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

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

• Small Business Innovative Research (SBIR) Phase I Feasibility Study
• The specific technical objectives of the SBIR Phase II effort are as follows:
  ▪ Successfully pass the following performance parameters, using a prototype tank installation:
    • Installation of a prototype tank for plating demonstration on test coupons and full size components
    • Adhesion
    • Hydrogen Embrittlement
    • Re-embrittlement
    • Liquid and Solid Metal Embrittlement (LME/SME)
    • Fatigue
    • Corrosion
    • Brush Plating for repair of damaged LHE Zn-Ni coatings (Touch Up)
    • Torque Tension
    • Review requirements for Non Destructive Inspection (NDI)
    • Optimize conversion coating
    • Option 1: Develop an accelerated hydrogen embrittlement test procedure:
ES3 implemented a tank of approximately 325 gallons for the purpose of demonstrating the LHE Zn-Ni plating process on some full sized gear components.

The demonstration tank was used to develop uniform plating thicknesses and process parameters on test coupons and full scale landing gear components.

During the plating operations Quality Assurance testing has been conducted to ensure the alkaline LHE Zn-Ni solution is within proper process limits.
Prototype Tank Implementation

Tri-Chromium Conversion Coat Tank
Bend to Break Adhesion Test Coupons

- Adhesion of the LHE Zn-Ni coating to the substrate was tested per ASTM B571
- All adhesion test coupons were manufactured from 1”x 4”x 0.040” 4130 steel sheet
- Results: All Test coupons passed
Dipsol™ LHE Zn-Ni Test Panels After Dry and Wet Tape Adhesion Test of Primer

Deft 44-GN-072

Deft 44-GN-098
Dry and Wet Paint Adhesion Test Results
per ASTM D3359

<table>
<thead>
<tr>
<th>PANEL</th>
<th>ID</th>
<th>PRETREATMENT</th>
<th>COATING</th>
<th>DRY TAPE ADHESION</th>
<th>WET TAPE ADHESION</th>
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<tbody>
<tr>
<td></td>
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<td></td>
<td>PERCENTAGE</td>
<td>PERCENTAGE</td>
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<td>COATING REMOVED</td>
<td>COATING REMOVED</td>
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<tr>
<td>1</td>
<td></td>
<td>LHE Zn-Ni Plating</td>
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<td>2</td>
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<td>0</td>
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<tr>
<td>1</td>
<td></td>
<td>Cd Plated w/ Hex Cr Conversion Coating</td>
<td></td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2</td>
<td></td>
<td></td>
<td></td>
<td>0</td>
<td>0</td>
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<tr>
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<tr>
<td>6</td>
<td></td>
<td></td>
<td></td>
<td>0</td>
<td>0</td>
</tr>
</tbody>
</table>

Notes:
Panels immersed in distilled water at room temperature for 24 hours.

(1): ASTM D 3359 Criteria:
- 5A - No peeling or removal
- 4A - Trace peeling or removal along incisions
- 3A - Jagged removal along incisions up to 1/16 inch on either side
- 2A - Jagged removal along most of incisions up to 1/8 inch on either side
- 1A - Removal from most of the area of the “X” under the tape
- 0A - Removal beyond the area of the “X”

(2): The primer shall show no adhesion failure.
LHE Zn-Ni Hydrogen Embrittlement Testing

- Coupons manufactured per ASTM F519 specifications (4340)
- Coupons plated and tested 28th April, 2009 upon initial installment of LHE Zn-Ni demonstration tank
- Additional coupons plated and tested at additional dates
- All coupons tested per ASTM F519 and passed the 200 hour sustained load tests @ 75% of the tensile notch fracture strength

ASTM F519 Type 1A.1 Test Coupons
HE Plated Cross Section

Cad Plated in Bldg 505 for 5 mins - 200x

.274 mils

.772 mils
LHE Zn-Ni Plated @ 40 ASF for 20 mins - 200x
LHE Zn-Ni Hydrogen Re-Embrittlement Testing

LHE Zn-Ni Re-Embrittlement Testing Machine

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LHE Zn-Ni Hydrogen Re-Embrittlement Testing

