Integration of LHE alkaline Zn-Ni into NAVAIR Overhaul and Maintenance Facilities

SBIR Topic AF071-320

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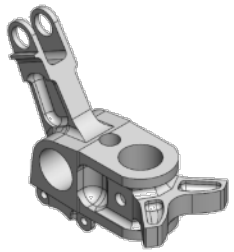
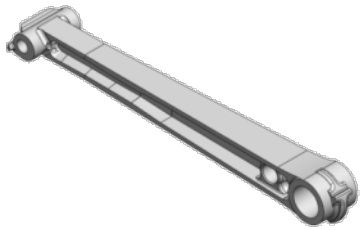
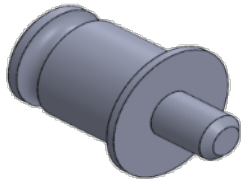
NAVAIR Technical Objectives

- **Transition Zn-Ni electroplating under SBIR Topic AF071-320 as a cadmium replacement for High Strength Steel.**

  - Phase II.5 SBIR STTP - Conduct NAVAIR certification testing with ES3 LHE Alkaline Zn-Ni process.
  - ONR RIF (Phase III) – Implementation of LHE Alkaline Zn-Ni plating line at FRCSE - Completed

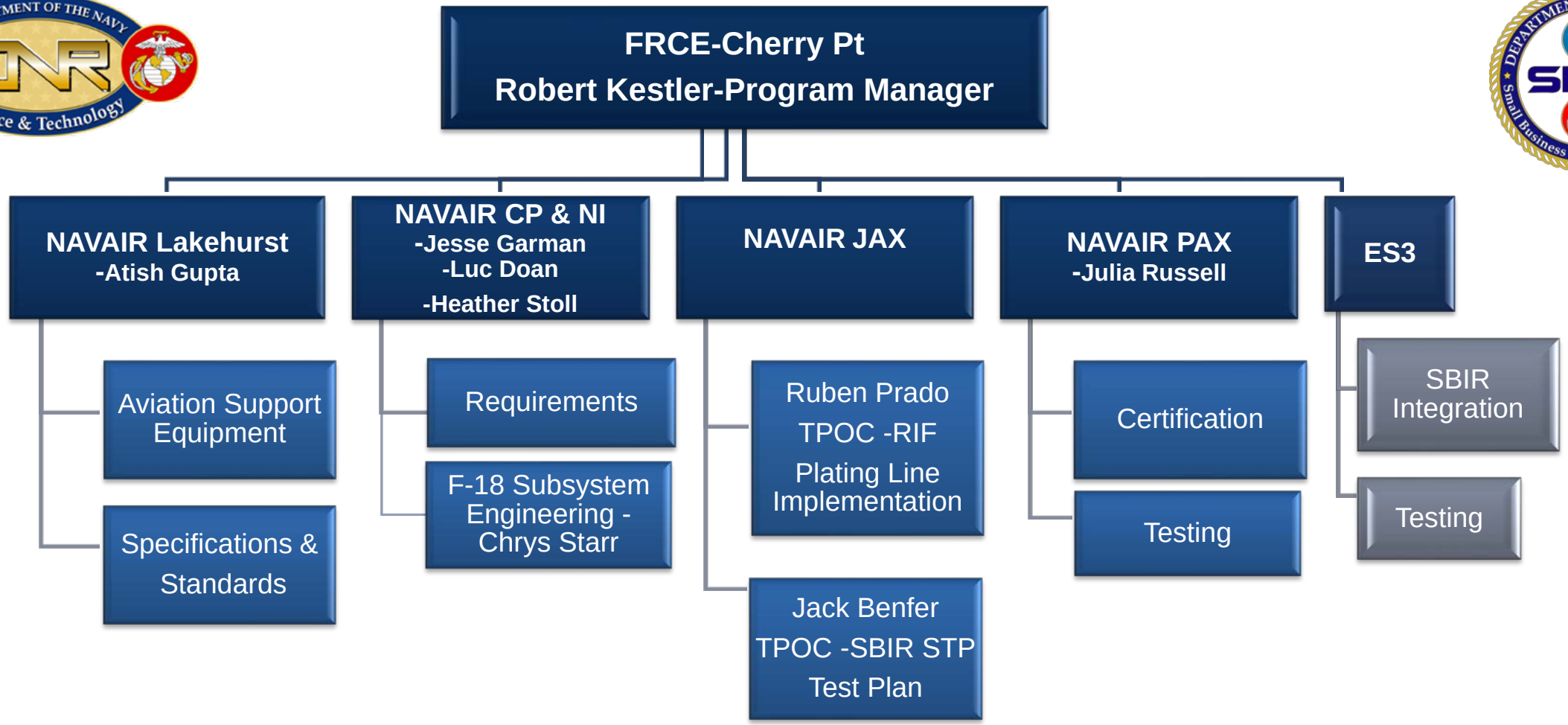






NAVAIR Zn-Ni SBIR Project Team

ONR RIF & SBIR Phase II.5 Project Team



SBIR Phase II.5 Development Process

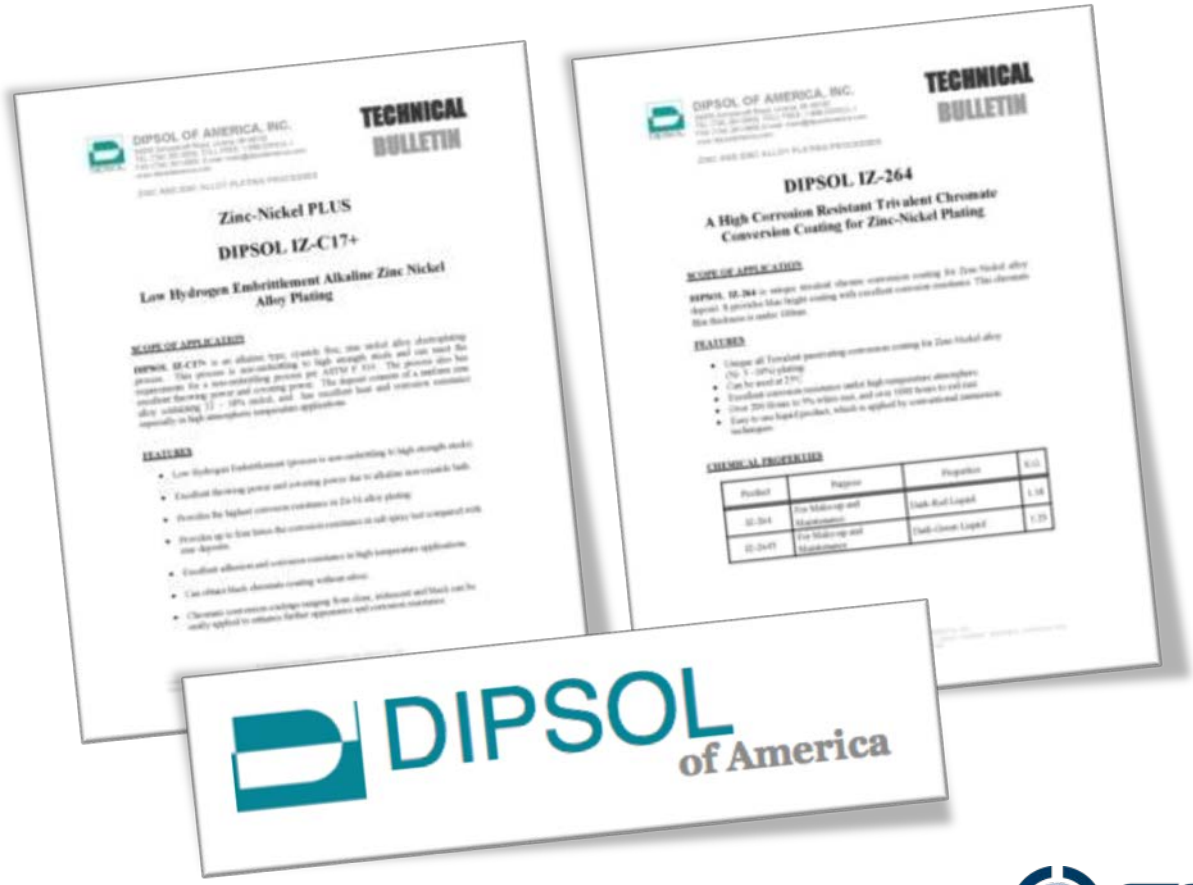
DIPSOL IZ-C17+ (Zn-Ni) & DIPSOL IZ-264 (Conversion Coating)

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 – 15% Ni
- Excellent heat and corrosion resistant properties
- Plating rate: 0.8 – 1 mil/hr @ 45 ASF

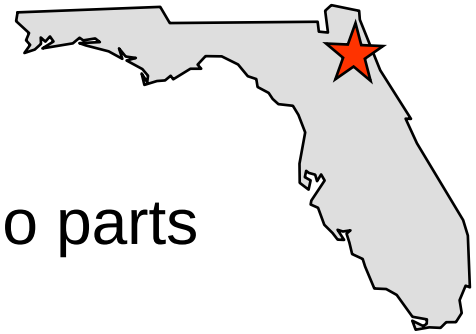
DIPSOL IZ-264 is a trivalent chrome conversion coating for DIPSOL IZ-C17+.

- Can be applied prior to HE Baking
- Blue bright coating/ excellent corrosion resistance.
- Ambient Temperature Bath



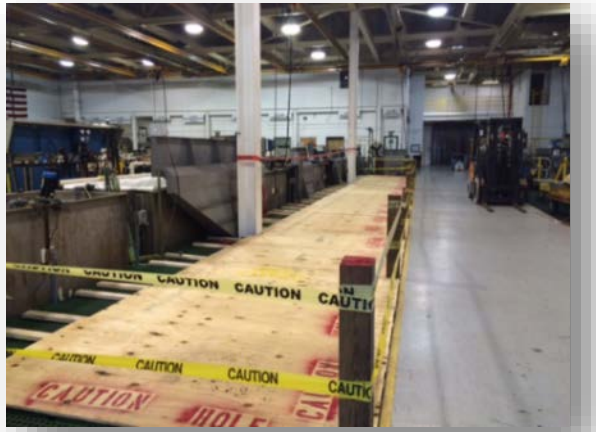
Technical Progress: ONR RIF (Phase III)

Completed September 2017



- Installed new Zn-Ni plating line into FRCSE industrial facility.
- Plating line passed all quality control testing
- Demonstration/Validation successfully completed on aircraft demo parts
- Operator training completed
- Phase III RIF was successfully completed on on 30 September 2017.

