



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



**U.S. AIR FORCE**

Dave Frederick, USAF  
Craig Pessetto, ES3  
Steve Gaydos, Boeing

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# Report Documentation Page

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# 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 :

# Prototype Tank Implementation

- 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



**Dipsol IZ-C17+ LHE Zn-Ni Plating Tank**

# Prototype Tank Implementation



**Tri-Chromium Conversion Coat Tank**

*www.ES3inc.com • 1669 E. 1400 S • Clearfield, UT 84015  
(801) 926-1150 • fax (801) 926-1155*

# 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



**Adhesion Test Coupons**

# 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

## Scribed Dry and Wet Tape Adhesion Test Results

4" x 6" x 0.040" 4130 Steel Substrate

PANEL	ID	PRETREATMENT	COATING	DRY TAPE ADHESION			WET TAPE ADHESION		
				PERCENTAGE COATING REMOVED	ASTM D 3359 (1)	Pass / Fail (2)	PERCENTAGE COATING REMOVED	ASTM D 3359 (1)	Pass / Fail (2)
1		LHE Zn-Ni Plating	Deft 44-GN-72	0	5A	Pass	0	4A	Pass
2				0	5A	Pass	2	4A	Fail
3				0	5A	Pass	0	5A	Pass
1		Cd Plated w/ Hex Cr Conversion Coating		0	5A	Pass	0	5A	Pass
2				0	5A	Pass	0	5A	Pass
3				0	5A	Pass	0	5A	Pass
4		LHE Zn-Ni Plating	Deft 44-GN-098	0	5A	Pass	0	5A	Pass
5				0	5A	Pass	0	5A	Pass
6				0	5A	Pass	10	4A	Fail
4		Cd Plated w/ Hex Cr Conversion Coating		0	5A	Pass	0	5A	Pass
5				0	5A	Pass	0	5A	Pass
6				0	5A	Pass	0	5A	Pass

**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.

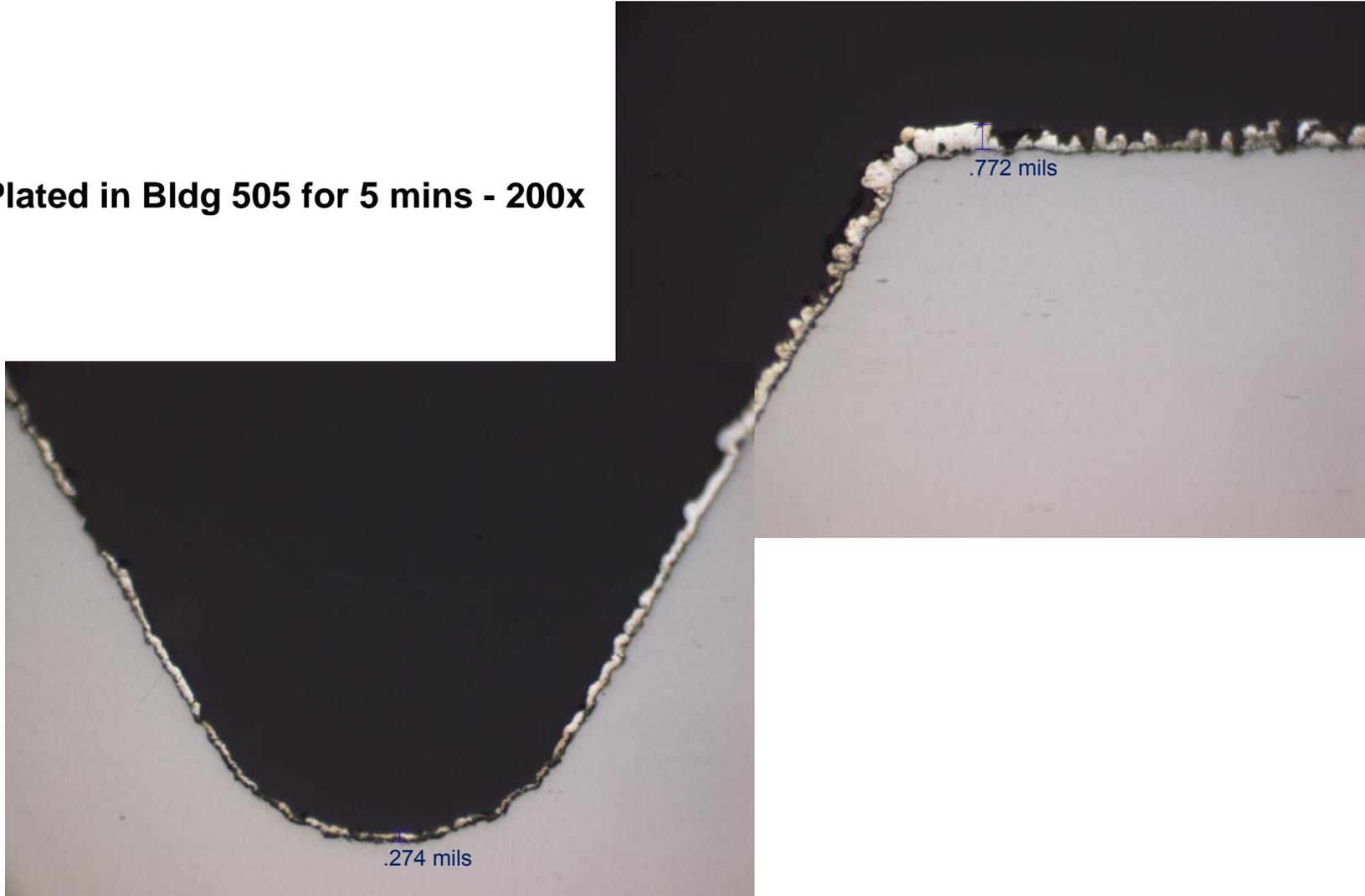
- Coupons manufactured per ASTM F519 specifications (4340)
- Coupons plated and tested 28<sup>th</sup> April, 2009 upon initial installment of LHE Zn-Ni demonstration tank
- Additional coupons plated and tested at additional dates
- All coupons tested per ASTM F519 and passed the 200 hour sustained load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



