Cadmium (Tank) Electroplating Alternative
(NESDI Project ID: 450)

John E. Benfer, M.S.
Corrosion Engineering Team Lead
NAVAIR Associate Fellow

NAVAIR Jacksonville
Phone: (904) 542-4516, x153
Email: john.benfer@navy.mil

Ruben A. Prado, CEF
Inorganic Coatings SME
NAVAIR Associate Fellow

NAVAIR Jacksonville
Phone: (904) 542-3444, x106
Email: ruben.prado@navy.mil
Report Documentation Page

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Standard Form 298 (Rev. 8-98)
Proscribed by ANSI Std Z39-18
Technical Objectives

1.) Dem/Val Alkaline Zn-Ni (DIPSOL IZ-C17+) as an alternative to tank cadmium electroplating on high strength steel/general surfaces within Depot level maintenance

2.) Dem/Val Tri-Cr (DIPSOL IZ-264) as an alternative to conventional hexavalent post treatments on the above alkaline Zn-Ni deposit.

- Fully define deposition parameters and properties
- Establish production plating processes (i.e., cleaning, racking, masking, activation, pre-plates, stripping, etc.)
- Test/Validate performance
- NAVAIR Authorization Letter
- Develop Eng Tech Data Packages
  - Manuals
  - Specifications
  - Eng. Circular

FRCSE Toxic Metal Control Program (FRCSEINST 5103.15) requires replacement with available alternative technology.

Demo Site: FRC JAX
Project Team

FRCSE-JAX
Luzmarie Guzman-Santiago, PI

NAVAIR JAX
Luzmarie Guzman-Santiago
- Process Line
- Set-Up
- Producibility
- Testing
- Testing
- Component
- Plating and
- demonstration
- Reporting

NAVAIR JAX
Jack Benfer
- Testing and
- Approval

NAVAIR JAX
Ruben Prado
- Optimization
- Transition

NAVAIR PAX
Craig Matzdorf
- Requirements

Luzmarie Guzman-Santiago
NAVAIR Jacksonville
Phone: 904-790-6396
E-mail: luzmarie.guzman-sant@navy.mil
DIPSOL IZ-C17+ (Zn-Ni) & DIPSOL IZ-264 (Conversion Coating)

DIPSOL IZ-C17+ is an alkaline, cyanide free, Zn-Ni alloy electroplating process.
- Meets requirements for a non-embrittling process per ASTM F 519 for HSS.
- Has excellent throwing/covering power
- Uniform zinc alloy deposit containing 12 – 18% Ni
- Excellent heat and corrosion resistant properties
- Plating rate: 0.8 – 1 mil/hr @ 46.5 ASF
- Hardness: 350-450 kg/mm² (VHN)

DIPSOL IZ-264 is a trivalent chrome conversion coating for DIPSOL IZ-C17.
- Blue bright coating/ excellent corrosion resistance.
- Chromate film thickness is under 100nm.
- Can be applied prior to HE Baking
- Ambient Bath
Dem/Val Site Description

- FRCSE cyanide waste stream for FY09 was approximately 20.8K lbs (30% associated with cadmium tank electroplating).

- Existing line in FRCSE JAX Bldg 794 will be utilized for setting up the prototype line.

- 46 process tanks
- 2 IVD Chambers
- 23,800 ft² Plating facility
### Technology Description

**Alkaline Zn-Ni (IZ-C17+) Dem/Val Process Line, FRCSE**

<table>
<thead>
<tr>
<th>Tank#</th>
<th>Process Step</th>
<th>Chemistry</th>
<th>Gals</th>
<th>Temp (°F)</th>
</tr>
</thead>
<tbody>
<tr>
<td>J-8</td>
<td>Activation †</td>
<td>Acid</td>
<td>-</td>
<td>Ambient</td>
</tr>
<tr>
<td>J-7</td>
<td>Rinse</td>
<td>Di H20</td>
<td>175</td>
<td>Ambient</td>
</tr>
<tr>
<td>J-6</td>
<td>Zn-Ni Plate</td>
<td>Dipsol IZ-C17+</td>
<td>210</td>
<td>73 - 83</td>
</tr>
<tr>
<td>J-5</td>
<td>Rinse</td>
<td>Di H20</td>
<td>210</td>
<td>75 - 80</td>
</tr>
<tr>
<td>J-4</td>
<td>Activation</td>
<td>HCL (0.1%)</td>
<td>140</td>
<td>Ambient</td>
</tr>
<tr>
<td>J-3</td>
<td>Rinse</td>
<td>Di H20</td>
<td>180</td>
<td>Ambient</td>
</tr>
<tr>
<td>J-2</td>
<td>Conversion Coat</td>
<td>Dipsol IZ-264</td>
<td>135</td>
<td>70 - 85</td>
</tr>
<tr>
<td>J-1</td>
<td>Rinse</td>
<td>Di H20</td>
<td>-</td>
<td>140</td>
</tr>
</tbody>
</table>