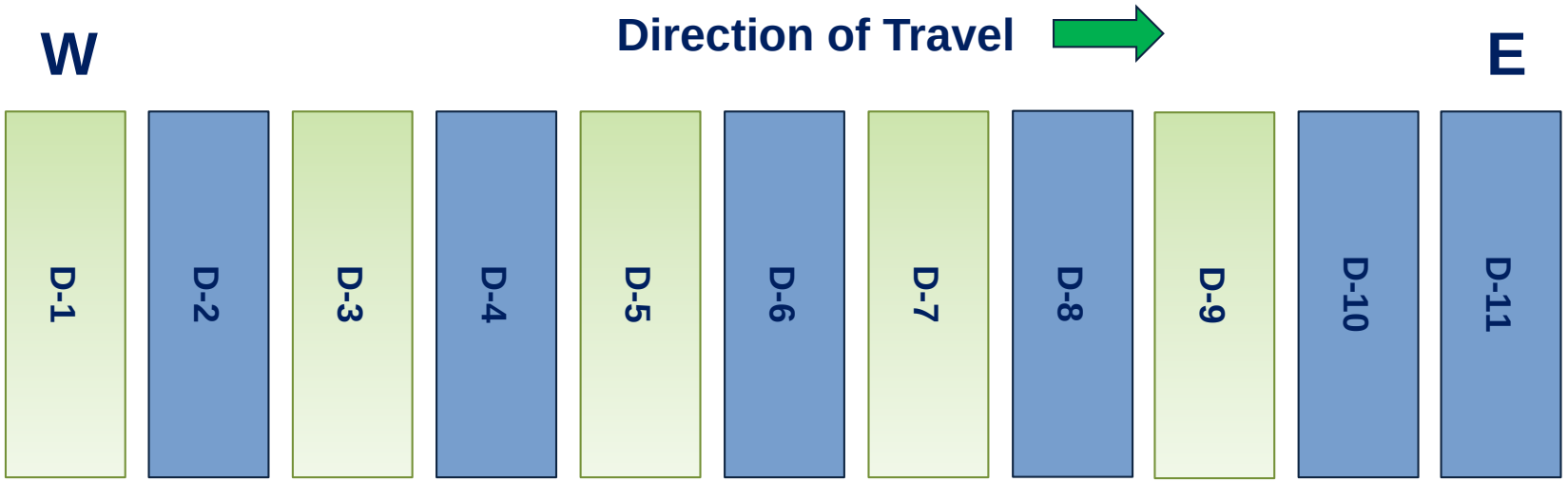
Original State



Under Construction

Line Complete

Process Line Layout - FRCSE



Tank No.	Description	Tank No.	Description
D-1	Electrocleaner	D-7	Dilute Acid (HCL)
D-2	Rinse	D-8	Rinse
D-3	Acid Activation	D-9	IZ-264 Post Treat
D-4	Rinse	D-10	Rinse
D-5	IZ-C17+ Zn-Ni	D-11	Hot Rinse
D-6	CC* Rinse		

