average of the absolute values of the roughness profile ordinates. Ra is one of the most used values in engineering.

Rt = total high of the roughness profile (measuring area $ln$)

Rz = average surface roughness (average of 5 Rz values achieved in 5 single measurements $lr$)
Characterization of Aluminium Oxide Layers

Method – Roughness Profiles

😊 uniform roughness but very deep crater

😊 uniform roughness,

😊 non uniform roughness but no deep craters or cracks

😊 many craters, not acceptable

😊 good roughness structure but several cracks

😊 no cracks but the non uniform structure
Characterization of Aluminium Oxide Layers
Method – WYKO NT 8000 (Roughness + Surface Topography)

WYKO - White Light Interference Microscopy
2D and 3D characterization of the surface topography by using a non-contact optical profiler.
This technique has been applied to characterize surface topography, surface profile, and surface roughness.

3-Dimensional Interactive Display

Date: 12/01/2015
Time: 10:59:23
Surface Stats:
Ra: 699.82 nm
Rq: 886.40 nm
Rz: 9.05 nm
Measurement Info:
Magnification: 10.08
Measurement Mode: VSI
Sampling: 982.44 nm
Array Size: 640 X 480
Ssk: -0.53
Skw: 3.61

Title: 18611
Note: Eloxal weisser Fleck
Test of abrasion resistance on flat specimen approximately 100mm square or round to a turntable platform that rotates on a vertical axis at a fixed speed. Two abrasive wheels, which are applied at a specific pressure, are lowered onto the specimen surface. Wear action is produced by contact of the test specimen against the sliding rotation of the two abrading wheels.

Commonly used parameters for oxide layers:
- CS-17 wheels
- Load 1000g
- 1000, 5000 and 10000 rotation cycles
Characterization of Aluminium Oxide Layers

Method – SEM (Scanning Electron Microscope)

- Cross section characterisation
  Micro-section preparation by embedding in resign and polishing of the material.
  Used for oxide layer characterization, material structure and failure analysis. Combination with EDX (Energy dispersive X-ray).

- Fractured layer characterisation
  Preparation of fracturing the oxide layer.
  Used for oxide layer characterization – pore structure, layer growth by using higher magnification (useful range 2000x to 100000x).
Characterization of Aluminium Oxide Layers

Method – Break Down Voltage (ISO 2376:2010)

Determination of the electric break down potential.
Characterization of Aluminium Oxide Layers

Method – Neutral Salt Spray Test (NSS) acc. ISO 9227 or ASTM B117

Methode to evaluate the corrosion resistance of materials and surface treatments under salt spray conditions.

Suitable as:

• Corrosion protection tests for rapid analysis
• Analysis of pores and damage in coatings
• Quality control purposes
• Comparison of coatings

Parameters: T=35°C ± 2°C, NaCl solution 50g/L ± 5g/L, pH 6.5-7.2
Running cycles for final oxide layers ≥1000h (depending on application)
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Results
Test results
Test Results – EN AW 2219 – plate top

Roughness by WYKO

Title: 19230
Note: 2219 - 10D Vorderseite
Test Results – EN AW 2219 - plate top
Topography WYKO

3-Dimensional Interactive Display

Date: 10/09/2018
Time: 14:19:23
Surface Stats:
Ra: 621.97 nm
Rq: 779.79 nm
Rt: 6.80 um

Measurement Info:
Magnification: 5.39
Measurement Mode: VSI
Sampling: 1.34 um
Array Size: 640 X 480
Ssk: -0.20
Sku: 3.01

Title: 19230
Note: 2219 - 10D Vorderseite
Test Results – EN AW 2024 – plate centre

Roughness by WYKO

X Profile

Y Profile

Title: 19232
Note: 2024 - 17D Rückseite
Test Results – EN AW 2024 - plate centre
Topography WYKO

3-Dimensional Interactive Display

Date: 10/09/2018
Time: 14:51:54
Surface Stats:
Ra: 1.39 um
Rq: 1.77 um
Rt: 13.62 um

Measurement Info:
Magnification: 5.39
Measurement Mode: VSI
Sampling: 1.84 um
Array Size: 640 x 480
Ssk: -0.56
Sku: 3.62

