Best Plating Practices for the Application of Brush Plated Zinc-Nickel

Sarah Medeiros
Corrosion Engineer
Who We Are

• SIFCO Applied Surface Concepts
  • Founded in 1959
  • Headquartered in Cleveland, Ohio, USA
  • 4 USA, 3 European locations
    • USA: Texas, Virginia, Connecticut, Ohio
    • Europe: UK, France, Sweden
  • Acquired as part of the Surface Coatings Division of Norman Hay in 2012

• Norman Hay Group
  • Founded in 1940, doing chromium plating and hard anodizing
  • Headquartered in Coventry, UK
  • Ultraseal International, Surface Technology, and NHE
Application of Brush Plated Zinc-Nickel Agenda

• What is Selective Plating (aka Brush Plating)?
• Specifications (ASTM F519, ASTM B117, Boeing 5664, ASM 2451)
• Hydrogen Embrittlement
  • Bar preparation
  • Testing results
• ASTM B117 Corrosion Testing
  • Boeing Specification 5664
  • Dip Plating
  • Flow Plating
What is Selective Plating?

• The SIFCO Process® is a portable method of electroplating localized areas without the use of an immersion tank.

Key Requirements:
1. Workpiece
2. Power Pack
3. Plating Tools
4. Solution

Other:
• Solution Flow System
• Masking Materials
• Auxiliary Equipment
Selective Plating/Brush Plating

Surface Preparation
- Pre-Clean
- Electroclean
- Etch
- Desmutting
- Activation
- Preplate
- Plate
ASTM F519

- Purpose: Describes mechanical test methods and defines acceptance criteria for plating processes that can cause hydrogen embrittlement in steels
ASTM F519 cont.

- Procedure requirements - Sustained Load Test (SLT)
  - Minimum of four specimens per test
  - For 1a.1 and 1a.2 bars, the entire notch and 0.5 inch on either side must be plated
  - Sustained load specimens shall be maintained for 200 hours minimum at 75% of the tensile notched fracture strength (NFS)

- Results Interpretation
  - No fracture: Plating process is non-embrittling
  - One of four fracture: Remaining three specimens may be step loaded every 2 hours in 5% increments to 90% of the NFS after completing the 200 hour sustained load. Considered non-embrittling if specimens survive 2 hours at 90% NFS.
  - Two or more fracture: Plating process is considered embrittling
ASTM B117

• Purpose: Describes the apparatus, procedure and conditions to create and maintain salt spray test environment

• Apparatus: fog chamber, salt solution reservoir, compressed air, atomizing nozzle, specimen supports, chamber heating, means of control

• Position of test specimens: 15-30° from vertical, no metal contact, unencumbered exposure to fog

• Salt solution: 5% sodium chloride in Type IV water
  • Atomized at 35 °C ± 2 °C, pH range from 6.5 to 7.2
  • Minimum of 2 clean fog collectors in the chamber, 1.0-2.0mL per hr collected

• Compressed air supply must be clean from oil, dirt and grease

• Continuous testing, except to check once daily
Boeing 5664

- **Purpose:** Requirements for materials and procedures for deposition of ZnNi plating on low-alloy steel using the stylus electroplating process

- **Anode**
  - Graphite of high purity and density
  - Platinum, platinum-iridium, platinum plated titanium, platinum plated niobium
  - Cotton batting and cotton or dacron sleeving

- **Adjustable powerpack with adjustable DC voltage that displays voltage, amperage and ampere-hour**

- **Maximum area of ZnNi stylus plating is 5 square inches**

- **Minimum thickness of ZnNi stylus plating is 0.0005 inches thick**

- **ZnNi is not pre-wetted and may be applied with squeeze bottle or dip method**
AMS 2451

• Purpose: Requirements for brush plating ZnNi LHE by electrodeposition

• Application: Improve corrosion resistance of steel parts under 500 °F as-plated or under 250 °F when chromate treated, to repair ZnNi deposits and damaged or worn parts

• Classification
  • Type 1: As-plated
  • Type 2: As-plated with supplementary surface treatment via brush, spray or dip
    - Grade A: Hexavalent chromate treatment
    - Grade B: Trivalent chromium treatment