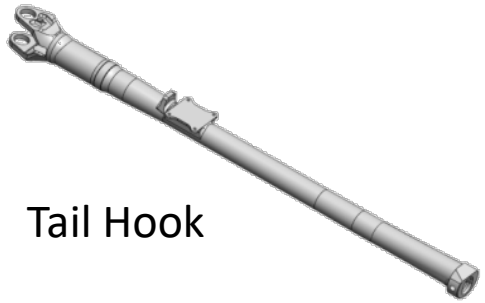
** Counter Current*

Process Tanks

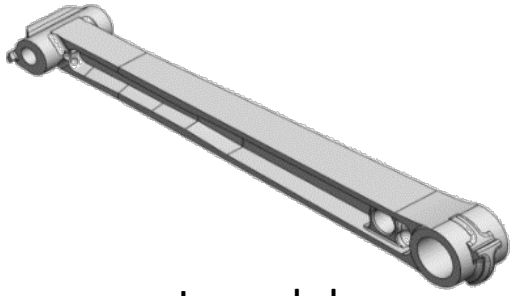
Rinse Tanks

Component Plating Demonstration

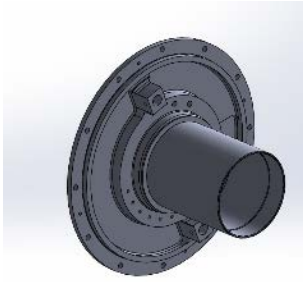
Aircraft Components & Support Equipment



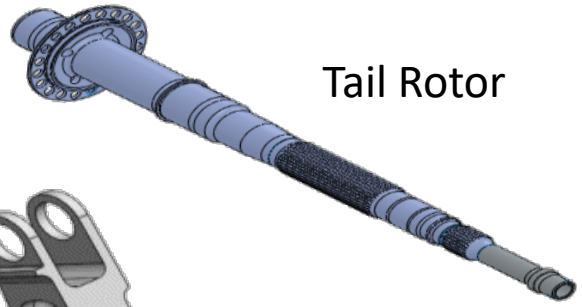
Tail Hook



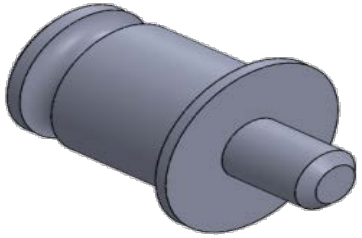
Launch bar



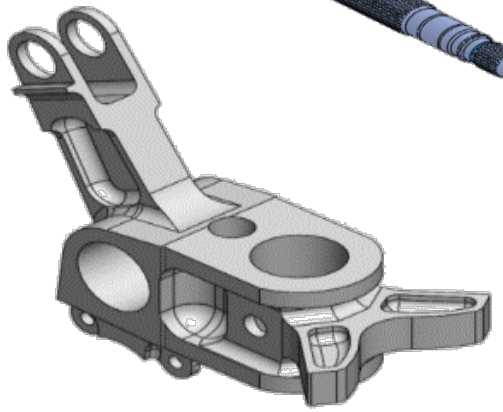
ARS Back Plate



Tail Rotor



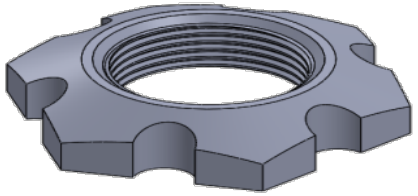
Dolly pin



Tail Hook Pivot

Assorted bushings

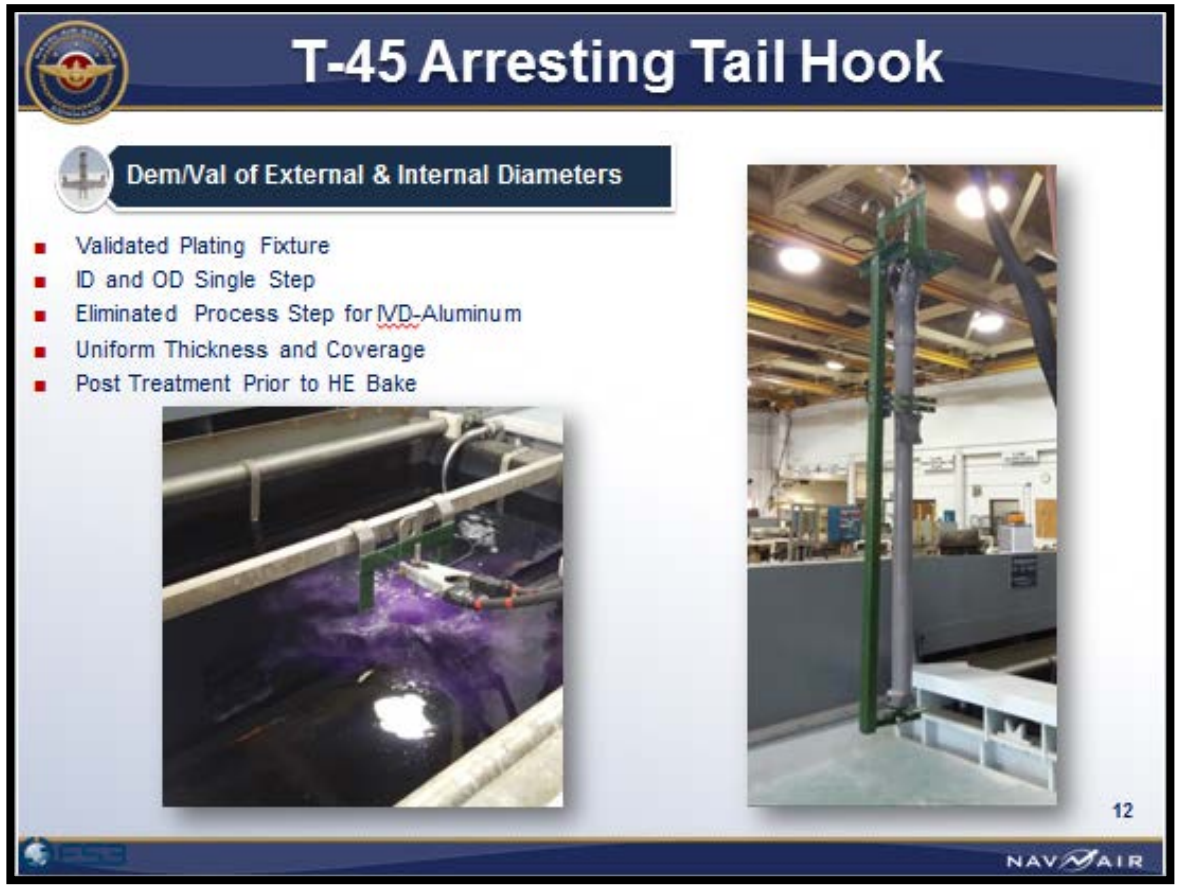
C-130 Propeller Bushing



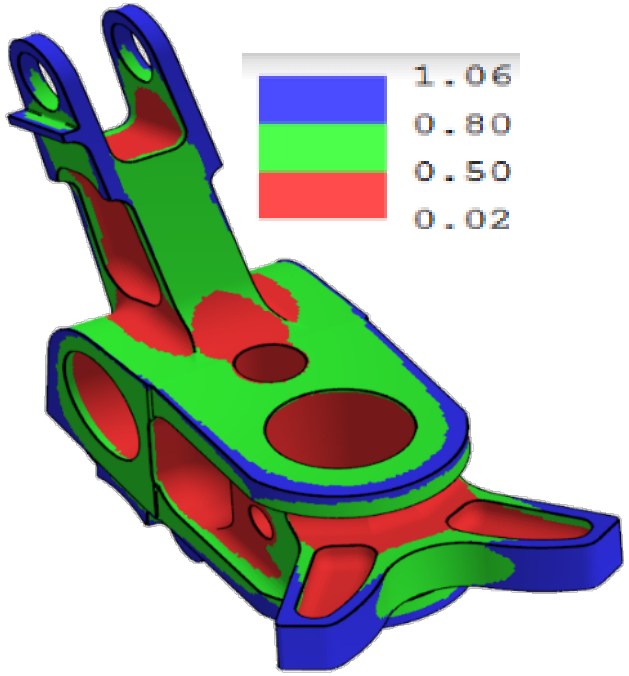
Anchor nut

Clevis pin

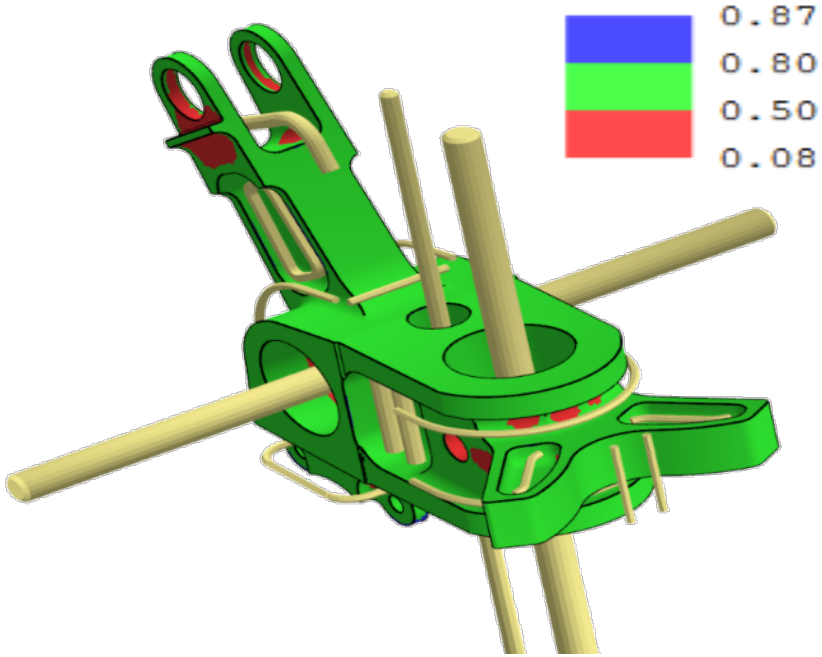
Component Plating Demonstration

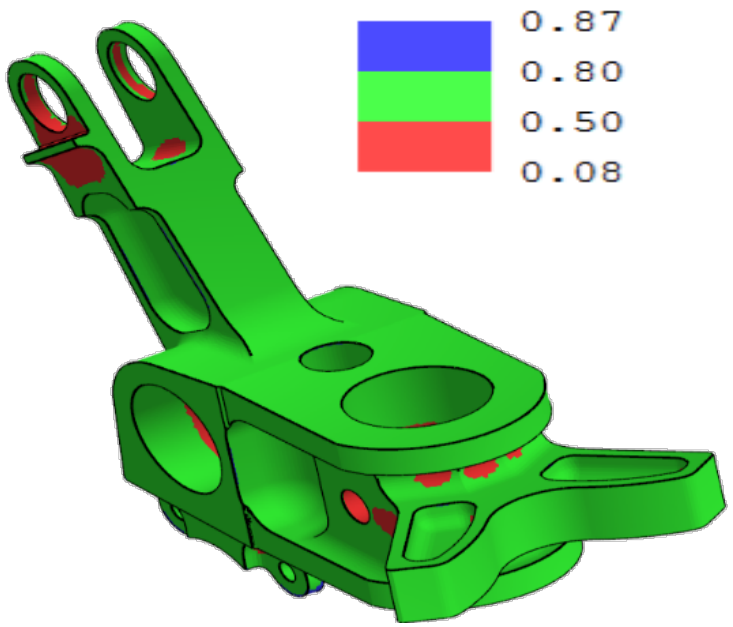


Technical Progress: Anode Design



Plated with no conforming anode



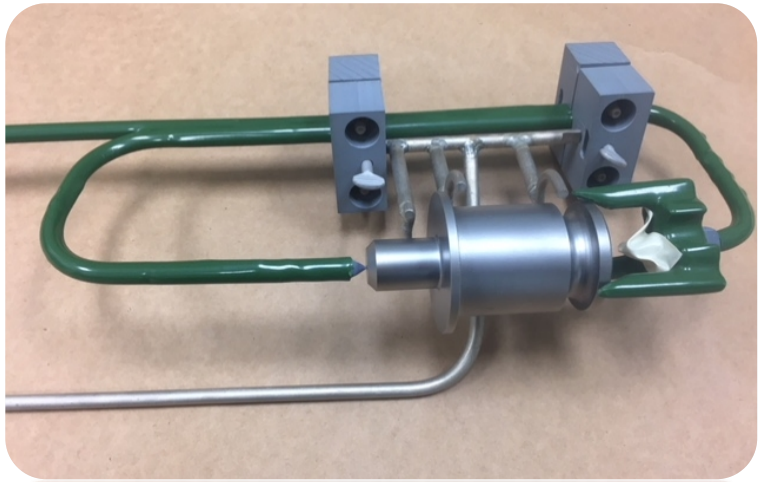


Plating result with conformal anode

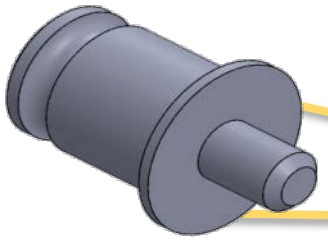
Conformal Anode Design
Simulated Zn-Ni Layer Thickness Distribution Over a Tail Hook Pivot

LHE Zn-Ni – Spotting Dolly Pin Dem/Val

- CVN-75 Demo with (6) Spotting Dollies.
- Direct comparison between Cd and LHE Zn-Ni
- Electroplated at FRCSE plating facility
- Installed June 2017

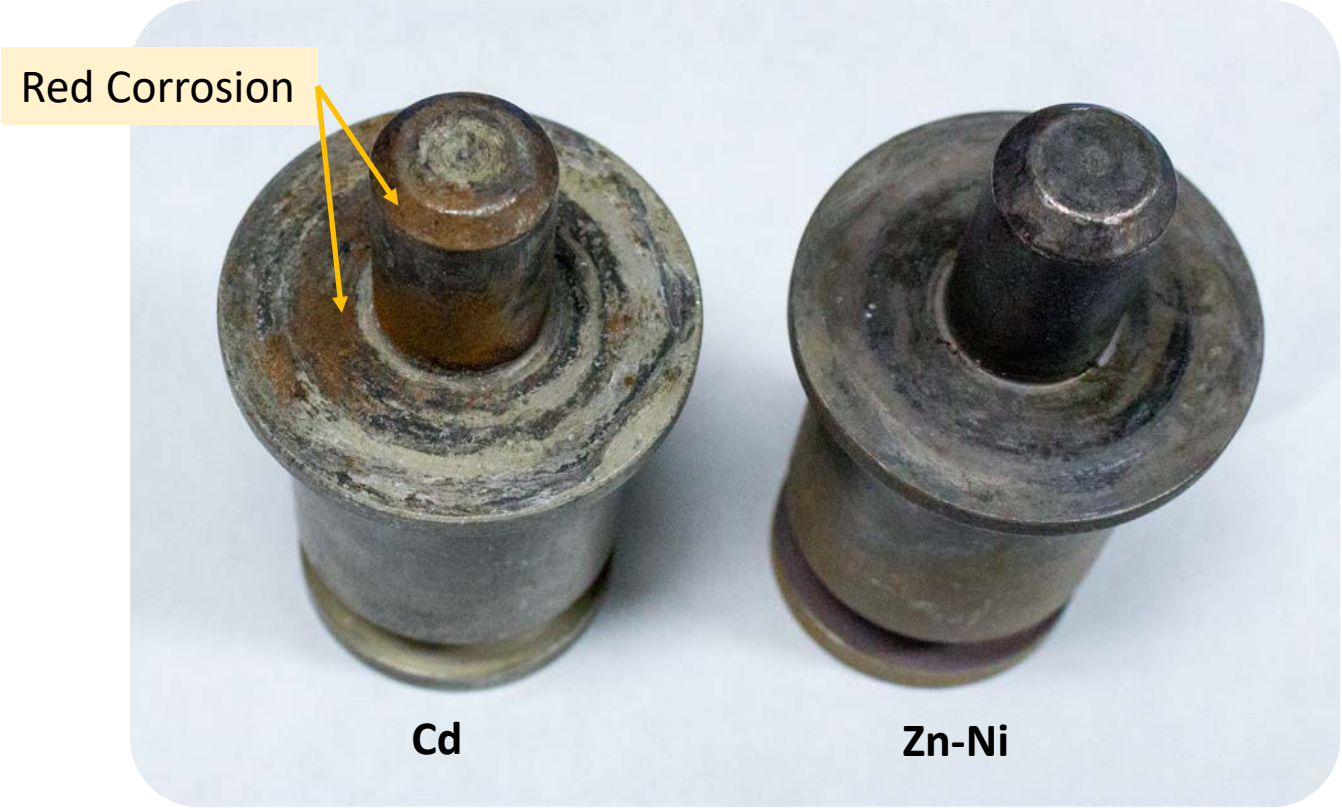


Plating Fixture



Dolly Pin

LHE Zn-Ni – Spotting Dolly Pin Dem/Val



180-day Inspection Results (Jun 17 - Jan 18)