**ASTM F519 Type 1A.1 Test Coupons**

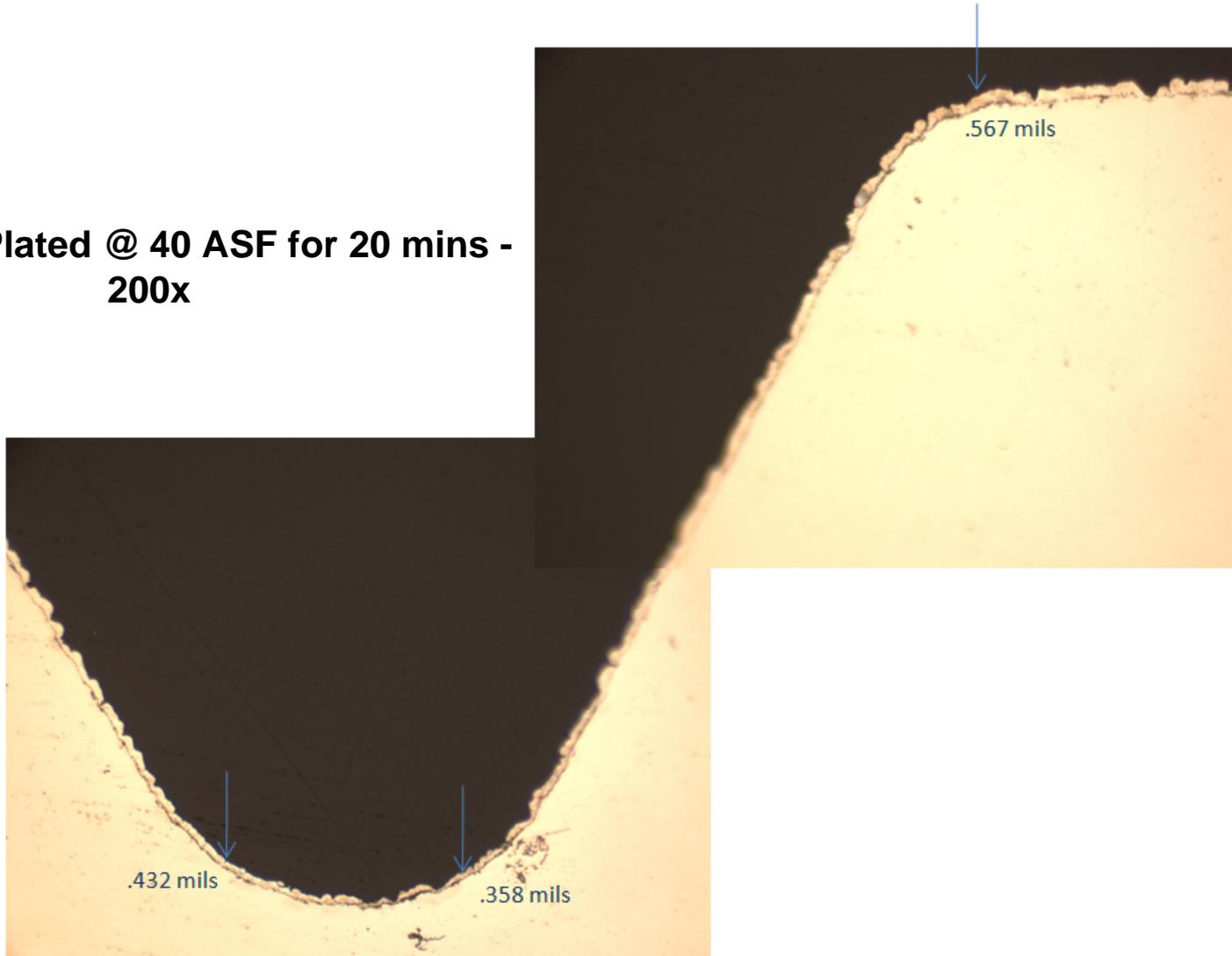
# HE Plated Cross Section

**Cad Plated in Bldg 505 for 5 mins - 200x**



# HE Plated Cross Section

**LHE Zn-Ni Plated @ 40 ASF for 20 mins -  
200x**





# LHE Zn-Ni Hydrogen Re-Embrittlement Testing



Re Embrittlement Test Matrix						
Plating	Test Solution					
	Distilled Water @ Room Temp Tested 45% NFS for 150Hrs	3.5% Salt Water @ Room Temp Tested 45% NFS for 150Hrs	Dwg 9825019* Diluted Calla 296 @ Max Temp 180 °F Tested 75% NFS for 200Hrs	Dwg 9825019* Diluted Calla 602 LF Max Temp 160 °F Tested 75% NFS for 200Hrs	Concentrated Calla 296 @ Room Temp tested 45% NFS for 150Hrs	Concentrated Calla 602LF @ Room Temp tested 45% NFS for 150Hrs