• ZnNi deposit must contain 6-20% Ni, balance Zn
<table>
<thead>
<tr>
<th></th>
<th>Boeing 5664</th>
<th>AMS 2451</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface Preparation</td>
<td>Solvent cleaned, dry abraded</td>
<td></td>
</tr>
<tr>
<td>Chromate Conversion Coating</td>
<td>Yes</td>
<td>Type 1 vs Type 2</td>
</tr>
<tr>
<td>Anode</td>
<td>Stylus</td>
<td></td>
</tr>
<tr>
<td>Voltage</td>
<td>6V</td>
<td></td>
</tr>
<tr>
<td>Hydrogen Embrittlement</td>
<td>F519</td>
<td>F519</td>
</tr>
<tr>
<td></td>
<td>1a.2 notched bars</td>
<td>1a.1 or 1a.2 notched bars</td>
</tr>
<tr>
<td></td>
<td>0.0005-0.0008 inches</td>
<td>0.0003-0.0006 inches</td>
</tr>
<tr>
<td>Corrosion</td>
<td>B117</td>
<td>B117</td>
</tr>
<tr>
<td></td>
<td>500 hours ±1 hour</td>
<td></td>
</tr>
<tr>
<td></td>
<td>0.0005-0.0008 inches</td>
<td>0.0003-0.0007 inches</td>
</tr>
<tr>
<td></td>
<td>No white or red corrosion</td>
<td>No white corrosion products after 96hrs</td>
</tr>
<tr>
<td></td>
<td>products after 96hrs</td>
<td>No red corrosion after 500hrs</td>
</tr>
</tbody>
</table>
Hydrogen Embrittlement Preparatory Steps

1. Clean and degrease notch bars with acetone

2. Mask bar threads and radial areas with tape and sandblast with 120 grit silicon carbide

3. Remove masking, rinse with DI water, and clean with acetone using lint free wipe

4. Mask bar threads and radial areas with tape, thread notch bar into barrel fixture and insert into turning head

5. Set RPM to 60-65

6. Plate ZnNi LHE with no pre-wet, rinse with DI water

7. Immerse in trivalent chromium conversion coating for 90 seconds, rinse with DI water and dry with compressed air, No Bake Required
Hydrogen Embrittlement Goals

- Goal: Pass hydrogen embrittlement testing within 24 hours on 1a.1 and 1a.2 bars with no bake

- Variables
  - 6asi vs 3asi vs 6V
  - Conforming (50% contact) anode vs stylus (25% contact) anode
  - Graphite anode vs DSA anode

- Phase 1 Goal: Pass HE testing in under 72 hours at 3asi and 6asi
- Phase 2 Goal: Pass HE testing in under 48 and 24 hours at 6V (Boeing 5664) and 6asi
- Phase 3 Goal: Pass HE testing in under 24 hours at 6asi
HE Phase 1 Results

• Compare:
  • 72 hours to 1 week
  • 3asi to 6asi
  • 1a.1 to 1a.2
  • Full and partial length plating of 1a.2

<table>
<thead>
<tr>
<th></th>
<th>1a.1</th>
<th>1a.1</th>
<th>1a.1</th>
<th>1a.1</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bar Type</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CD</td>
<td>3 asi</td>
<td>3 asi</td>
<td>6 asi</td>
<td>6 asi</td>
</tr>
<tr>
<td>Plate to Test Time</td>
<td>&lt;72 hrs</td>
<td>~1 week</td>
<td>&lt;72 hrs</td>
<td>~1 week</td>
</tr>
<tr>
<td>HE ASTM F519</td>
<td>PASS</td>
<td>PASS</td>
<td>PASS</td>
<td>PASS</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th></th>
<th>1a.2</th>
<th>1a.2</th>
<th>1a.2</th>
<th>1a.2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bar Type</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>CD</td>
<td>6 asi</td>
<td>6 asi</td>
<td>6 asi</td>
<td>6 asi</td>
</tr>
<tr>
<td>Masking</td>
<td>Plated Full Bar</td>
<td>Plated Full Bar</td>
<td>Plated 1/2 inch on either side of notch</td>
<td>Plated 1/2 inch on either side of notch</td>
</tr>
<tr>
<td>Plate to Test Time</td>
<td>&lt;72 hrs</td>
<td>~1 week</td>
<td>&lt;72 hrs</td>
<td>~1 week</td>
</tr>
<tr>
<td>HE ASTM F519</td>
<td>PASS</td>
<td>PASS</td>
<td>PASS</td>
<td>PASS</td>
</tr>
</tbody>
</table>

• All specimens passed in Phase 1
HE Phase 2 Results

- Compare:
  - 48 hours to 24 hours
  - 6V to 6asi
  - 1a.1 to 1a.2
  - Conforming to stylus anode

<table>
<thead>
<tr>
<th>Bar Type</th>
<th>Constant Voltage or Constant Current</th>
<th>Plate to Test Time</th>
<th>Anode Type</th>
<th>HE ASTM 519</th>
</tr>
</thead>
<tbody>
<tr>
<td>1a.1</td>
<td>6V</td>
<td>48 hrs</td>
<td>Conforming</td>
<td>Fail (3 Failures)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>(14E -1, 2, 3 &amp; 4)</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>@ 1.6, 1.9 &amp; 5.3 hrs</td>
</tr>
</tbody>
</table>