NAVAIR Test Protocol

30 Core Certification Tests

Design of Experiment

- Temperature
- Composition
- Current Density

Product Quality

- Adhesion
- Thickness
- Composition

Corrosion Performance

- B117
- Cyclic
- KSC Beach

Processing & Wear

- NDI Sensitivity
- Gravelometery
- Break Away Torque

Mechanical Properties

- HE
- Fatigue
- SCC
- Ti Compatibility

Expansion

- Low Temp HE Bake
- IWTP
- Rapid HE
- Selective Repair
- Alloy Activation
- Galling

NAVAIR HSS Certification Test Protocol – SBIR Phase II.5 (DEC 2018)

NAVAIR

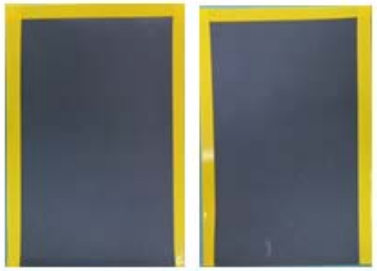
ES3

Design of Experiment (DOE)

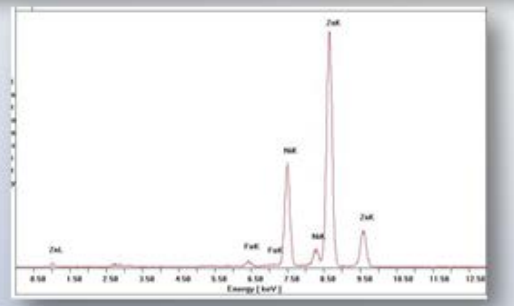
Design of Experiment (DOE)

Certification Test Plan

Zn/Ni Ratio	Temperature (°F)	Current Density (ASF)
5.4	78	56
8.2	78	56
8.2	78	28
8.2	68	28
5.4	78	28
8.2	68	56
5.4	68	28
5.4	68	56



Zn-Ni Plated Specimens



HE Testing

21

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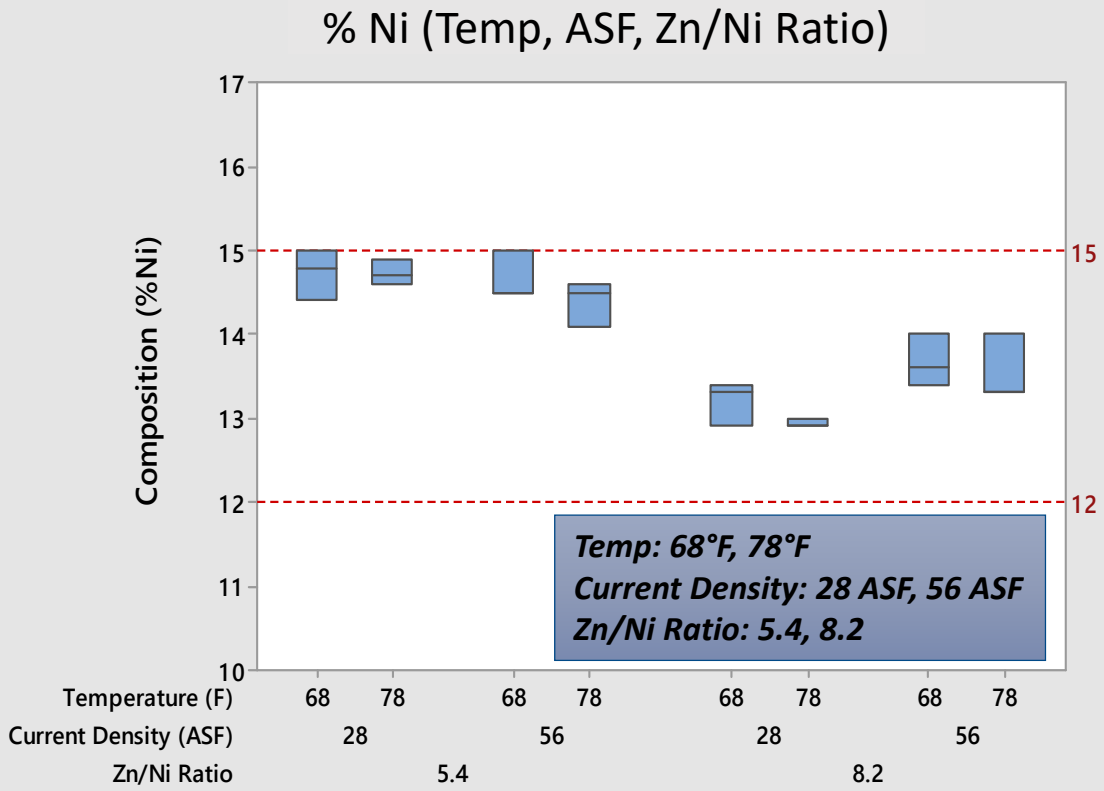

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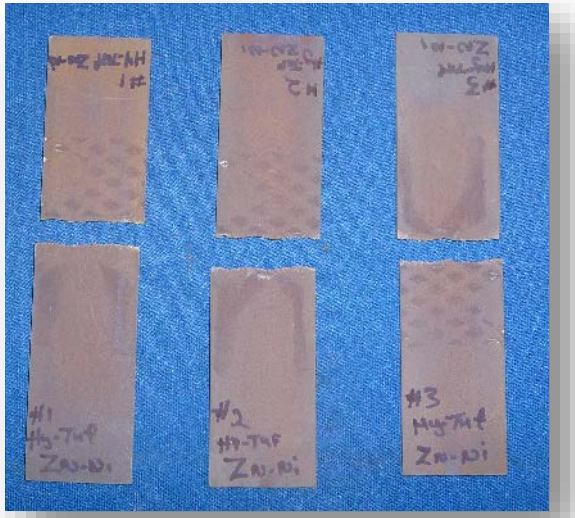
ES3

Adhesion Testing

Bend to Break Test



Zinc-Nickel on Hy-Tuf:

PASS

Cadmium on Hy-Tuf:

PASS

Bend-to-Break		
Material	**Cd**	**Zn-Ni**
4130 steel	Pass	Pass
Hy-Tuf	Pass	Pass
15-5 PH*	Pass	Pass
Al-Ni-Bronze*	Pass	Pass
Al-Bronze*	Pass	Pass
13-8*	Pass	Pass

Developed non-standard pre-plate etch using normal plating line chemicals (No nickel strike used)

Wet Tape		
Material	**Cd**	**Zn-Ni**
4130 Steel (Primer, Non-Chromated)	Pass	Pass
4130 Steel (Primer, Chromated)	Pass	Pass