LHE Zn-Ni	Pass	Failed	Pass	Pass	Pass	Pass
Cadmium	Pass	Failed	Pass	Pass	Pass	Pass
IVD	Failed	Failed	Not Tested	Not Tested	Not Tested	Not Tested

\*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:

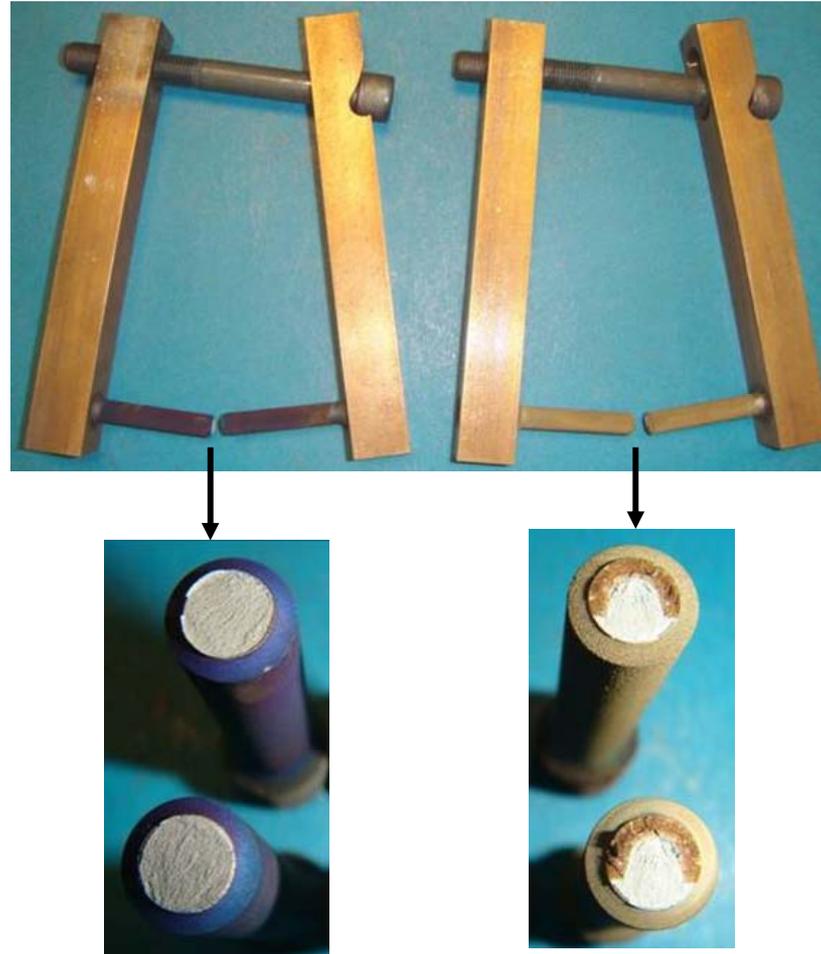
- Coupons tested by an ISO 9001 certified facility. Coupons tested IAW ASTM F519.  
The coupons tested immersed in solutions of Water, 3.5% Salt Water, Dilute\* Calla 296, Dilute\* Calla 602LF, Concentrated Calla 296, and Concentrated Calla 602LF.  
*\*NOTE – Dilute means mix cleaning solution to manufacturer's 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