* Tank is to have necessary electrical requirements to support existing chiller unit, solution pump & In-tank filtration pump, heater element & associated controllers. A valve shall be put in place (chiller loop) to maintain operating temperature of bath. All plumbing, connectors, etc. are to be chemically resistant (alkaline material). † Mechanical Activation/Blasting may be used.
**Demonstration Facility**

**NAVAIR Fleet Readiness Center Jacksonville**

- Alkaline Zn-Ni Dem/Val line
- Chiller/Solution Pump
- 210 gallon Plating Tank
- DC Power Rectifier

![Images of the demonstration facility, including a Zn-Ni Plating Tank and a Chiller/Pump.]
Integration at Hill AFB

- NAVAIR JAX Visit to Hill AFB (Phase III SBIR Effort)

- Visit to ES3 facility
- Gained Lessons Learned from Hill AFB setup
- Discussed Process Flow & Plating issues

IZ-C17+ Zn-Ni Tank @ Hill AFB
IZ-264 TriCr Tank @ Hill AFB

Plating Line @ Hill AFB
Technical Approach

**Phase I**
- Set-Up & Modification of J-Line
- Procure Chemistries
- Coupon Testing
- Validation/Optimization of Process

**Phase II**
- Selection of Dem Val parts
- Demonstrate capability
- Test Technology in a Fleet operational testing
- NAVAIR approval letter + Implement technology

(NEStD Project ID: 450)
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<th>METRIC</th>
<th>DATA REQUIREMENTS</th>
<th>SUCCESS CRITERIA</th>
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<tbody>
<tr>
<td>Appearance</td>
<td>Visual examination</td>
<td>Visual per MIL-STD 870B</td>
<td>Smooth/Continuous</td>
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<tr>
<td>Adhesion</td>
<td>Bend/chisel ASTM B571</td>
<td>180° bend to break</td>
<td>No lose of adhesion</td>
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<tr>
<td>Throwing Power &amp; Alloy Composition Uniformity</td>
<td>(XRF) / SEM Method</td>
<td>Composition: 12 – 18% nickel</td>
<td>Consistent Alloy Composition</td>
</tr>
<tr>
<td>Thickness</td>
<td>Microscopic ASTM B487</td>
<td>Thickness measurement (mils).</td>
<td>Compare w/ LHE</td>
</tr>
<tr>
<td>Porosity</td>
<td>Ferroxyl Test</td>
<td>Performance =&gt; Cadmium</td>
<td>Compare w/ LHE</td>
</tr>
<tr>
<td>Usability</td>
<td>Efficiency of personnel to plate</td>
<td>Feedback from artisans/electroplaters on usability of technology and time requirements</td>
<td>Minimal operator training required</td>
</tr>
<tr>
<td>Solution Maintenance</td>
<td>Efficiency of personnel to analyze the solutions</td>
<td>Feedback from Chemist on maintenance issues</td>
<td>Less or equal maintenance</td>
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<tr>
<td>Corrosion (B117)</td>
<td>Salt fog ASTM B117</td>
<td>Shall not show white corrosion at the end of 96 hrs</td>
<td>Compare w/ LHE</td>
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<tr>
<td>Corrosion (SO₂)</td>
<td>SO₂ salt fog ASTM G85 A4</td>
<td>Dependant on thickness</td>
<td>Compare w/ LHE</td>
</tr>
<tr>
<td>SCC</td>
<td>Stress-corrosion cracking</td>
<td>Performance =&gt; Cadmium</td>
<td>Compare w/ LHE</td>
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<tr>
<td>Fatigue</td>
<td>Corrosion fatigue testing</td>
<td>Per ASTM E466</td>
<td>Compare w/ LHE</td>
</tr>
<tr>
<td>Hydrogen Embrittlement and Re-embrittlement</td>
<td>Hydrogen embrittlement ASTM F519</td>
<td>HE: 75% NFS 200 hrs HRE: 45% NFS 150 hrs</td>
<td>Threshold limit greater than /equal to LHE Cd</td>
</tr>
</tbody>
</table>
Key Performance Criteria

- **Stress Corrosion Cracking:** Evaluate effect of the Zn-Ni coating on the fracture properties of the underlying substrate as compared to Cadmium.