** Yellow highlight = PASS

- 3 out of 9 groups passed in Phase 2
  - 1 sat for 96 hours (non-conforming 6V)
  - 1 plated at higher current density (10-12 asi) (Non-conforming 6asi)
  - 1 plated as planned (Conforming 6asi)
HE Phase 3 Results

• Compare:
  • Conforming to stylus anode
  • DSA to Graphite anode

• Additional variables
  - Nickel neutral strike
  - High voltage strike
  - Heated ZnNi solution

• 5 out of 8 groups passed in Phase 3
  • Conforming DSA and Graphite 1a.1 bars
  • Nickel Neutral strike, 20V ZnNi strike and heated solution

Yellow highlight = PASS
Corrosion Testing Goals

- Pass AMS 2451
  - Type 1: No red corrosion at 500hrs
  - Type 2: No white corrosion at 96hrs and no red corrosion at 500hrs
- Follow B117
- Salt Spray testing parameters:
  - Follow Boeing 5664
  - Cotton Wrap vs White Tuff Wrap vs Red Tuff Wrap
  - Dip plating vs flow plating
  - No conversion coating vs trivalent vs hexavalent
  - Dip vs squirt conversion coating application
  - 6asi vs 6V
  - 0.00065 inches thick
Salt Spray Comparing Wet and Dry Pictures

- All salt spray pictures to be shown have been rinsed with DI, dried then photographed
- More accurate and severe pictures
- Ex: ZnNi at 500hrs wet and dry
Round 1: Salt Spray Boeing 5664 Testing

- Stylus, graphite anode
- 6V throughout plating
- Trivalent conversion coating
- Dip vs squirt conversion coating application
- Minimum thickness is 0.0005 inches
### Salt Spray: Boeing Spec. – 6V Dip Tri. Cr

<table>
<thead>
<tr>
<th>Time</th>
<th>Appearance</th>
<th>Composition</th>
<th>Plating Parameters</th>
<th>Test Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>0hrs</td>
<td>Clean</td>
<td>8.7% Ni, bal Zn</td>
<td>Room Temperature</td>
<td>Salt Spray ASTM B117</td>
</tr>
<tr>
<td>96hrs</td>
<td>Darkening</td>
<td></td>
<td>Current Density: 6 V</td>
<td></td>
</tr>
<tr>
<td>250hrs</td>
<td>Further darkening</td>
<td></td>
<td>Target Thickness: 0.00065 inches</td>
<td></td>
</tr>
<tr>
<td>500hrs</td>
<td>Rusting</td>
<td></td>
<td>*Conversion Coating applied via dip for 90 sec followed by rinse and air dry.</td>
<td></td>
</tr>
<tr>
<td>1000hrs</td>
<td>Erosion</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Salt Spray: Boeing Spec. – 6V Squirt Tri. Cr

<table>
<thead>
<tr>
<th>0hrs</th>
<th>96hrs</th>
<th>250hrs</th>
<th>500hrs</th>
<th>1000hrs</th>
</tr>
</thead>
</table>

**Plating Parameters**
- Room Temperature
- Current Density: 6 V
- Target Thickness: 0.00065 inches
- Composition: 8.9% Ni, bal Zn
- Salt Spray ASTM B117

*Conversion Coating applied via squeeze bottle for 90 sec followed by rinse and air dry.*
Round 1: ZnNi Boeing 5664 Salt Spray Testing

• Dipped trivalent conversion coating produced slightly better results compared to squirted trivalent conversion coating

• All samples passed AMS 2451 – Type 2
  • Slight blushing but no white corrosion at 96 hours
  • No red corrosion at 500 hours
Round 2: Dip Plating Testing

- Cotton Wrap
- 6asi
- No conversion coating vs trivalent vs hexavalent
- Squirt conversion coating application
Salt Spray: No Conversion Coating

<table>
<thead>
<tr>
<th>0hrs</th>
<th>96hrs</th>
<th>500hrs</th>
<th>1000hrs</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
<td><img src="image3.png" alt="Image" /></td>
<td><img src="image4.png" alt="Image" /></td>
</tr>
</tbody>
</table>

