Low Hydrogen Embrittlement (LHE) Zinc-Nickel (Zn-Ni) Qualification Test Result and Process Parameters Development

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**Report Title:** Low Hydrogen Embrittlement (LHE) Zinc-Nickel (Zn-Ni) Qualification Test Result and Process Parameters Development

**Performing Organization:** ES3 Inc, 1346 South Legend Hills Drive, Clearfield, UT, 84015

**Abstract:**
Focused Workshop on Cadmium Plating Alternatives, August 30-31, 2011, Baltimore, MD. Sponsored by SERDP/ESTCP.
LHE Zn-Ni Background

- Boeing C-17 Program cadmium replacement research (2002-2006)
- Small Business Innovative Research (SBIR) Phase I Feasibility Study (2007-2008)
- SBIR Phase II Qualification Testing (2008-2010):
A Technical Interchange Meeting (TIM) was conducted at ASC/EN on 26 May 2011 with participants from: OOA-ALC/GH, ES3, AFRL/RX, Boeing, and ASC/EN. Following AFRL concerns were discussed about the Zn-Ni Phase II test report:

- Scribe Tests (Corrosion Tests)
- Hydrogen ‘Re-’Embrittlement Tests
- Fatigue Tests

These were the only items in which action items were opened.
Corrosion (Scribe) Testing

- **Scribe Test Action Item #1:**
  - Provide 1000 hrs panel scribe data to AFRL for evaluation.

- **Response:**
  - Original 1000 hrs panels are not available - they were tested to 5000 hrs
  - Consequently, Boeing Research & Technology (BR&T) evaluated standard carbide scribe technique and the team determined that the process is repeatable and exposes a sufficient amount of base metal to provide valid test results
  - Also, BR&T determine that the original corrosion panels were machined scribed and not carbide scribed by hand (i.e. machined scribed the preferred AFRL scribing method)
  - BR&T hand scribed panels and ran SEM EDX scans to determine IF they could reach the bare metal substrate; see following slides
Corrosion (Scribe) Testing

IVD Aluminum

Specimen: IVD Aluminum
Cadmium

Specimen: Cadmium
Corrosion (Scribe) Testing

Zn-Ni

Specimen: Zn-Ni, 4-10F, 0.9 ± 0.1
Corrosion (Scribe) Testing

• Scribe Test Action Item #2:
  ▪ Boeing to machine scribe Zn-Ni and Cd panels and test them for 1000 hours for a direct comparison

• Response:
  ▪ Additional machined scribed Zn-Ni and Cd panels have been corrosion tested by BR&T per ASTM B 117
  ▪ All the Zn-Ni plated panels passed the corrosion requirements called out in QQ-P-416 (no white corrosion products for 96 hours)
    • Results are shown in following slides below
## Table 1 - Machine vs. Carbide Scribed Corrosion Test