- **Corrosion Fatigue – ASTM E466-96:** Evaluate effect of the Zn-Ni coating on the fatigue performance properties of the underlying substrate as compared to Cadmium.

- **Corrosion – ASTM B117 and G85:** Evaluate corrosion protection using standard accelerated corrosion tests. The protocol includes both corrosion ranking (amount of corrosion product on the surface) and protection ranking (extent of coating damage), to provide an overall assessment of corrosion performance.

- **Hydrogen Embrittlement – ASTM F519:** Evaluate process susceptibility to hydrogen embrittlement and characterize environmentally assisted cracking per ASTM F519.
SCC Testing

Test Details:
- RSL on notched four point bend specimens and determine threshold stress for initiation of SCC cracks.
- ASTM F519, Type 1.e notched square bar
- Conduct at 3.5% NaCl at OCP w/ Cathodic Over-Potential
- Report: $V_{\text{SCE}}$ -vs- $K_{\text{OSCC}}$
- Report: $K_{\text{OSCC}}$ at OCP (ksi-in)

Variation of $K_{\text{OSCC}}$ with $V_{\text{SCC}}$ for Bare and Coated Specimens

Variation of Threshold Stress Intensity for SCC in Un-Precracked (As-Machined) and Pre-cracked Bare Specimens ($K_{\text{OSCC}}$ with $V_{\text{ISCC}}$, respectively) with Applied Electric Potential $V_{\text{SCC}}$

Type 1.e Notched Square Bar
52-54 HRC
Corrosion Fatigue

- ASTM E466
- AISI 4340, 260-280 KSI
- R= 0.1, f=10hz
- Air, 3.5% NaCl (pH 7.3)
- Generate S-N Curve
- Hour Glass Bar (20/Condition)
HRE Testing

Environmentally Assisted Cracking

Specimens:
ASTM F519 1a.1 Notch Bars
AISI 4340 Alloy Steel, HRC 50-52
NFS is 390 KSI

Re-Embrittlement Test Fluids:
- DI Water, ASTM 1132, Type 2
- Synthetic Sea Water, ASTM D 1141
  (2.5 or 3.5% NaCl)
- MIL-PRF-85570, Type II Cleaner

Test Details:
- ASTM F519
- 45% NFS for 24 hrs +5ksi/hr (Phase I)
  or 45% NFS 150 hrs +5ksi/hr (Phase II)
- Recommend 90° Notch Test Specimens
- Reporting Sustained/Threshold load (%NFS), Time to failure.

Recommended

Hydrogen Re-Embrittlement Test Equipment

Cd Zn-Ni IVD Al LHE Cd
Hydrogen Embrittlement/HRE
ASTM F519 A5, Type 1.a.1

Avg Time to Failure (hrs)

LHE Cd  | Brush Zn-Ni  | IVD Al
--- | --- | ---
Air  | DI Water  | Sea Water  | Cleaner
--- | --- | --- | ---
205 | 195 | 185 | 175 | 165 | 155 | 145
Questions

Ruben Prado, CEF
Inorganic Coatings SME
Naval Air Systems Command
904-790-6381
Ruben.prado@navy.mil

Jack Benfer, M.S.
Corrosion Engineering
Team Lead
Naval Air Systems Command
904-790-6405
John.benfer@navy.mil

Luzmarie G. Santiago
Materials Engineer
Naval Air Systems Command
904-790-6396
luzmarie.guzman-sant@navy.mil
Technology Description

Equipment Set-Up for IOC at JAX

- Ni Anodes used
- Zinc Anodes or Liquid (NZ-777)
- Filtration 2 or 3 turnovers (Eductors)
- Cathode Rocker (1-3 m/min)
Cd Brush Plating Alternative
LHE Zinc-Nickel

FRCSE Evaluation of Brush Zn-Ni

Corrosion (B-117/SO2)
- Overall B-117 showed Cd repair area to perform better than Zn-Ni
- Overall Painted SO2 performance rating (1 = Best; 4 = Worst):
  1 – LHE Cd/Cd
  2 – ZnNi/Cd
  3 – LHE Cd/IVD
  4 – ZnNi/IVD

Hydrogen Embrittlement/Re-Embrittlement
- All coupons passed (LHE Cd, IVD Al, & Brush Zn-Ni)
- Zn-Ni showed slight performance increase compared to baseline (however, with more variability)