Composition: ~11.4% Ni, bal Zn

**Plating Parameters**
- Room Temperature
- Current Density: 6 ASI
- Target Thickness: 0.00065 inches

Salt Spray ASTM B117
Salt Spray: Trivalent Cr Conversion Coating

<table>
<thead>
<tr>
<th>0hrs</th>
<th>96hrs</th>
<th>500hrs</th>
<th>1000hrs</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="image1.png" alt="Image 1" /></td>
<td><img src="image2.png" alt="Image 2" /></td>
<td><img src="image3.png" alt="Image 3" /></td>
<td><img src="image4.png" alt="Image 4" /></td>
</tr>
</tbody>
</table>

**Plating Parameters**
- Room Temperature
- Current Density: 6 ASI
- Target Thickness: 0.00065 inches
- *Conversion Coating applied via squeeze bottle for 90 sec followed by rinse and air dry.*

Composition: ~11.9% Ni, bal Zn

Salt Spray ASTM B117
Salt Spray: Hexavalent Cr Conversion Coating

<table>
<thead>
<tr>
<th>Time (hrs)</th>
<th>0hrs</th>
<th>96hrs</th>
<th>500hrs</th>
<th>1000hrs</th>
</tr>
</thead>
<tbody>
<tr>
<td>[Image]</td>
<td>[Image]</td>
<td>[Image]</td>
<td>[Image]</td>
<td>[Image]</td>
</tr>
</tbody>
</table>

**Composition:** ~11.8% Ni, bal Zn

**Plating Parameters**
- Room Temperature
- Current Density: 6 ASI
- Target Thickness: 0.00065 inches
- *Conversion Coating heated to 95 - 100 °F, applied via squeeze bottle for 90 sec followed by rinse and air dry.*

Salt Spray ASTM B117
Round 2: ZnNi Dip Salt Spray Testing

- 6asi has higher Ni content than 6V
  - 11.5% versus 8.8%
- Both conversion coatings pass AMS 2451
  - No white corrosion for 96hrs, no red corrosion at 500hrs
- Without conversion coating, failed AMS 2451
  - Red corrosion visible between 100-230hrs
Round 3: Flow Plating Testing

- Cotton Wrap vs White Tuff Wrap vs Red Tuff Wrap
- 6V vs 6asi
- Trivalent conversion coating
- Squirt conversion coating application
## Salt Spray: Cotton Wrap with Tri. Cr – 6V

<table>
<thead>
<tr>
<th>Time</th>
<th>0hrs</th>
<th>96hrs</th>
<th>285hrs</th>
<th>500hrs</th>
<th>1000hrs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Image</td>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
<td><img src="image3.png" alt="Image" /></td>
<td><img src="image4.png" alt="Image" /></td>
<td><img src="image5.png" alt="Image" /></td>
</tr>
</tbody>
</table>

**Composition:** 8.8% Ni, bal Zn

**Plating Parameters**
- Room Temperature
- Current Density: 6 V
- Target Thickness: 0.00065 inches
*Conversion Coating applied via squeeze bottle for 90 sec followed by rinse and air dry.

Salt Spray ASTM B117
Salt Spray: Cotton Wrap Tri. Cr – 6ASI

<table>
<thead>
<tr>
<th>0hrs</th>
<th>96hrs</th>
<th>285hrs</th>
<th>500hrs</th>
<th>1000hrs</th>
</tr>
</thead>
</table>

Plating Parameters
- Room Temperature
- Current Density: 6 ASI
- Target Thickness: 0.00065 inches
- *Conversion Coating applied via squeeze bottle for 90 sec followed by rinse and air dry.

Composition: 13.6% Ni, bal Zn

Salt Spray ASTM B117
Salt Spray: White Tuff Wrap Tri. Cr – 6V

Plating Parameters
- Room Temperature
- Current Density: 6 V
- Target Thickness: 0.00065 inches
*Conversion Coating applied via squeeze bottle for 90 sec followed by rinse and air dry.

Composition: 8.7% Ni, bal Zn

Salt Spray ASTM B117
Salt Spray: White Tuff Wrap Tri. Cr – 6ASI

<table>
<thead>
<tr>
<th>0hrs</th>
<th>96hrs</th>
<th>285hrs</th>
<th>500hrs</th>
<th>1000hrs</th>
</tr>
</thead>
<tbody>
<tr>
<td><img src="wtw6asie3007.png" alt="Image" /></td>
<td><img src="wtw6asie3007.png" alt="Image" /></td>
<td><img src="wtw6asie3007.png" alt="Image" /></td>
<td><img src="wtw6asie3007.png" alt="Image" /></td>
<td><img src="wtw6asie3007.png" alt="Image" /></td>
</tr>
</tbody>
</table>