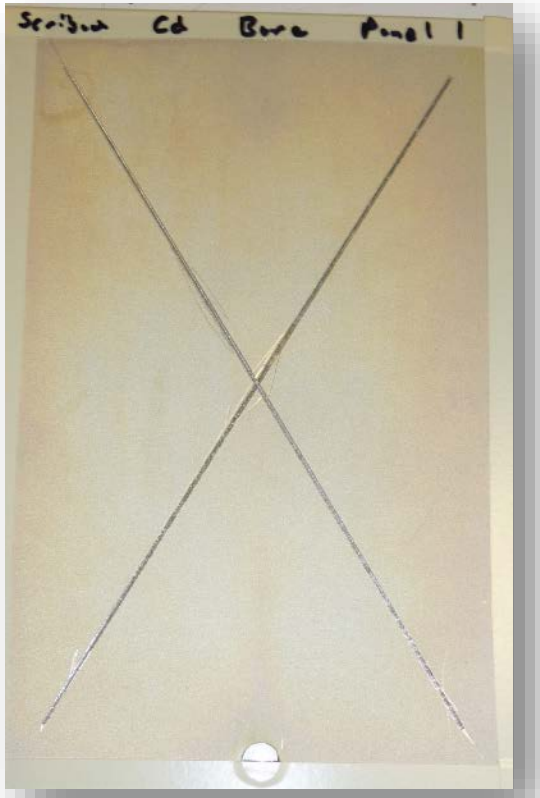
** Different activation process*

ASTM B117 Corrosion Testing

Unpainted/Scribed Panels

Before Test

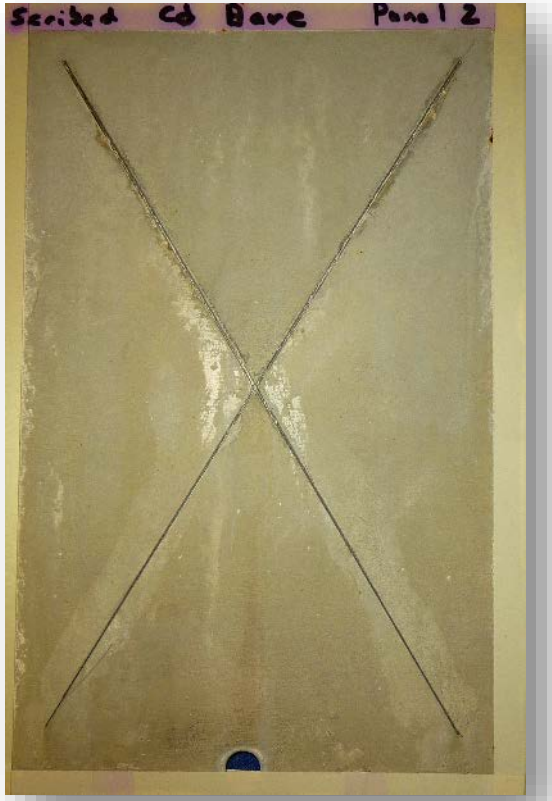
Zinc-Nickel
Pre-test



Cadmium
Pre-test

500 hr - Interim Results

Zinc-Nickel
500 Hrs



Cadmium
500 Hrs

ASTM B117 Corrosion Testing

Primed (Class N) & Painted/Scribed Panels

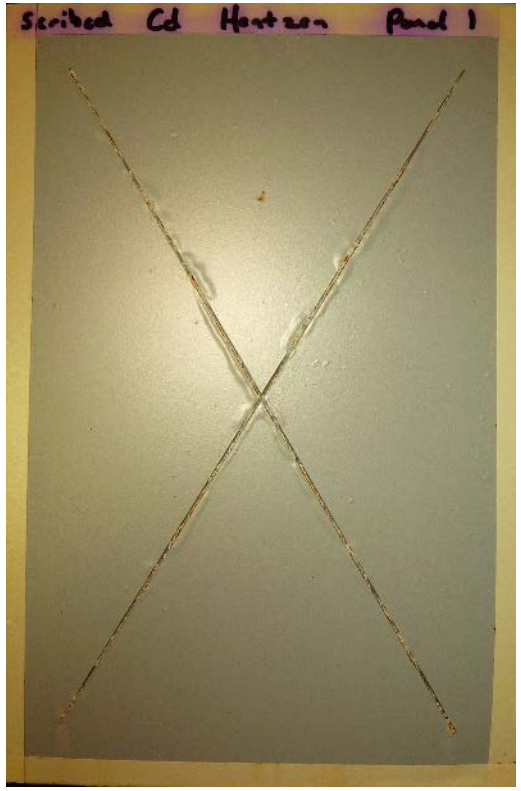
Before Test

Zinc-Nickel
Pre-test

Cadmium
Pre-test

500 hr - Interim Results

Zinc-Nickel
500 Hrs



Cadmium
500 Hrs

ASTM B117 Threaded Surface Testing

Scribed Across Threads

Zinc-Nickel

Cadmium

Before Test

Zinc-Nickel
2500 hrs,
no red corrosion

Zinc-Nickel
4100 hrs,
no red corrosion

Cadmium
2500 hrs,
coating failed

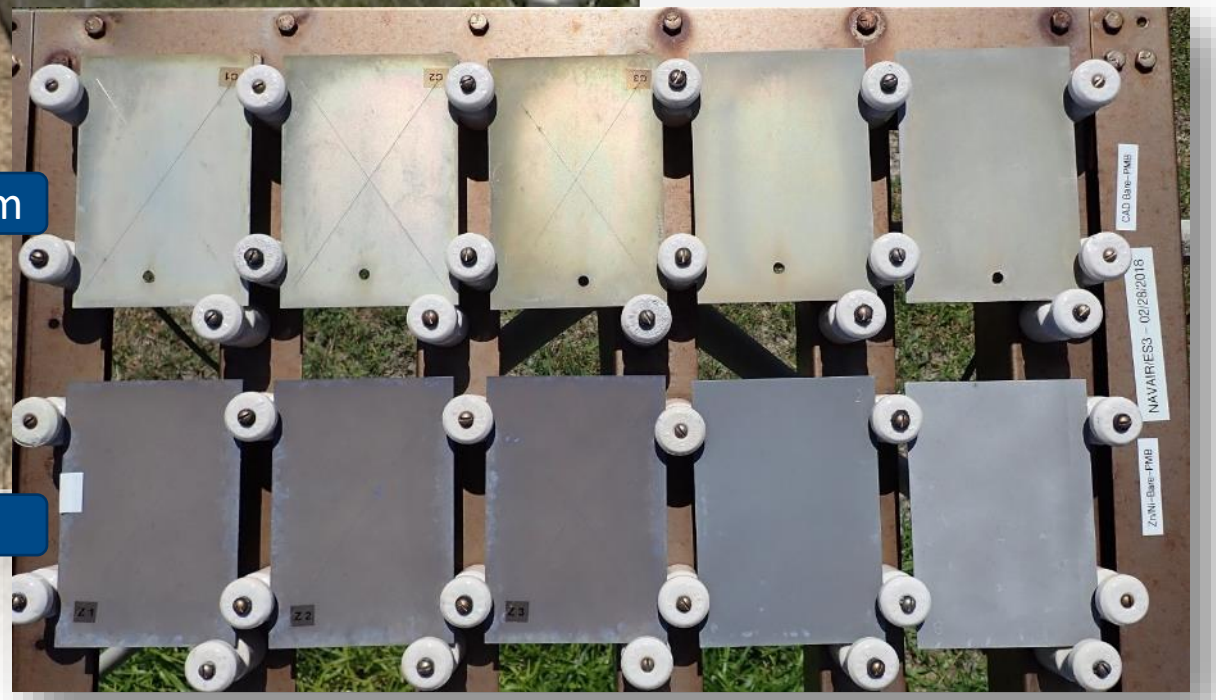
KSC Beach Initial Exposure Corrosion Testing

Beach Site
4 Months

4-months Exposure

Cadmium

Zn-Ni



Unpainted/Scribed

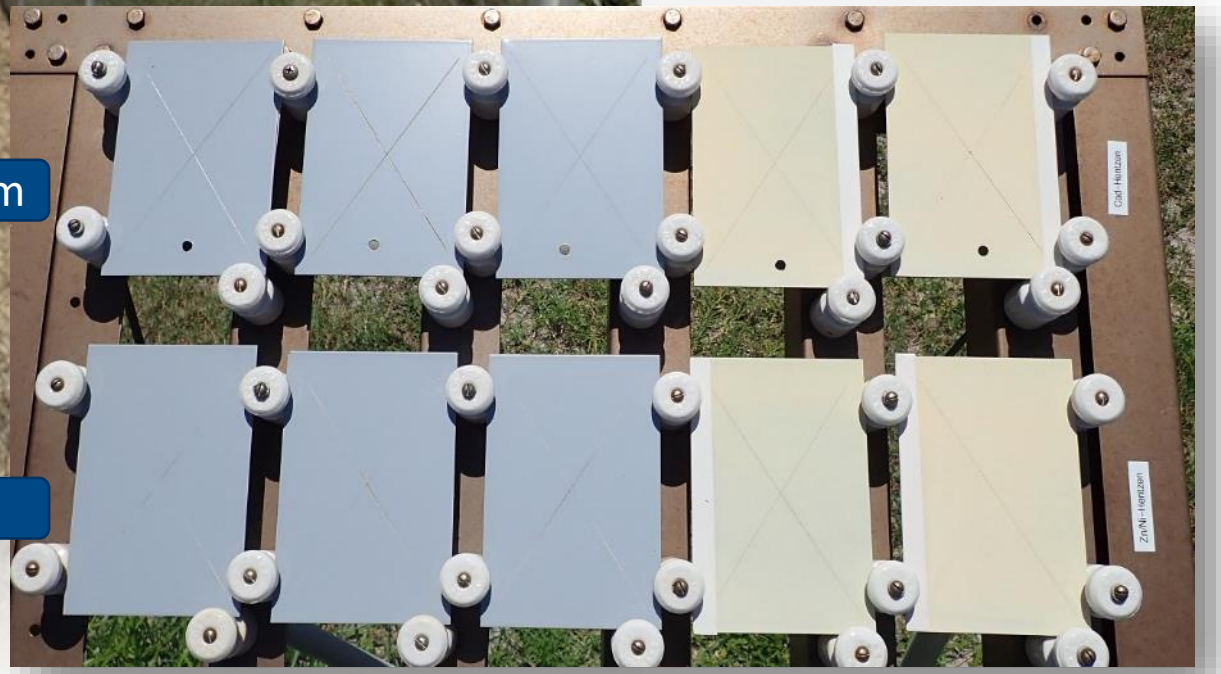
KSC Beach Initial Exposure Corrosion Testing

Beach Site
4 Months

Cadmium

Zn-Ni

4-months Exposure



Painted/Scribed

NRL Key West Exposure Corrosion Testing

Atmospheric Rack at 3 Months

Unpainted/Scribed

NRL Key West Exposure Corrosion Testing

Spray Rack at 3 Months

Unpainted/Scribed

Embrittlement Testing

Coating	Tested	Pass	Fail
Bare	5	5	0
Cd	5	5	0
Zn-Ni	5	5	0

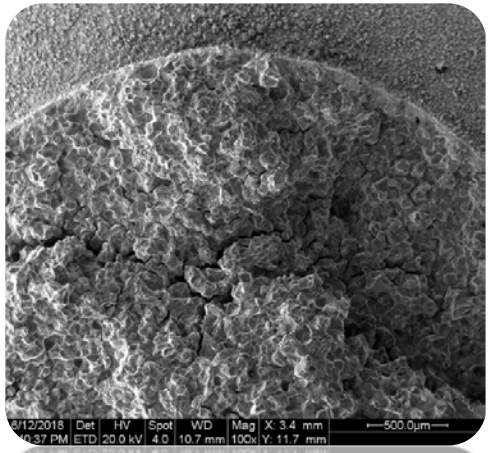
ASTM F519 Hydrogen Embrittlement 200-hr test

Solid Metal Embrittlement Coupons Mounted in Test Fixtures

Coating	Hours at 75% NFS	Result
Cd	8	Fail
Cd	8	Fail
Cd	8	Fail
Zn-Ni	200	Pass
Zn-Ni	200	Pass
Zn-Ni	200	Pass