Production Application
- Process sensitivity – Current Density
- Appearance
- Odor
### Air Environment

<table>
<thead>
<tr>
<th>Coating</th>
<th>Replicate</th>
<th>Fracture Strength (%)</th>
<th>Time to Failure (Hrs)</th>
<th>Pass/Fail</th>
</tr>
</thead>
<tbody>
<tr>
<td>LHE Cd</td>
<td>1</td>
<td>94.0%</td>
<td>204</td>
<td>Pass</td>
</tr>
<tr>
<td>IVD Al</td>
<td>1</td>
<td>84.6%</td>
<td>201</td>
<td>Pass</td>
</tr>
<tr>
<td>Brush Zn-Ni</td>
<td>4</td>
<td>97.0%</td>
<td>200</td>
<td>Pass</td>
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</tbody>
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**200 hr Sustained Load Test at 75% NFS then step 5% per hr until failure**

### Sea Water Environment

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<th>Coating</th>
<th>Replicate</th>
<th>Fracture Strength (%)</th>
<th>Time to Failure (Hrs)</th>
<th>Pass/Fail</th>
</tr>
</thead>
<tbody>
<tr>
<td>LHE Cd</td>
<td>1</td>
<td>65.2%</td>
<td>154</td>
<td>Pass</td>
</tr>
<tr>
<td>IVD Al</td>
<td>1</td>
<td>55.1%</td>
<td>151</td>
<td>Pass</td>
</tr>
<tr>
<td>Brush Zn-Ni</td>
<td>1</td>
<td>55.0%</td>
<td>152</td>
<td>Pass</td>
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</table>

### DI Water Environment

<table>
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<th>Coating</th>
<th>Replicate</th>
<th>Fracture Strength (%)</th>
<th>Time to Failure (Hrs)</th>
<th>Pass/Fail</th>
</tr>
</thead>
<tbody>
<tr>
<td>LHE Cd</td>
<td>1</td>
<td>65.2%</td>
<td>154</td>
<td>Pass</td>
</tr>
<tr>
<td>IVD Al</td>
<td>1</td>
<td>55.1%</td>
<td>151</td>
<td>Pass</td>
</tr>
<tr>
<td>Brush Zn-Ni</td>
<td>1</td>
<td>55.0%</td>
<td>152</td>
<td>Pass</td>
</tr>
</tbody>
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### MIL-PRF-85570, Type II Cleaner Environment

<table>
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<tr>
<th>Coating</th>
<th>Replicate</th>
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<th>Time to Failure (Hrs)</th>
<th>Pass/Fail</th>
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<tr>
<td>LHE Cd</td>
<td>1</td>
<td>60.3%</td>
<td>153</td>
<td>Pass</td>
</tr>
<tr>
<td>IVD Al</td>
<td>1</td>
<td>89.2%</td>
<td>159</td>
<td>Pass</td>
</tr>
<tr>
<td>Brush Zn-Ni</td>
<td>1</td>
<td>75.5%</td>
<td>155.1</td>
<td>Pass</td>
</tr>
</tbody>
</table>
Repair Test Panels

4” x 6” Panel, AISI 4130 Steel

LHE Cd (2023) on Cd Plating (Tank)

SIFCO 4018/5970 Zinc-Nickel on Cd Plating (Tank)

IVDCD CD IVD

IVD

CD

ZN-Ni

LHE Cd (2023) on IVD Aluminum

SIFCO 4018/5970 Zinc-Nickel on IVD Aluminum

CD

ZN-Ni
NOTES:
4" x 6" Steel Panel
Substrate material: AISI 4130 Steel
Plating per Manufacturer Instructions
Target thickness: 0.5 mil

Primer = MIL-PRF-85582
Top Coat = MIL-PRF-85285

Brush Plated Repair
Existing Plated Area, Cd or IVD
Scribed Area

Area to be Brush Plate Repaired
Up to ~ 0.5" overlap is allowed
Brush Plate Repair Configuration

SO$_2$ Test
ASTM G85 Annex 4
*Average Rating of 3 replicates
Throwing Power

Throwing Power/Composition Uniformity

Test Methodology:
- AISI 4340 Steel Coupons (different orientations to “coating” chamber)
- Measure coating thickness (several locations along panel, ASTM B568)
- Measure Alloy Composition across surface using X-Ray Fluorescence Spectrometry (ASTM E1621)
- Acceptance Criteria: Composition stays within specs. (Document thickness variation.)

Figure 12. Fixture for “Throwing Power” and Alloy Composition Test