Re-Embrittlement Test Matrix

<table>
<thead>
<tr>
<th>Plating</th>
<th>Test Solution</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Distilled Water @ Room Temp Tested 45% NFS for 150Hrs</td>
</tr>
<tr>
<td>LHE Zn-Ni</td>
<td>Pass</td>
</tr>
<tr>
<td>Cadmium</td>
<td>Pass</td>
</tr>
<tr>
<td>IVD</td>
<td>Failed</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 results:
• Coupons tested by an ISO 9001 certified facility. Coupons tested IAW ASTM F519.
  The coupons tested immersed in solutions of Water, 3.5% Salt Water, Dilute* Calla 296, Dilute* Calla 602LF, Concentrated Calla 296, and Concentrated Calla 602LF.
  *NOTE – Dilute means mix cleaning solution to manufacturer’s recommended use concentration and heat to manufacturer’s maximum recommended use temperature.
• Cleaning solutions used in testing were:
  Calla 296
  Calla 602LF
• LHE Zn-Ni performs better than IVD and as well as Cad
Liquid/Solid Embrittlement Testing

- Liquid and Solid Metal Embrittlement (LME and SME) occur when one metal, either as a liquid or solid, intrudes into the structure of another, potentially causing embrittlement in the base metal.
- Melting points for the coating metals are as follows:
  - Cadmium ~610°F
  - Zinc ~787°F
  - Nickel ~2650°F

<table>
<thead>
<tr>
<th>Temp/NFS</th>
<th>Material</th>
<th>LHE Zn-Ni 200Hr</th>
<th>Cad 200Hr</th>
<th>LHE Zn-Ni Step Load</th>
<th>Cad Step Load</th>
</tr>
</thead>
<tbody>
<tr>
<td>600F/85%</td>
<td>300M</td>
<td>Pass</td>
<td>Fail</td>
<td>100% NFS</td>
<td>-N/A-</td>
</tr>
<tr>
<td>500F/85%</td>
<td>300M</td>
<td>Pass</td>
<td>Fail</td>
<td>100% NFS</td>
<td>87% NFS</td>
</tr>
<tr>
<td>400F/85%</td>
<td>300M</td>
<td>Pass</td>
<td>Fail</td>
<td>100% NFS</td>
<td>91% NFS</td>
</tr>
<tr>
<td>400F/75%</td>
<td>300M</td>
<td>Pass</td>
<td>Fail</td>
<td>100% NFS</td>
<td>81% NFS</td>
</tr>
</tbody>
</table>
LHE Zn-Ni and Cad Type 1a.1 Specimens After ISL Test to Determine the NFS After Exposure to 400°F for 200 Hours

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Fatigue Testing

• Phase II LHE Zn-Ni fatigue testing is an extension of Phase I work
• Phase II LHE Zn-Ni fatigue testing continues to broaden the data base and increase the statistical validity of the data
• Manufacturing of coupons and Fatigue Testing IAW ASTM E466
  ▪ All coupons were plated per manufacture’s plating solution limits
Phase I Fatigue Testing
(Shotpeened Coupons)

C-17 P2 Program Fatigue Data (2005)
(IZ-C17 with Hex conversion coating)
## Phase II Fatigue Testing

<table>
<thead>
<tr>
<th>Shotpeened Coupons</th>
<th>Stress Loads (KSI) R= -0.3</th>
<th>Total Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>160</td>
<td>180</td>
</tr>
<tr>
<td>Bare</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Cad Plated</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Zn-Ni Plated Tri Chrome</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Zn-Ni Plated Hex Chrome</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>*Zn-Ni Plated Atotech Tri Chrome</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Zn-Ni Plated Atotech Hex Chrome</td>
<td>5</td>
<td>5</td>
</tr>
<tr>
<td>Total Fatigue Coupons</td>
<td></td>
<td></td>
</tr>
<tr>
<td>*Bake before Tri CC</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Phase II Fatigue Testing

Peened Fatigue Coupon Test Results

Test Load (KSI) R = -0.3

Number of Cycles

Bare ▲ Cad ▲ Zn-Ni Tri CC (Dipsol) ▲ Zn-Ni Hex CC (Dipsol) ▲ Zn-Ni Tri CC (Atotech) ▲ Zn-Ni Hex CC (Atotech)
Phase II Fatigue Testing

Means Compared @ 95% limits: 200 ksi Peened

- The means of groups Cad and bare are significantly different.