Temp/NFS	Material	LHE Zn-Ni 200Hr	Cad 200Hr	LHE Zn-Ni Step Load	Cad Step Load
600F/85%	300M	Pass	Fail	100% NFS	-N/A-
500F/85%	300M	Pass	Fail	100% NFS	87% NFS
400F/85%	300M	Pass	Fail	100% NFS	91% NFS
400F/75%	300M	Pass	Fail	100% NFS	81% NFS



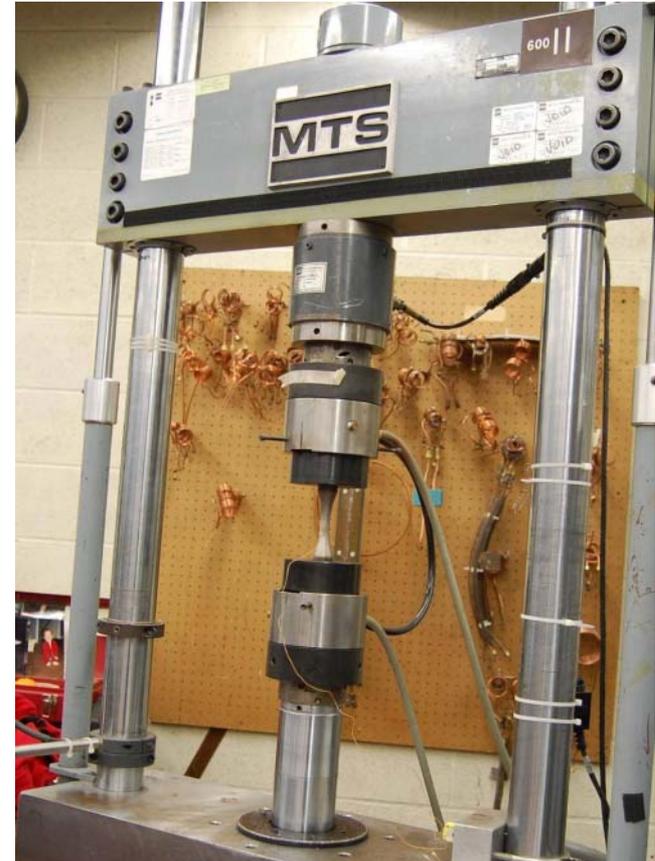
**LHE Zn-Ni and Cad Type 1a.1 Specimens After ISL Test to Determine the NFS After Exposure to 400°F for 200 Hours**