Title: 19232
Note: 2024 - 17D Rückseite

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## Test Results

### Break down voltage / Coating thickness

<table>
<thead>
<tr>
<th>Alloy</th>
<th>EN AW 5754</th>
<th>EN AW 6082</th>
<th>EN AW 2219</th>
<th>EN AW 2024</th>
<th>EN AW 2024</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness (µm)</td>
<td>20.9</td>
<td>32.4</td>
<td>22.4</td>
<td>36</td>
<td>50.1</td>
</tr>
<tr>
<td>BDV – DC (kV)</td>
<td>1.15</td>
<td>1.02</td>
<td>0.55</td>
<td>1.03</td>
<td>2.2</td>
</tr>
<tr>
<td>Ramp 0.1kV/s</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>BDV – DC (kV)</td>
<td>1.10</td>
<td>1.0</td>
<td>0.52</td>
<td>1.05</td>
<td>2.18</td>
</tr>
<tr>
<td>Ramp 0.2kV/s</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Values for Break Down Voltage (BDV) and Coating Thickness: Average of 5 measurements.

Break Down voltage, Ramp 0.1kV/s and 0.2kV/s, current 0.5mA

Coating Thickness according DIN EN ISO 2360 (Eddy current method)

All samples sealed after anodizing (hot water sealing)
# Test Results

**NSS corrosion test according ISO 9227**

<table>
<thead>
<tr>
<th>Alloy</th>
<th>EN AW 5754 (µm)</th>
<th>EN AW 5754</th>
<th>EN AW 6082</th>
<th>EN AW 2024</th>
<th>EN AW 2219</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickness</td>
<td>26.3</td>
<td>33</td>
<td>25.9</td>
<td>35.3</td>
<td>19.1</td>
</tr>
<tr>
<td>After 500h</td>
<td>no corrosion</td>
<td>no corrosion</td>
<td>no corrosion</td>
<td>no corrosion</td>
<td>first pitting</td>
</tr>
</tbody>
</table>

Values for coating thickness: Average of 5 measurements. Coating Thickness according DIN EN ISO 2360 (Eddy current method) Corrosion test in progress, first results after 500 hours NSS. All samples sealed after anodizing (hot water sealing)
Test Results EN AW 2024

SEM observation - cross section

Uniform oxide layer
Coating Thickness: 40-45µm
Test Results EN AW 2219

SEM observation – cross section

Dissolution of intermetallic phases in oxide layer.

Uniform oxide layer
Coating Thickness: 40-45µm
3 kinds of interaction of intermetallic phases in oxide layers
Integration, Oxidation, Dissolution of intermetallic phases

800x (Alloy EN AW 6061, plate)
Test Results EN AW 5754
SEM observation – fractured cross section

2000x

20000x
Test Results EN AW 5754

SEM observation – fractured cross section

200 nm  EHT = 5.00 kV  WD = 5.2 mm  Signal A = InLens  REM18_8739

50000x
Test Results EN AW 2024

SEM observation – fractured cross section

2000x

20000x
Test Results EN AW 2024

SEM observation – fractured cross section

100 nm

EHT = 5.00 kV
WD = 4.1 mm
Signal A = InLens
REM18_8713

50000x
Test Results EN AW 2219

SEM observation – fractured cross section

2000x

20000x
Test Results EN AW 2219

SEM observation – fractured cross section
Summary

- High deposition rates of 2-4µm/min (2219, 2024, 5754, 6082) possible
- Intermetallic phases and material structure significant influence of layer properties and oxide structure
- Break Down Voltage of 2024 comparable to 6082
- Roughness in a good range
- DH Technik floating system inside the anodizing bath essential
- First corrosion results looks promising
- Standard bath equipment useful with adaption of DH Technik - Floating System
- Suitable aluminium coatings for machining and moulding industry, aerospace and semiconductor industry

Promising Process
Anodizing Process – Future Prospects

ANODIZING
THANK YOU

- Material support by Constellium Sierre, Switzerland
- WYKO, NSS, SEM and Taber Test done by Suisse Technology Partners, Neuhausen Rheinfall, Switzerland, www.suisse-tp.ch
- Break down voltage measurement done by FME GmbH Fuchs Materials & Engineering, Neuhausen Rheinfall, Switzerland, www.fme-gmbh.ch

And a big thank you for your attention!

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