- The means are: 33104, 26275, 30496, 33482, and 31037.
Phase II Fatigue Testing

Means Compared @ 95% limits: 180 ksi Peened

No groups have means significantly different from Cad.
Phase II Fatigue Testing

Means Compared @ 95% limits: 160 ksi Peened

No groups have means significantly different from Cad

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Corrosion Performance

- Corrosion tests were conducted on LHE Zn-Ni coupons with cadmium as the baseline.
- Testing was also performed on both cadmium and LHE Zn-Ni coated coupons with a prime/paint topcoat after being scribed (See Table below). All test coupons were 4”x 6”x 0.040” 4130 steel sheet.
- All testing was performed per ASTM B117.
- Test specimens were both scribed and un-scribed.

### Corrosion Test Matrix

<table>
<thead>
<tr>
<th># of steel Panels</th>
<th>Plating</th>
<th>Scribed</th>
<th>Prime/Paint</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>LHE Zn-Ni</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>3</td>
<td>LHE Zn-Ni</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td>3</td>
<td>Cd</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>3</td>
<td>Cd</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td>3</td>
<td>LHE Zn-Ni</td>
<td>Yes</td>
<td>Yes</td>
</tr>
<tr>
<td>3</td>
<td>Cd</td>
<td>Yes</td>
<td>Yes</td>
</tr>
</tbody>
</table>
Cadmium Coatings – Phase II ASTM B 117 Panels @ Boeing Unscribed

Cadmium with Hexavalent Chrome Conversion Coating
Unscribed – ASTM B 117

Figure 3

Test Panel Removed From Salt Spray Cabinet – Excess Amount of Red Rust Detected

5000 hours

3000 hours

1000 hours

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Corrosion Performance
Corrosion Performance

IZ-C17+ Zn-Ni with Trivalent Chrome Conversion Coating
Unscribed – ASTM B 117 Panels @ Boeing (Unscribed)

Figure 4

1000 hours

3000 hours

5000 hours

Zinc Nickel Coatings – Phase II ASTM B 117 Panels @ Boeing (Unscribed)

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Corrosion Performance

Figure 5

Cadmium with Hexavalent Chrome Conversion Coating

Test Panel Removed From Salt Spray Cabinet – Excess Amount of Red Rust Detected

Test Panel Removed From Salt Spray Cabinet – Excess Amount of Red Rust Detected

1000 hours

Test Panel Removed From Salt Spray Cabinet – Excess Amount of Red Rust Detected

Test Panel Removed From Salt Spray Cabinet – Excess Amount of Red Rust Detected

3000 hours

Test Panel Removed From Salt Spray Cabinet – Excess Amount of Red Rust Detected

Test Panel Removed From Salt Spray Cabinet – Excess Amount of Red Rust Detected

5000 hours

Cadmium Coatings – Phase II ASTM B 117 Panels @ Boeing (Scribed)

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Corrosion Performance

IZC17+ Zn-Ni with Trivalent Chrome Conversion Coating Scribed – ASTM B 117

1000 hours

3000 hours

5000 hours

Zinc Nickel Coatings – Phase II ASTM B 117 Panels @ Boeing (Scribed)
Corrosion Performance

Cadmium Coatings – Phase II ASTM B 117 Panels @ Boeing (Painted/Scribed)

Test Panel
Removed From
Salt Spray Cabinet –
Excess Amount
of Red Rust
Detected

Test Panel
Removed From
Salt Spray Cabinet –
Excess Amount
of Red Rust
Detected

Test Panel
Removed From
Salt Spray Cabinet –
Excess Amount
of Red Rust
Detected

Test Panel
Removed From
Salt Spray Cabinet –
Excess Amount
of Red Rust
Detected

Test Panel
Removed From
Salt Spray Cabinet –
Excess Amount
of Red Rust
Detected

Test Panel
Removed From
Salt Spray Cabinet –
Excess Amount
of Red Rust
Detected

Cadmium with Hexavalent Chrome Conversion Coating
Scribed Painted – ASTM B 117

Figure 9

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Corrosion Performance

IZ-C17+ Zn-Ni with Trivalent Chrome Conversion Coating
Scribed Painted – ASTM B 117

Test Panel
Removed From Salt Spray Cabinet – Excess Amount of Red Rust Detected

Zinc Nickel Coatings – Phase II ASTM B 117 Panels @ Boeing (Painted/Scribed)

1000 hours
3000 hours
5000 hours

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G 85 SO$_2$ Corrosion-Cad

![Corrosion samples after 120, 168, and 336 hours](image)