<table>
<thead>
<tr>
<th>Group No.</th>
<th>Test Specimen Identification</th>
<th>Plating Material</th>
<th>Conversion Coat Type</th>
<th>Plating Thickness (mils)</th>
<th>Primer + Topcoat</th>
<th>Type of Scribe</th>
<th>Test Duration**</th>
<th>Test Results</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>BC1  Zn-Ni</td>
<td>TrCr  0.6 +/- 0.15</td>
<td>Yes</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>BC2  Zn-Ni</td>
<td>TrCr  0.7 +/- 0.1</td>
<td>Yes</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>BC3  Zn-Ni</td>
<td>TrCr  0.7 +/- 0.1</td>
<td>Yes</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>HC1  Zn-Ni</td>
<td>TrCr  0.8 +/- 0.2</td>
<td>Yes</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>HC2  Zn-Ni</td>
<td>TrCr  0.8 +/- 0.1</td>
<td>Yes</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>HC3  Zn-Ni</td>
<td>TrCr  0.8 +/- 0.2</td>
<td>Yes</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>HC4  Cd</td>
<td>HexCr 0.8 +/- 0.05</td>
<td>Yes</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>HC5  Cd</td>
<td>HexCr 0.7 +/- 0.1</td>
<td>Yes</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>HC6  Cd</td>
<td>HexCr 0.5 +/- 0.1</td>
<td>Yes</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>BS1  Zn-Ni</td>
<td>TrCr  0.8 +/- 0.05</td>
<td>No</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>BS2  Zn-Ni</td>
<td>TrCr  0.7 +/- 0.05</td>
<td>No</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>BS3  Zn-Ni</td>
<td>TrCr  0.8 +/- 0.05</td>
<td>No</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>HS1  Zn-Ni</td>
<td>TrCr  0.8 +/- 0.1</td>
<td>No</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>HS2  Zn-Ni</td>
<td>TrCr  0.8 +/- 0.05</td>
<td>No</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>HS3  Zn-Ni</td>
<td>TrCr  0.8 +/- 0.1</td>
<td>No</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>PASS</td>
<td></td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>HS4  Cd</td>
<td>HexCr 0.8 +/- 0.1</td>
<td>No</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>FAIL</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>HS5  Cd</td>
<td>HexCr 0.7 +/- 0.1</td>
<td>No</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>FAIL</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>HS6  Cd</td>
<td>HexCr 0.8 +/- 0.1</td>
<td>No</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>FAIL</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>BS4  Zn-Ni</td>
<td>None  0.8 +/- 0.1</td>
<td>No</td>
<td>Machined Scribe</td>
<td>1000 hrs</td>
<td>FAIL ****</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>BN1  Zn-Ni</td>
<td>None  0.7 +/- 0.1</td>
<td>No</td>
<td>No Scribe</td>
<td>1000 hrs</td>
<td>PASS ****</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**** Group 7 test coupons were run without conversion coating and were not required to pass (i.e. information only)

BR&T ASTM B 117 Corrosion Test Results
BR&T IZ-C17+ Zn-Ni w/Tri CC
Scribed & Painted

336 hours

672 hours

1000 hours
Hill AFB IZ-C17+ Zn-Ni w/Tri CC
Scribed & Painted

336 hours
672 hours
1000 hours
Hill AFB LHE Cd w/Hex CC
Scribed & Painted

336 hours

672 hours

1000 hours
BR&T IZ-C17+ Zn-Ni w/Tri CC Scribed

336 hours

672 hours

1000 hours
Hill AFB IZ-C17+ Zn-Ni w/Tri CC Scribed

336 hours

672 hours

1000 hours
Hill AFB LHE Cd w/Hex CC Scribed

336 hours

672 hours

1000 hours

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Requirements for Chrome Reduction / Elimination; determine how the plating works without ‘any’ conv. coat.

Group 7 test coupons were run without conversion coating and were not required to pass (i.e. information only)
• ‘Re-’ Embrittlement Test Action Item #1:
  ▪ Determine the reason for the poor plating on the original LHE Zn-Ni 1a.1 re-embrittlement coupons

• Response: See following slides
The reason for the poor plating on the original LHE Zn-Ni 1a.1 re-embrittlement coupons are as follows:

- Zn-Ni tank contamination
  - Spring ‘09 Lab analysis showed organic contamination
  - The PVC tank liner had begun to break down and had to be replaced in the Summer ‘09 with a more robust grade of PVC liner.
- Two years operating with new liner with no problems
- Inconsistent plating in notch area
  - Specimens were chained in series when they were plated for the first series of tests
  - Now a fixture and conformal anode is used to ensure that there is uniform plating throughout the notch area
  - Also circulation has been added around the notch area during plating
Hydrogen ‘Re-’Embrittlement For Service Environments Testing

Original Coupons Chained in Series

New fixture and Conformal Anode
• Additional, 3.5% salt water, re-embrittlenent testing has been conducted on LHE Zn-Ni plated coupons and they all passed the ASTM 519-06 150 hour requirement.