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# Fatigue Testing



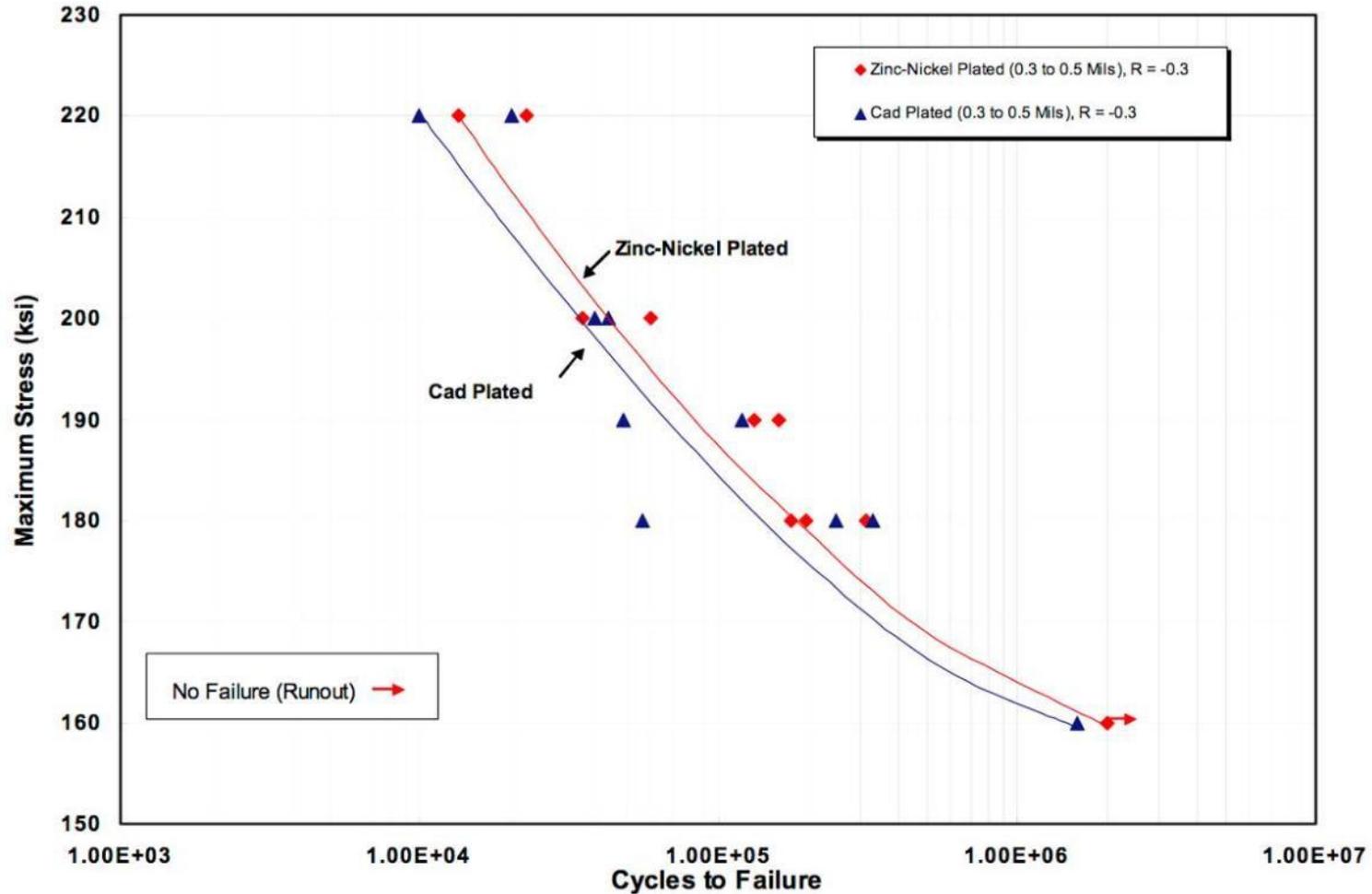
- Phase II LHE Zn-Ni fatigue testing is an extension of Phase I work
- Phase II LHE Zn-Ni fatigue testing continues to broaden the data base and increase the statistical validity of the data
- Manufacturing of coupons and Fatigue Testing IAW ASTM E466
  - All coupons were plated per manufacture's plating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