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G 85 SO$_2$ Corrosion-LHE Zn-Ni
Brush Plating Repair

• In order for a brush LHE Zn-Ni plating to qualify it must pass the following tests:
  ▪ Hydrogen Embrittlement (HE) testing per ASTM F519
  ▪ Bend to break adhesion test per ASTM B571
  ▪ Corrosion testing per ASTM B117

• SIFCO recommended procedures were used to plate several sets of HE type 1a.1 coupons, adhesion coupons, and corrosion coupons, using SIFCO 4018 No Bake LHE Zn-Ni brush plating solution

• Test Results Summary:
  ▪ Passed HE testing
  ▪ Passed adhesion testing on steel and LHE Zn-Ni plated steel
  ▪ Corrosion test performance is excellent
Torque Tension

- Robins AFB Cad plating replacement on threaded fastener and components
  - Typical chart for run on – break away test showing Cad vs. LHE Alkaline Zn-Ni
Torque Tension

- Robins AFB Cad plating replacement on threaded fastener and components
  - Typical chart for Torque Tension Test showing Cad vs. LHE Alkaline Zn-Ni with MIL-PRF-83483 Anti-seize grease lubricant
Non-Destructive Inspection (NDI)

- OSHA Permissible Exposure Limits (PELs) for LHE Zn-Ni are high enough, such that, the LHE Zn-Ni plating does not have to be removed during the overhaul process (currently all cadmium plated landing gear components which come into the overhaul facility are stripped and cleaned per OSHA PELs).

- There is no NDI interference due to the LHE Zn-Ni coating itself, consequently we can do the following inspections without removing the LHE Zn-Ni coating:
  - Fluorescence Magnetic Particle Inspection (FMPI)
  - Ultra Sonic Inspection
  - Eddy current
  - X-Ray
Conversion Coating Optimization

- Testing was conducted to determine the optimal conversion coating (CC) (Hexavalent vs. Trivalent) and parameters:
  - Baking before and after conversion coating
    - Hexavalent CC: must be applied after bake
    - Trivalent CC: can be applied before or after bake (process time savings)
  - Paint adhesion performance per ASTM D3359
    - Hex-CC – Passed
    - Tri-CC – Passed
  - Corrosion performance per ASTM B117
    - Hex-CC – Passed
    - Tri-CC – Passed (performs slightly better)
  - Hydrogen embrittlement per ASTM F519
    - Hex-CC – Passed
    - Tri-CC – Passed
Conversion Coating Optimization

- Fatigue Performance per ASTM E466
  - Hex-CC – Passed
  - Tri-CC – Passed

- Process parameters
  - Submerge in conversion coat solution for 1–2 minutes

- Environmental concerns
  - DoD is currently trying to eliminate Hex-CC use

- Recommend Trivalent CC
Development of an Accelerated HE Test

- Accelerated HE Test Development
  - Necessary to production process
  - Accelerated results in < 24 hours
  - Must correlate to 200 hour test
- Three rapid hydrogen embrittlement test methods have been identified for potential use at Hill AFB
  - The Messier-Dowty 16 Hour Hydrogen Embrittlement Test
  - ASTM F 1940 Rapid Hydrogen Embrittlement Test for High Strength Steel Fasteners
  - ASTM F 1624 – 09 Determination of Hydrogen Embrittlement Threshold Using the ISL (Incremental Step Load) Test Method

Rising Step Load Apparatus
The preferred method to carry out a rapid hydrogen embrittlement test is ASTM F1624 which is as follows:

- Using the incremental test method in ASTM F 1624, determine the NFS_{F1624} for new – unplated ASTM F 519 Type 1a.1 test specimens. NFS_{F1624} will be lower than NFS_{E8}
- Qualified rapid hydrogen embrittlement test method (< 20 hrs):
  - Load Type 1a.1 test specimen in Rising Step Load machine and perform step load using ISL conditions (20,5,1) = (20 steps, 5% NFS_{F1624} per step, hold for 1 hour at each step). Determine load where test specimen breaks, this is threshold load P_{th}
  - \%NFS = 100 x P_{th} / NFS_{F1624}
  - If \%NFS > 90% then process is non-embrittling