<table>
<thead>
<tr>
<th>Plating</th>
<th>Test Solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Distilled Water @ Room Temp</td>
<td>45% NFS for 150Hrs</td>
</tr>
<tr>
<td>3.5% Salt Water @ Room Temp</td>
<td>45% NFS for 150Hrs</td>
</tr>
<tr>
<td>Dwg 9825019* Diluted Calla 296 @ Max Temp 180 °F Tested 75% NFS for 200Hrs</td>
<td></td>
</tr>
<tr>
<td>Dwg 9825019* Diluted Calla 602 LF Max Temp 160 °F Tested 75% NFS for 200Hrs</td>
<td></td>
</tr>
<tr>
<td>Concentrated Calla 296 @ Room Temp tested 45% NFS for 150Hrs</td>
<td></td>
</tr>
<tr>
<td>Concentrated Calla 602LF @ Room Temp tested 45% NFS for 150Hrs</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Plating</th>
<th>Distilled Water @ Room Temp</th>
<th>3.5% Salt Water @ Room Temp</th>
<th>Dwg 9825019* Diluted Calla 296 @ Max Temp 180 °F Tested 75% NFS for 200Hrs</th>
<th>Dwg 9825019* Diluted Calla 602 LF Max Temp 160 °F Tested 75% NFS for 200Hrs</th>
<th>Concentrated Calla 296 @ Room Temp tested 45% NFS for 150Hrs</th>
<th>Concentrated Calla 602LF @ Room Temp tested 45% NFS for 150Hrs</th>
</tr>
</thead>
<tbody>
<tr>
<td>LHE Zn-Ni</td>
<td>Passed</td>
<td>Passed</td>
<td>Passed</td>
<td>Passed</td>
<td>Passed</td>
<td>Passed</td>
</tr>
<tr>
<td>Cadmium</td>
<td>Passed</td>
<td>Failed</td>
<td>Passed</td>
<td>Passed</td>
<td>Passed</td>
<td>Passed</td>
</tr>
<tr>
<td>IVD</td>
<td>Failed</td>
<td>Failed</td>
<td>Not Tested</td>
<td>Not Tested</td>
<td>Not Tested</td>
<td>Not Tested</td>
</tr>
</tbody>
</table>

*The specimens were immersed in the cleaning compound at the manufacturer’s maximum recommended temperature, and appropriate cleaning concentration, for 30 minutes. Removed. Air dried and loaded to 75% NFS for 200Hrs.
• ‘Re-’Embrittlement Test Action Item #2:
  ▪ If possible, repeat the Boeing voltage potential test on plating of the original LHE Zn-Ni 1a.1 re-embrittlement coupons and compare the voltage readings to current LHE Cd and LHE Zn-Ni plated coupons. Boeing indicated that it might not be possible due to the shape and amount of surface area on the 1a.1 re-embrittlement coupons. (if possible)

• Response:
  ▪ An accurate corrosion potential test in salt water per ASTM G5 could not be conducted because of the following:
    • Geometry differences of the 1a.1 coupon vs. standard ASTM G5 corrosion potential test coupon
    • The 1a.1 coupons were contaminated by salt water exposure during initial re-embrittlement testing.
• ‘Re-’Embrittlement Test Action Item #3:
  ▪ Determine the quality thickness of the Cd, Zn-Ni and IVD evaluated in the salt water test.

• Response:
  ▪ Quality and thickness of plating were unacceptable
  ▪ See Action Item #1 addressed poor plating quality
  ▪ Salt water testing was repeated
• Conclusion: Poor plating cause identified and corrected
• Zn-Ni testing was repeated with production plating process and passed ASTM 519 hydrogen ‘re-’ embrittlement service environment test in 3.5% salt water
Fatigue Testing

• Fatigue Test Action Item #1:
  • Clearly document any test data that was discarded in the fatigue test plots and the supporting rational and provide it to OO-ALC/GH, AFRL/RX and ASC/EN

• Response:
  • All fatigue test data was included in the statistical analysis
Fatigue Testing

- Fatigue Test Action Item #2:
  - Provide information on the measured plating thickness on each specimen to OO-ALC/GH, AFRL/RX, and ASC/EN