# Phase I Fatigue Testing (Shotpeened Coupons)



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

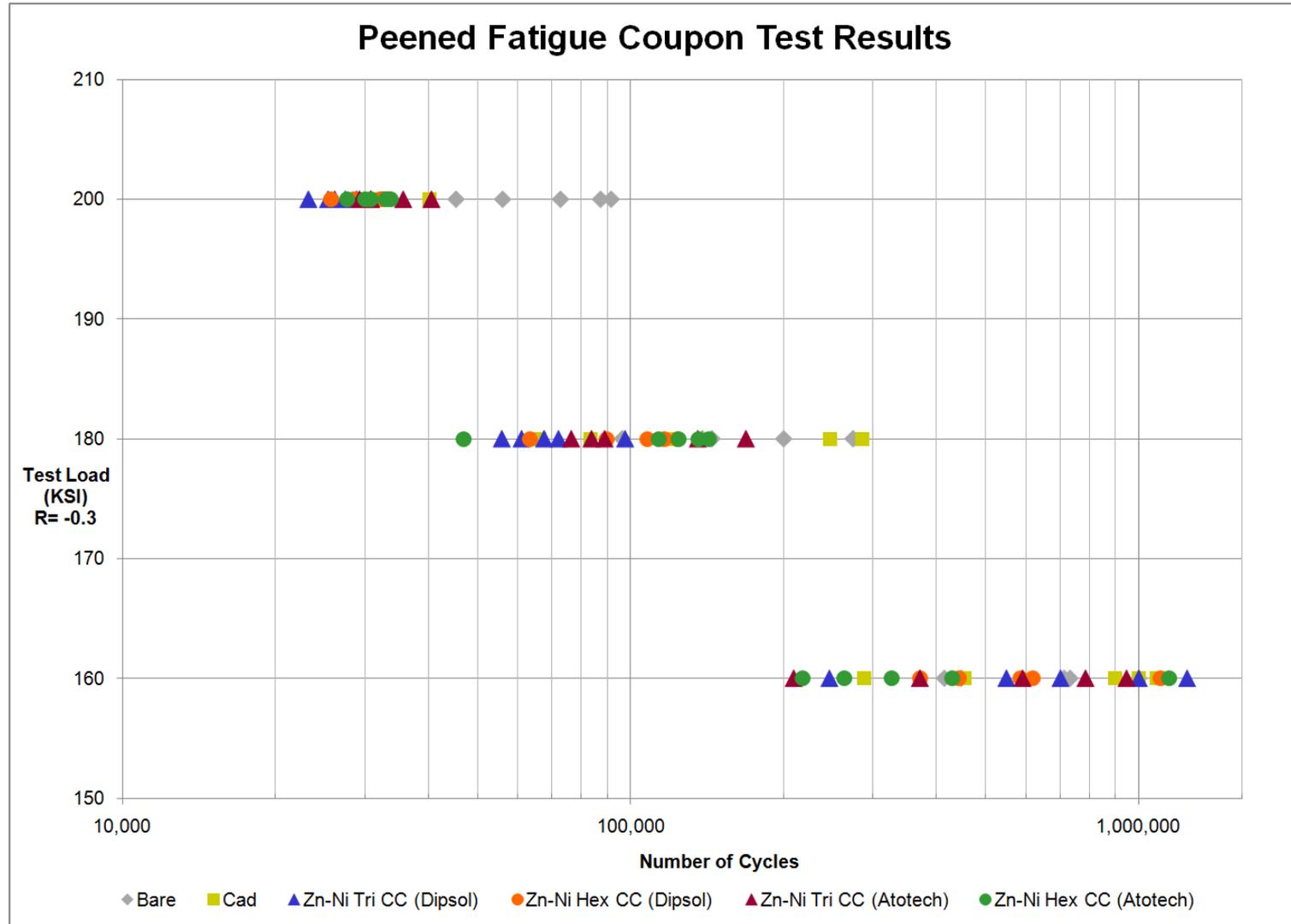


# Phase II Fatigue Testing



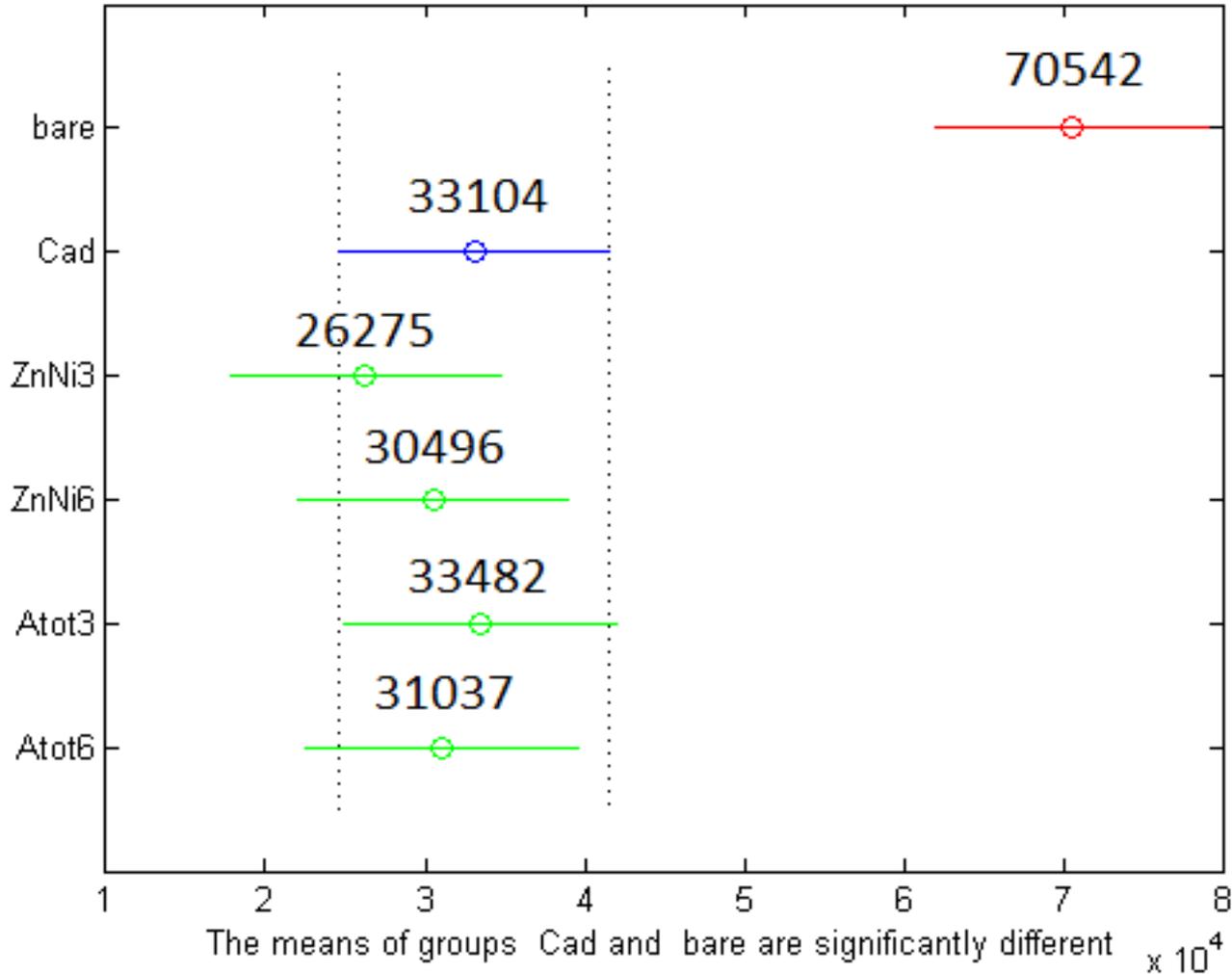
Shotpeened Coupons <sup>↗</sup>	Stress Loads (KSI) R= -0.3			Total Quantity
	160	180	200	
Bare	5	5	5	15
Cad Plated	5	5	5	15
Zn-Ni Plated Tri Chrome	5	5	5	15
Zn-Ni Plated Hex Chrome	5	5	5	15
*Zn-Ni Plated Atotech Tri Chrome	5	5	5	15
Zn-Ni Plated Atotech Hex Chrome	5	5	5	15
<b>Total Fatigue Coupons</b>				<b>90</b>
<b>* Bake before Tri CC</b>				