Rising Step Load Apparatus
Phase III - Efforts

- Phase III – tasks consisted of the following:
  - Waste stream characterization and mitigation
    - Similar wastewater treatment as cadmium
  - Permitting for hazardous material
    - New source sheets ready to submit
    - Awaiting finalization of the LHE Zn-Ni plating line (i.e. tank size and location)
    - Will apply for Title V air permit when the LHE Zn-Ni plating line is installed
  - Process validation
  - Specification and source control drawings
  - Prototype depot LHE Zn-Ni process line
    - With landing gear components
  - Process orders and Process Orders Document and Display System (PODDS) support
  - Engineering and Program Management Support
Critical LHE Zn-Ni process parameters were established for the following:

- **Type of Anode**
  - Nickel 200

- **Plating fixtures**
  - Fixture material can be either stainless (preferred) or carbon steel

- **Current density**
  - 40–60 Amps per square foot (ASF)

- **Plating time**
  - 10–20 minutes

- **Plating thickness and type of conversion coating**
  - **Class**
    - I – minimum of 0.0005” ~ (40 ASF for 20 minutes)
    - II – minimum of 0.0003” ~ (40 ASF for 13 minutes)
    - III – 0.0001 – 0.0002” ~ (40 ASF for 10 minutes)
  - **Type**
    - I – No conversion coat
    - II – Trivalent or Hexavalent conversion coat
• Critical LHE Zn-Ni process parameters were established for the following:
  ▪ Bath chemistry
    • Requires bi-weekly chemical analysis of the LHE Zn-Ni plating tank using titration
      ▫ Zinc
      ▫ Nickel
      ▫ Carbonate
      ▫ Sodium Hydroxide
    • Requires bi-weekly chemical analysis of the Trivalent CC tank:
      ▫ pH level
      ▫ Cr level
  ▪ Agitation requirements
    • Agitation should be sufficient enough to re-circulate the tanks plating solution a minimum of 3 times per hour
    • No air agitation
Tank chemical maintenance

- Chemical maintenance for the LHE Zn-Ni process is based on usage and production rates
- Analysis and adjustment should be performed on an amp-hour used basis, or on a weekly basis
- Some degree of tracking will be required to establish a historical database of information, which can then be used to recommend regular analysis intervals
- Specific limits and analysis instructions are provided in the LHE Zn-Ni specification drawing

Effects of precipitation (carbonate) build up

- Carbonate formation will decrease the plating efficiency, and perhaps the adhesion performance
- The best solution is to continually treat out carbonates rather than batch treat in the winter and deal with cyclic carbonate levels and larger replenishment additions
- A Carbolux™ system is recommended for treatment of the carbonates
  - It is a mechanism which continually removes solution, precipitates out carbonates, places them in a disposal hopper, and then returns the treated solution back to the plating tank
Quality compliance testing

- The following monthly compliance testing is required:
  - Bend to break adhesion coupons per ASTM B571 on plated 1”x 4”x 0.040” coupons (4ea)
  - Neutral salt spray corrosion testing per ASTM B117 on plated 4”x 6” coupons (2ea)
  - Hydrogen embrittlement testing per ASTM F519 on plated Type 1.a1 coupons (4ea)
## Phase III Effort
### Prototype Process Line

<table>
<thead>
<tr>
<th>Component</th>
<th>Part #</th>
</tr>
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<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>
A-10 NLG Axle

A-10 MLG Torque Arm
Phase III Effort
Prototype Parts

B-1B MLG Axle

C-5 MLG Rotation Collar

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Phase III Effort
Prototype Parts

F-15 MLG Cylinder     F-16 NLG Inner Cylinder     F-15 MLG Lower Drag Brace
Phase III Effort
Prototype Anode Design

C-5 MLG Rotation Collar

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Phase III Effort
Prototype B-1 Bushing

Plating B-1 Bushings LHE Zn-Ni

Plated LHE Zn-Ni B-1 Bushings
Phase III Effort
Proposed Prototype Plating Line

TANK REQUIREMENTS

- Zn-Ni PLATING
- 1ST RINSE
- 2ND RINSE
- ACID ACTIVATOR
- RINSE
- CONVERSION COAT
- RINSE
- EAST END

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### APPLICATION REVISIONS

<table>
<thead>
<tr>
<th>NEXT ASSY</th>
<th>USED ON</th>
<th>REV</th>
<th>DESCRIPTION</th>
<th>DATE</th>
<th>APPROVED</th>
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**U.S. AIR FORCE**

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