Solid Metal Embrittlement 300M Coupons 200-hr test @ <u>500°F</u>

Solid Metal Embrittlement Fixture



SEM view of Cd coupons failure

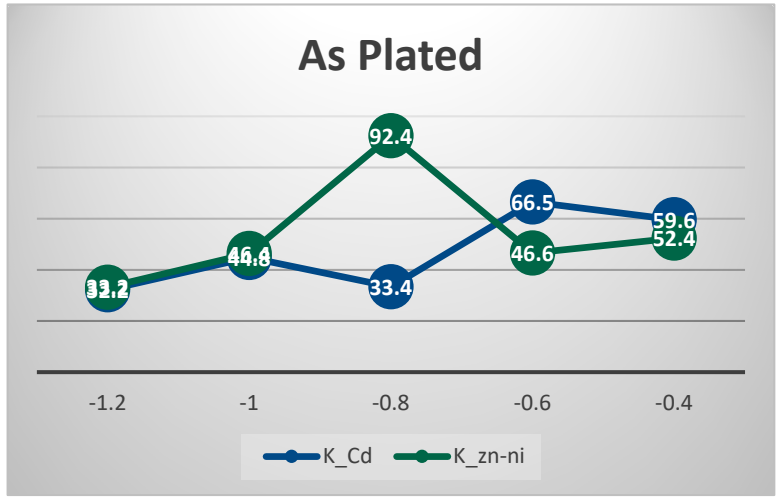
Titanium Embrittlement Coupons

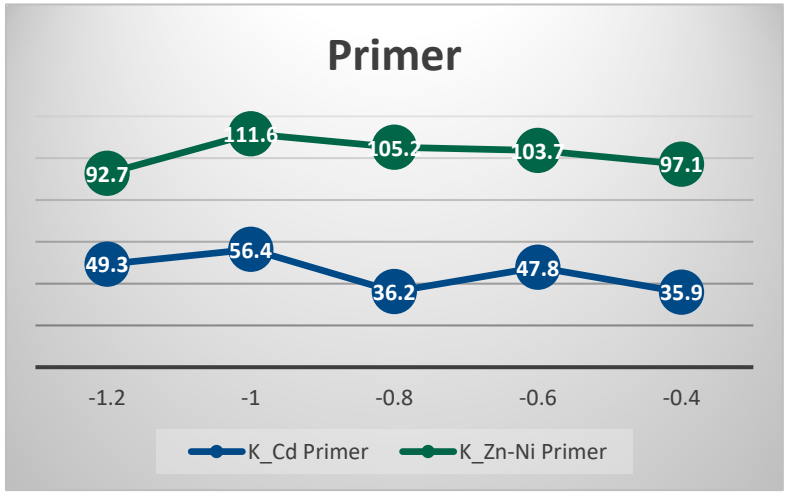
NAVAIR

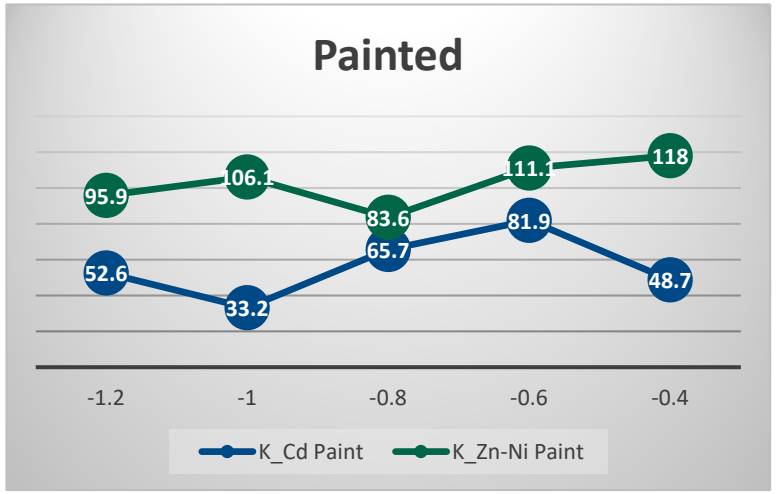
ES3

Stress Corrosion Cracking Testing

Coupon Material - 4340 (260-280ksi)

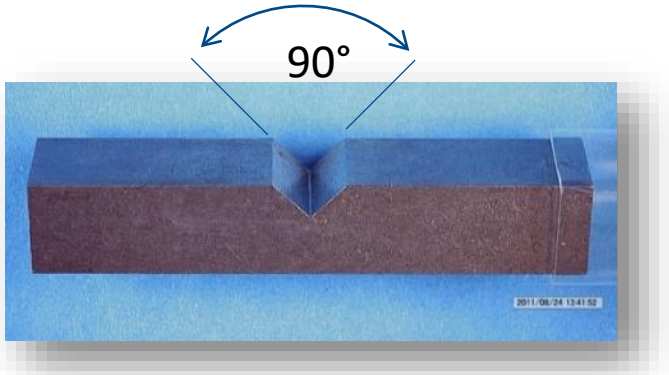






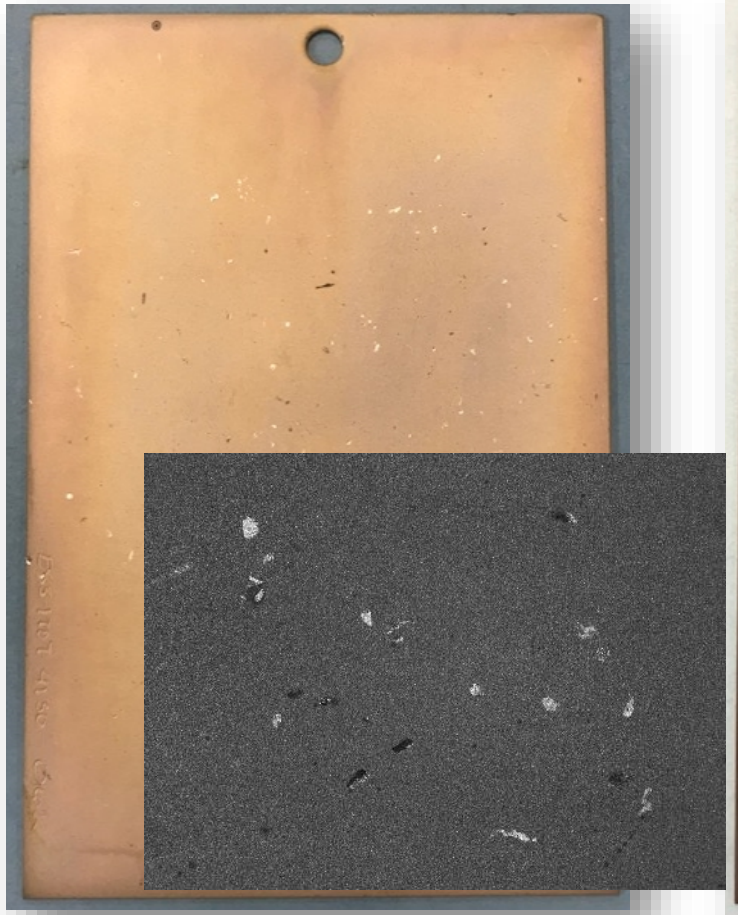
Test Details:

- 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_{SCE} –vs- K_{OSCC} (ksi-√in)