- Response:
  - A conservative approach was taken when the Zn-Ni fatigue coupons were plated:
    - All Zn-Ni fatigue coupons were plated thicker than cadmium fatigue coupons (typical thickness 0.0002 - 0.0006 inches)
    - The nickel content for Dipsol Zn-Ni IZ-C17+ was closer to the upper limit (18%) of the USAF 201027456 plating specification drawing

<table>
<thead>
<tr>
<th>Average Plating Thickness</th>
<th>(Inches)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cadmium</td>
<td>0.00044</td>
</tr>
<tr>
<td>Dipsol Zn-Ni Tri CC</td>
<td>0.00091</td>
</tr>
<tr>
<td>Dipsol Zn-Ni Hex CC</td>
<td>0.00104</td>
</tr>
<tr>
<td>Atotech Zn-Ni Tri CC</td>
<td>0.00089</td>
</tr>
<tr>
<td>Atotect Zn-Ni Hex CC</td>
<td>0.00081</td>
</tr>
</tbody>
</table>
Fatigue Testing

• Fatigue Test Action Item #3:
  - OO-ALC/GH to engage the Landing Gear Design Industry and determine if the fatigue testing and test results per the following fatigue testing matrixes is adequate to approve the use of LHE
  
  Response:

• Goodrich Landing Gear, John Goering; when asked, “…considering the test method for comparative fatigue debit that we have conducted; would you say that you have complete, very high, high, medium or low confidence that this LHE Zn-Ni coating would not induce a greater fatigue debit than cadmium?”  Response was high to very high.
Phase II Fatigue Testing

- Boeing Commercial (SDT) group evaluated the LHE Zn-Ni fatigue data and saw nothing that would alter their conclusion of the acceptability of the use of LHE Zn-Ni on high strength steel landing gear components
  - Boeing Commercial has approved LHE Zn-Ni for high strength steel and is currently installing a LHE plating line
  - Structural Design Team stated that only one stress ratio is necessary and testing at different R ratios will yield the same result.

- Dr. Andrew Halfpenny, a fatigue expert, from HBN Inc., reviewed the fatigue data and determined that the LHE Zn-Ni is a suitable drop in replacement for cadmium
Phase II Fatigue Testing

• Heroux-Devtek stress group evaluated the LHE Zn-Ni fatigue data and concluded it is acceptable for use on high strength steel landing gear components
  ▪ Heroux-Devtek has approved LHE Zn-Ni for high strength steel and is currently installing a LHE plating line
  ▪ Stress group stated that only one stress ratio is necessary and testing at different R ratios will yield the same result

• Boeing-Long Beach, structures group, would like to see additional testing (with more R ratios)
  ▪ ES3 conducted two different R rations during testing
  ▪ Boeing-Long Beach did not indicate how many test, at what R ratio would be satisfactory to them
Planned Evaluation

- Prototype line in Bldg 505
- Field Performance Evaluations
### Part Matrix

<table>
<thead>
<tr>
<th>Component</th>
<th>Part #</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-5 MLG Stop Plate</td>
<td>4G11453-101B</td>
</tr>
<tr>
<td>F-15 MLG Outer Cylinder</td>
<td>68A412702-1001/1002</td>
</tr>
<tr>
<td>B-1 MLG Axle</td>
<td>1881B85</td>
</tr>
<tr>
<td>F-15 MLG Lower Drag Brace</td>
<td>68A410792-2001</td>
</tr>
<tr>
<td>A-10 MLG Torque Arm</td>
<td>19046-1</td>
</tr>
<tr>
<td>F-16 NLG Inner Cylinder</td>
<td>2007644-103</td>
</tr>
<tr>
<td>C-5 MLG Rotation Collar</td>
<td>4G13565-101A/-101B</td>
</tr>
<tr>
<td>A-10 NLG Axle</td>
<td>18800-3</td>
</tr>
</tbody>
</table>
Phase III Effort
Prototype Parts

A-10 NLG Axle

A-10 MLG Torque Arm

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Phase III Effort
Prototype Parts

F-15 MLG Cylinder
F-16 NLG Inner Cylinder
F-15 MLG Lower Drag Brace
QUESTIONS