**Plating Parameters**
- Room Temperature
- Current Density: 6 ASI
- Target Thickness: 0.00065 inches
- *Conversion Coating applied via squeeze bottle for 90 sec followed by rinse and air dry.*
- Salt Spray ASTM B117

Composition: 15.2% Ni, bal Zn
Salt Spray: Red Tuff Wrap Tri. Cr – 6V

<table>
<thead>
<tr>
<th>Time</th>
<th>Image 1</th>
<th>Image 2</th>
<th>Image 3</th>
<th>Image 4</th>
<th>Image 5</th>
</tr>
</thead>
<tbody>
<tr>
<td>0hrs</td>
<td>![Image]</td>
<td>![Image]</td>
<td>![Image]</td>
<td>![Image]</td>
<td>![Image]</td>
</tr>
<tr>
<td>96hrs</td>
<td>![Image]</td>
<td>![Image]</td>
<td>![Image]</td>
<td>![Image]</td>
<td>![Image]</td>
</tr>
<tr>
<td>500hrs</td>
<td>![Image]</td>
<td>![Image]</td>
<td>![Image]</td>
<td>![Image]</td>
<td>![Image]</td>
</tr>
<tr>
<td>1000hrs</td>
<td>![Image]</td>
<td>![Image]</td>
<td>![Image]</td>
<td>![Image]</td>
<td>![Image]</td>
</tr>
</tbody>
</table>

**Plating Parameters**
- Room Temperature
- Current Density: 6 V
- Target Thickness: 0.00065 inches
- Conversion Coating applied via squeeze bottle for 90 sec followed by rinse and air dry.

Composition: 10.5% Ni, bal Zn

Salt Spray ASTM B117
Salt Spray: Red Tuff Wrap Tri. Cr – 6ASI

<table>
<thead>
<tr>
<th>Time</th>
<th>0hrs</th>
<th>96hrs</th>
<th>285hrs</th>
<th>500hrs</th>
<th>1000hrs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Image</td>
<td><img src="image1" alt="Image" /></td>
<td><img src="image2" alt="Image" /></td>
<td><img src="image3" alt="Image" /></td>
<td><img src="image4" alt="Image" /></td>
<td><img src="image5" alt="Image" /></td>
</tr>
</tbody>
</table>

**Plating Parameters**

- Room Temperature
- Current Density: 6 ASI
- Target Thickness: 0.00065 inches
- Composition: 14.8% Ni, bal Zn
- Salt Spray ASTM B117
- *Conversion Coating applied via squeeze bottle for 90 sec followed by rinse and air dry.*
Round 3: ZnNi Flow Salt Spray Testing

- Panels plated at 6asi performed better than 6V
- Cotton wrap, white tuff wrap and red tuff wrap all performed better than traditional dip plating at 250, 500 and 1000hrs
Best Results

• **Hydrogen Embrittlement**
  - 1a.1 bars within 24hrs
    - Conforming anode (graphite and DSA)
    - Nickel Neutral strike
    - High voltage strike
    - Heated solution

• **Corrosion**
  - Flow plating at 6asi
    - Cotton and white tuff wrap

<table>
<thead>
<tr>
<th></th>
<th>Cotton</th>
<th>White Tuff Wrap</th>
</tr>
</thead>
<tbody>
<tr>
<td>96hrs</td>
<td>500hrs</td>
<td>96hrs</td>
</tr>
<tr>
<td></td>
<td></td>
<td>500hrs</td>
</tr>
</tbody>
</table>

Cotton Flow 6asi 3007
Cotton Flow 6asi 3007
WTW 6asi 3007
WTW 6asi 3007
Future Work

• **Hydrogen Embrittlement**
  - Focus on 1a.2 bars passing in 24 hours
  - Passes at 72 hours and later
  - Attempt variables that worked on 1a.1 bars for 1a.2 bars
    - Nickel Neutral Pre-plate
    - 20V ZnNi strike
    - Heated ZnNi solution

• **Corrosion**
  - Explore alternative trivalent chromium conversion coatings
    - Improved corrosion performance
  - Expand work on flow plating via depletion testing
    - Determine lifetime of solution
    - Determine how well ZnNi solution works over the lifetime
Acknowledgements

Danijela Milosevic-Popvich, R&D Manager
Teri Zarnesky, Lab Technician
Derek Vanek, Technical Manager
Derek Kilgore, Design and Project Engineer
Ken Mabe, Solution Manufacturing Technician
Mike Torma, Solution Manufacturing Technician
QUESTIONS?

Sarah Medeiros
smedeiros@sifcoasc.com
booth #330

US Headquarters
SIFCO ASC
5708 E. Schaaf Rd
Independence, Ohio
1-216-524-0099
info@sifcoasc.com