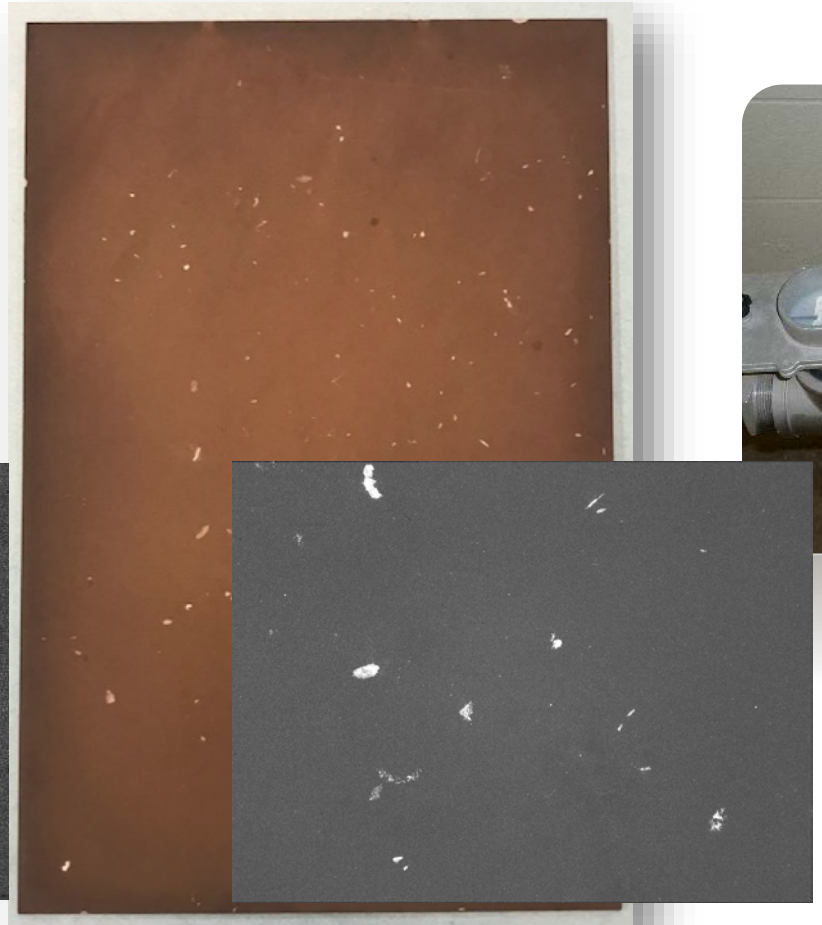


Type 1.e Notched Square Bar
52-54 HRC

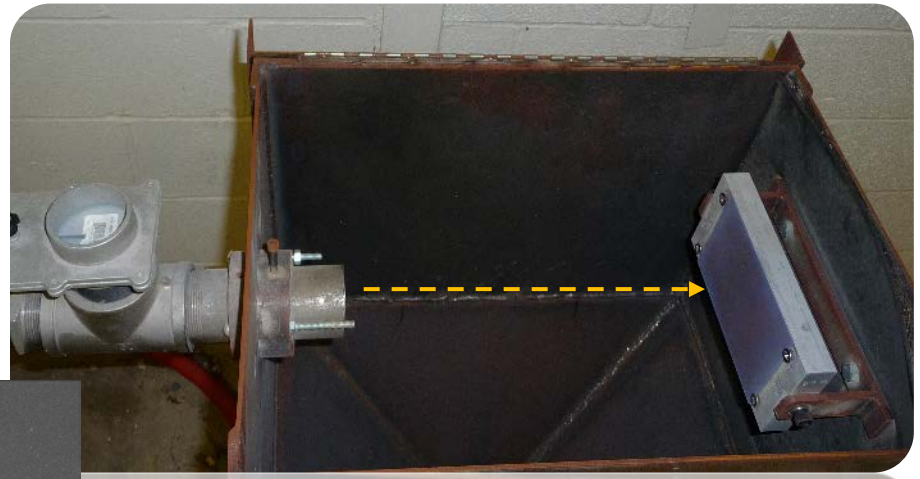
Gravelometry Testing



Cadmium Post-test



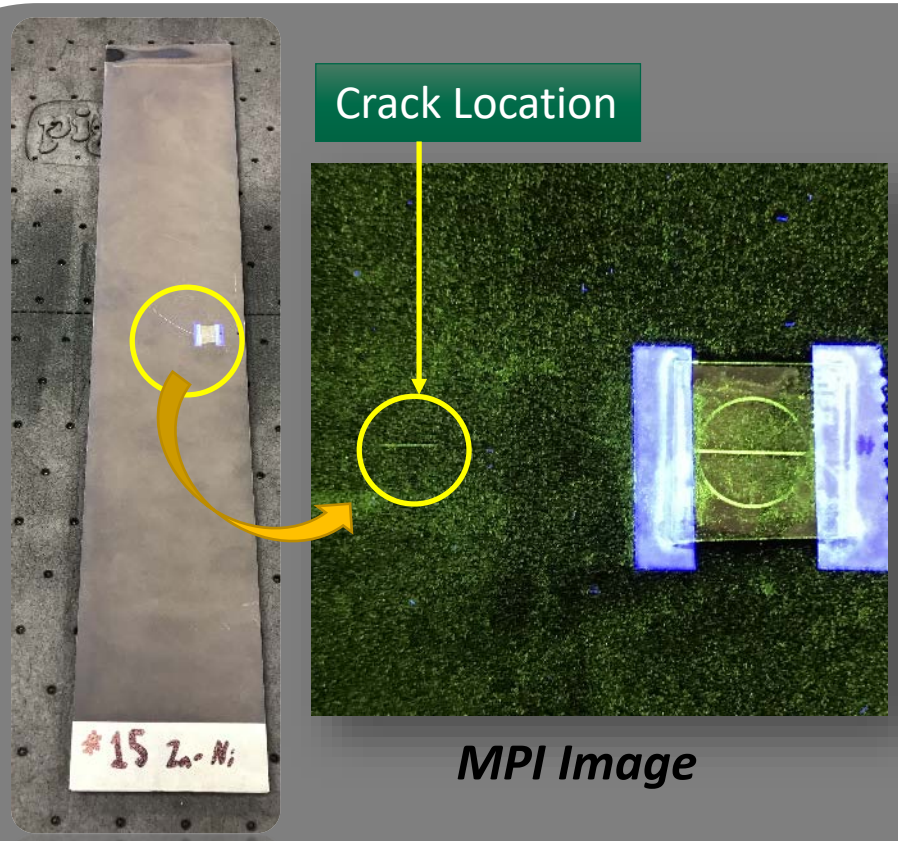
Zinc-Nickel Post-test



Zn-Ni coupon mounted in Test Apparatus

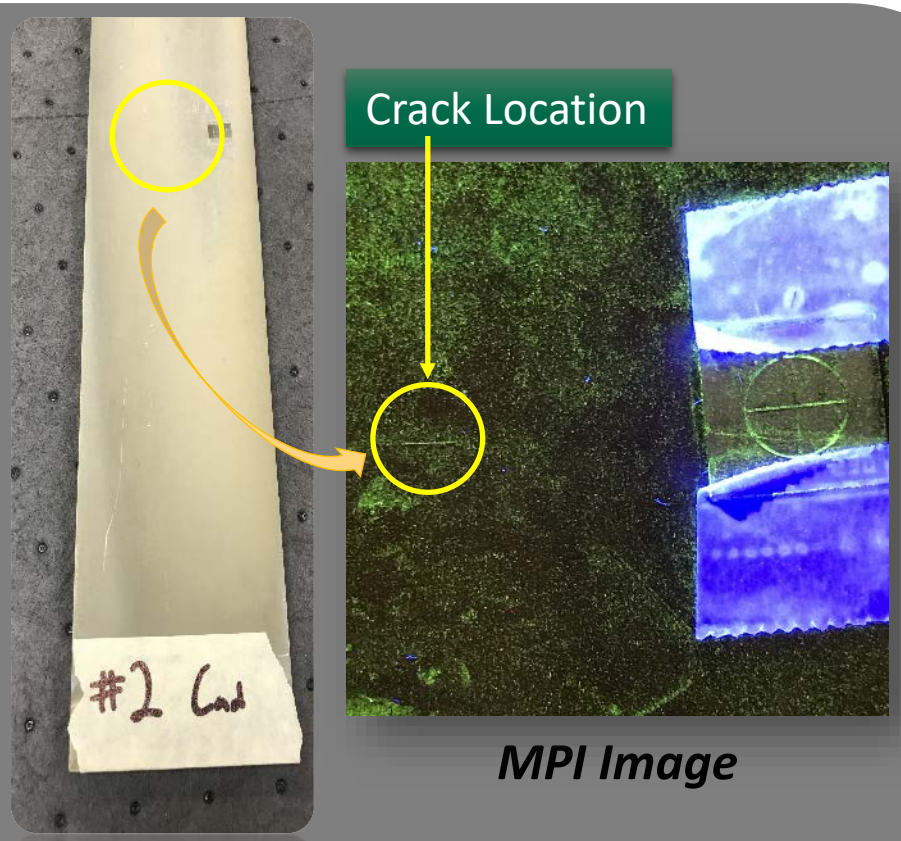
Gravelometry Test Apparatus (IAW ASTM D3170)

Non-Destructive Testing



MPI Image

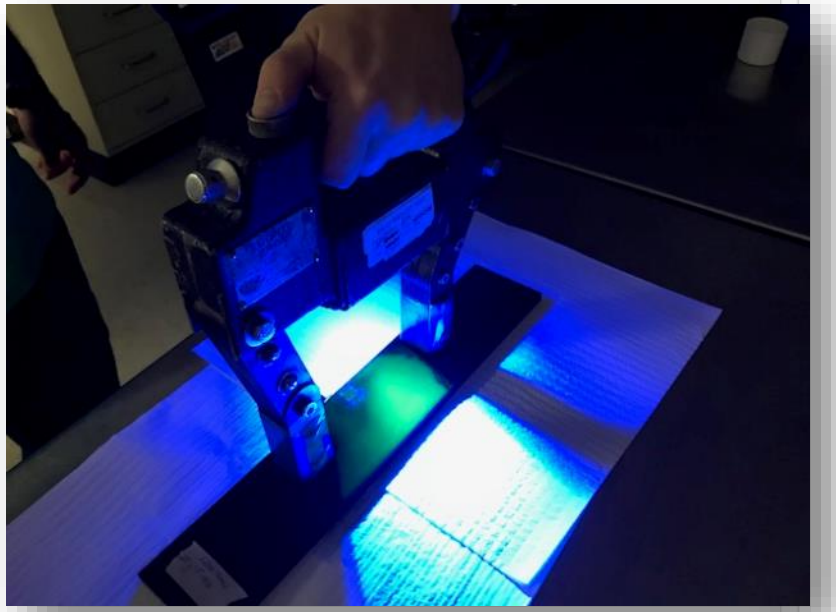
Zinc-Nickel



MPI Image

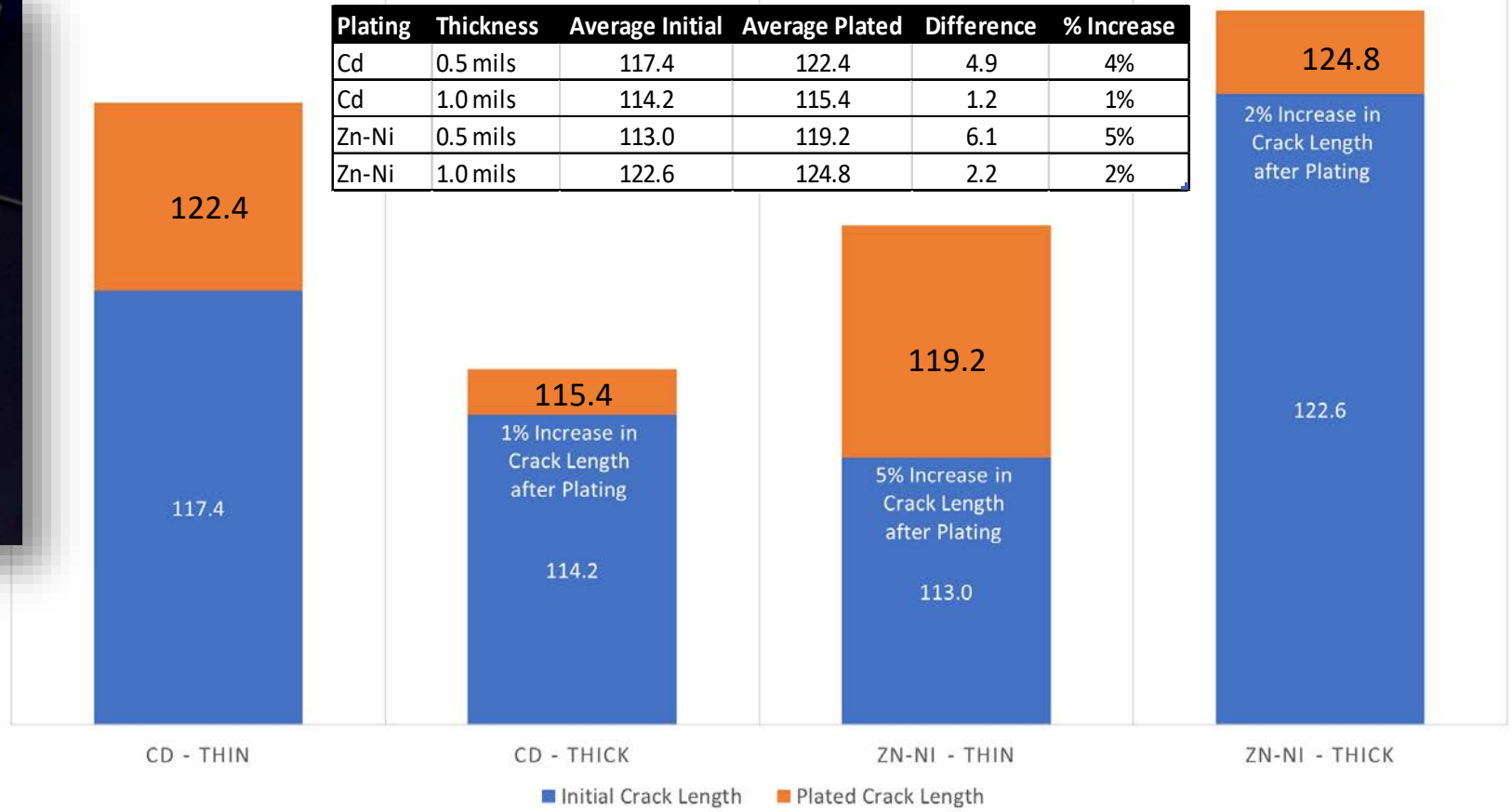
Cadmium

Non-Destructive Testing



Magnet Particle Inspection of a Zn-Ni plated NDI bar

Plating	Thickness	Average Initial	Average Plated	Difference	% Increase
Cd	0.5 mils	117.4	122.4	4.9	4%
Cd	1.0 mils	114.2	115.4	1.2	1%
Zn-Ni	0.5 mils	113.0	119.2	6.1	5%
Zn-Ni	1.0 mils	122.6	124.8	2.2	2%



Axial Fatigue Test Matrix

Coupon Material - 4340 0.25" Dia (260-280ksi) - Unpeened

Coupons Description	Material	Stress Levels (R = 0.1 @ 5 hz)					Total
		160	170	180	190	200	
Bare	4340	6	6	6	6	6	30
Zn-Ni	4340	6	6	6	6	6	30
Cd	4340	6	6	6	6	6	30
						Total	90

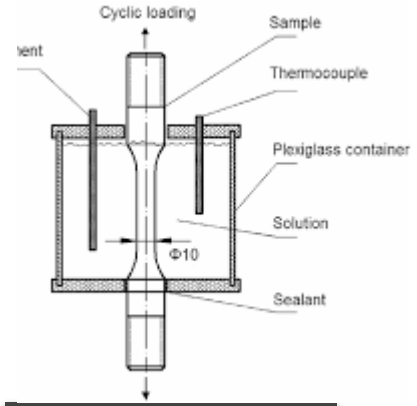
Coupons Description	Material	Stress Levels (R = -1 @ 5 hz)					Total
		160	170	180	190	200	
Bare	4340	6	6	6	6	6	30
Zn-Ni	4340	6	6	6	6	6	30
Cd	4340	6	6	6	6	6	30
						Total	90

Samples in final machining

Corrosion Fatigue Test Matrix

Coupon Material - 4340 0.25" Dia (260-280ksi)

Coupons Description	Material	Stress Levels (R = 0.1)					Fluid	Total
		160	170	180	190	200		
Bare	4340	6	6	6	6	6	3.5% NaCl solution (pH 7.3)	30
Zn-Ni	4340	6	6	6	6	6	3.5% NaCl solution (pH 7.3)	30
Cd	4340	6	6	6	6	6	3.5% NaCl solution (pH 7.3)	30
							Total	90



Samples in final machining

MIL-DTL-5002 Restrictions

3.6.1.1 Cadmium coatings shall not be used for the following applications:

a. Parts that may be in contact with hydraulic fluids, fuels, lubricating oil, and other petroleum based fluids.

b. Parts in frictional contact where gouging or binding may be a detrimental factor.

c. In confined spaces, in the presence of organic materials that give off corrosive or damaging vapors.

d. On titanium parts or fasteners. In addition, cadmium plated parts, including interference-fit fasteners and press-fit bushings, shall not be used in contact with titanium. Under certain conditions, titanium can be embrittled by contacting cadmium components. Embrittlement has been determined to occur in cadmium plated titanium parts at temperatures as low as 150° F.

e. Parts that will be subsequently soldered.

f. Components that will come into contact with graphite composites.

g. Parts that will be exposed to temperatures above 450° F (232° C).

NOT MEASUREMENT SENSITIVE

MIL-DTL-5002E
12 July 2011
SUPERSEDING
MIL-S-5002D
30 November 1989

DETAIL SPECIFICATION

SURFACE TREATMENTS AND INORGANIC COATINGS FOR METAL SURFACES OF WEAPONS SYSTEMS

Reactivated after 12 July 2011 and may be used for new and existing designs and acquisitions.

Expanded effort addresses additional cadmium use restrictions

NAVAIR

ES3

NAVAIR Integration Strategy

Industrial Capability

Process Line Installation

Complete

Process Specification

Detail processing requirements

LPS 4XX

90 % Draft

Military Specification

Performance Requirements

80 % Draft

MIL-DTL-5002

Design Authority

Process Restrictions

Pending Test Protocol

Authorization

NAVAIR Authorization for High Strength Steel Applications

Pending Test Protocol

NAVAIR

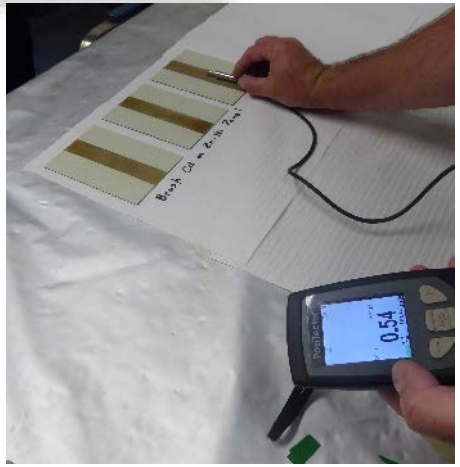
ES3

SELECTIVE ELECTROPLATING

WESTPAC Brush Electroplating Certification Training of Marine Aviation Logistics Squadrons (MALS).

Top concern was process safety and toxicity of cadmium.

SELECTIVE ELECTROPLATING



ASTM B-117 Salt Fog Corrosion Test
1000 hours exposure

Cd brush over Cd plate

Zn-Ni brush over Cd plate

Cd brush over Zn-Ni Plate

Zn-Ni brush over Zn-Ni plate

SELECTIVE ELECTROPLATING

As technology matures increased emphasis is necessary for selective repair using brush electroplating techniques.

NAVAIR SBIR Phase II.5 - ES3

- Evaluation of COTS Brush Zinc-Nickel Solutions (Expansion Effort)
  Topic: AF071-320

NAVAIR SBIR Phase II.5 – Corrdesa

- Improved Hardware and optimized processing (Dalistick & Modeling)
  Topic: N112-154

USAF SBIR Phase I – ES3

- Developing and evaluating new brush electroplating formulations
  Topic: AF172-002

SERDP/ESTCP

- Chemicals
- Process
- Tooling
- Hardware

Coordinated Effort Required

Questions

Jack Benfer
Ruben Prado
Julia Russell
Heather Stoll
Atish Gupta
Robert Kestler

NAVAIR

Jay Randolph
Fred Laguines
Tod Hickman

ES3

Quality Testing

Plating Quality

- Adhesion
- Thickness
- Plating Properties
- Repairability

Engineering Requirement	Test (s)	Specification	Status
Appearance & Finish Quality	Visual Examination	MIL-STD-870B; AMS-QQ-P-416	PASS
Thickness	X-Ray Spectrometry	ASTM B568 or ASTM E376	PASS
Hardness	Microhardness	ASTM E384	IN TEST
Metallography	As plated and post-heat treat	N/A	IN TEST
Adhesion	Bend-to-Break	ASTM B571	PASS
Adhesion	Wet Tape (Paint Adhesion)	MIL-PRF-85582; ASTM D3359	PASS
Throwing Power	Tube fixture / XRF	MIL-STD-870B, AMS-QQ-P416	PASS
Repair compatibility	Bend Adhesion	ASTM B571	PASS
Repair compatibility	Paint Adhesion	ASTM D3359	PASS

Corrosion Performance Testing

Corrosion Performance

- Salt Fog
- Cyclic
- Long term Outdoor
- Repairability

Engineering Requirement	Test (s)	Specification	Status
Corrosion	Salt Fog	ASTM B117	Completed
Cyclic Corrosion	Salt Fog (Cyclic)	GMW14872	In Test
Threaded component	Salt Fog	ASTM B117	Completed
Beach Exposure	Long-Term Outdoor	ASTM B537	In Test
Galvanic Corrosion Resistance	Salt Fog	ASTM B 117 JG-PP JTP BD-R-1-1	In Test
Galvanic Potential	Open Circuit Potential		In Test
Fluid Resistance	Fluid Immersion	ASTM F843	Completed
Repair compatibility	Salt Fog	ASTM B117	In Test

Process and Performance Testing

Process and Performance

- Lubricity
- Gravelometry
- NDI Sensitivity
- Strippability

Engineering Requirement	Test (s)	Specification	Status
Lubricity	Run-on/Break-away torque & Extended Reuse	NASM25027	IN TEST
Wear / Durability Test	Gravelometry Test	ASTM D3170	PASS
Strippability	Coater Recommendation or MIL-S-5002	MIL-STD-871	IN TEST
NDI Sensitivity Analysis	Magnetic Particle Testing	ASTM E1444	PASS
NDI Sensitivity Analysis	Eddy Current Testing	ASTM E566 or equivalent	PASS

Structural Mechanical Properties Testing

Mechanical Properties

- HE
- Fatigue
- SCC
- Ti Compatibility

Engineering Requirement	Test (s)	Specification	Status
Hydrogen Embrittlement	Hydrogen Embrittlement	ASTM F519	PASS
Hydrogen Re-embrittlement	Hydrogen Re- Embrittlement	ASTM F1624	PASS
Upper Use Temp	Liquid Metal/Solid Metal Embritttlement	ASTM F519	PASS
Titanium Embrittlement Test	Hydrogen Embrittlement	ASTM F945	IN TEST
SCC 1e Cathodic Overpotential	Stress Corrosion Testing	ASTM F519	IN TEST
Corrosion Fatigue	Axial Fatigue	ASTM E466	IN TEST
Fatigue	Axial	ASTM E466	IN TEST
Tensile Test	Tensile Test (Su, Sy, E, %Elong, %RA)	ASTM E8	IN TEST