# Phase II Fatigue Testing



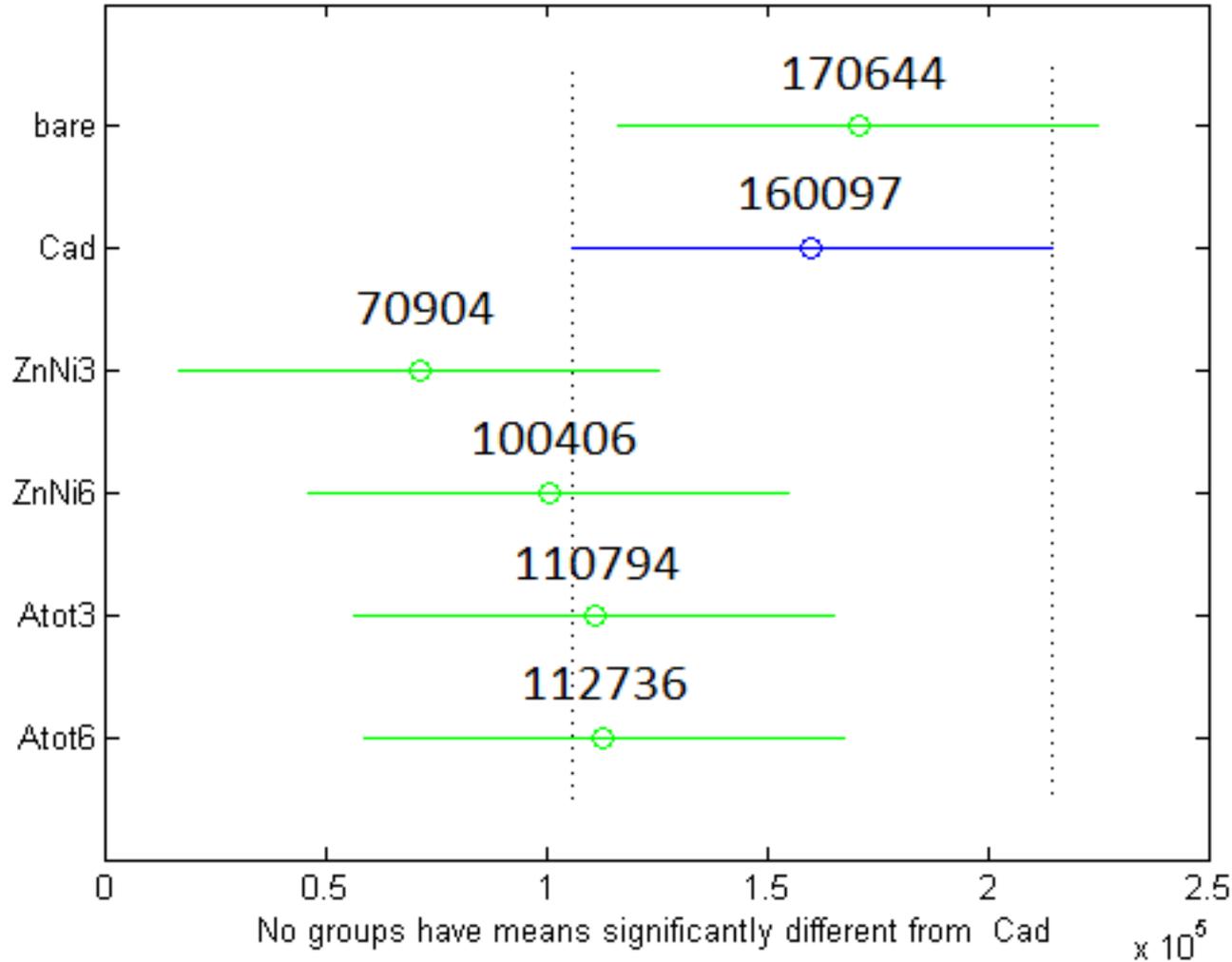
# Phase II Fatigue Testing

Means Compared @ 95% limits: 200 ksi Peened



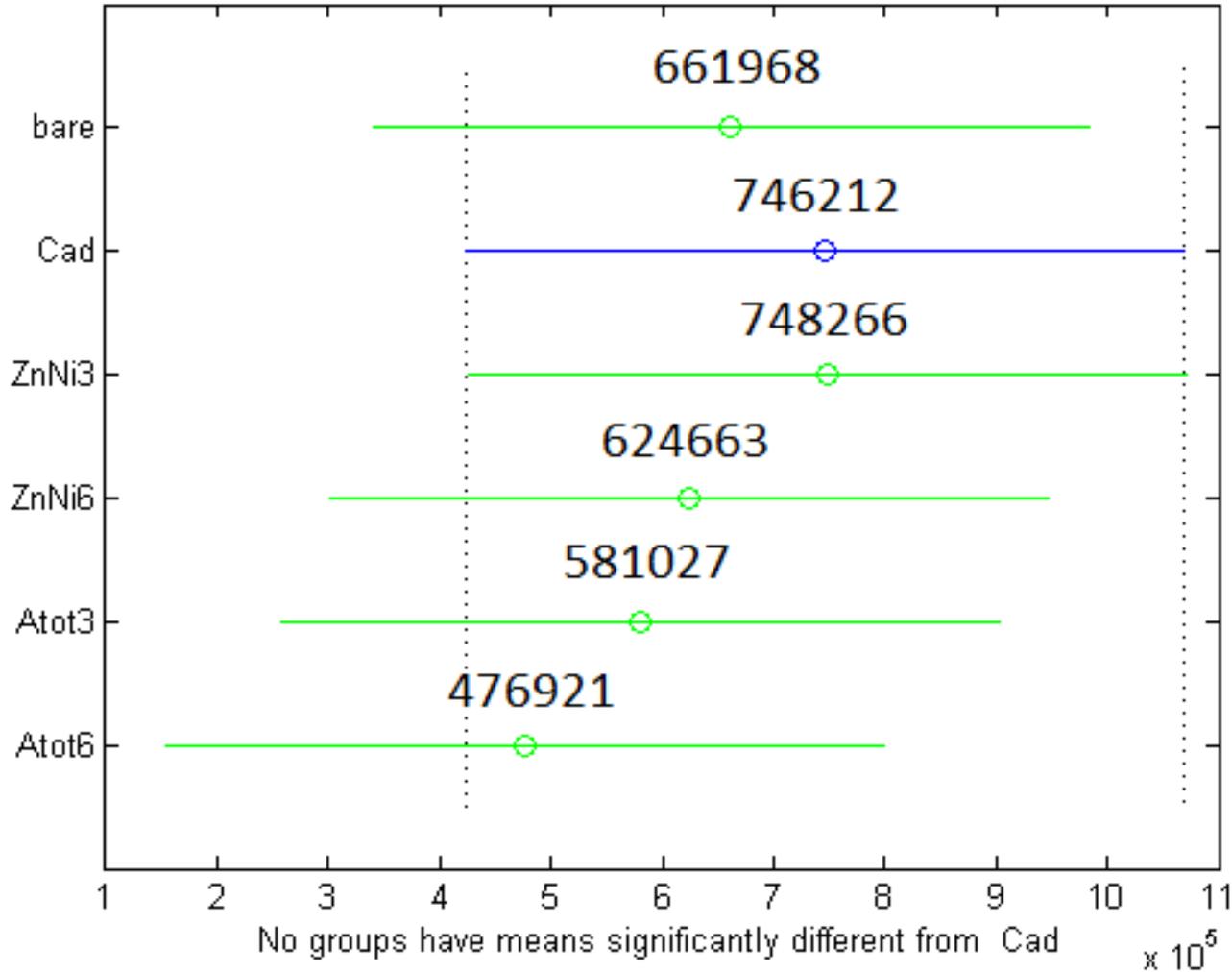
# Phase II Fatigue Testing

Means Compared @ 95% limits: 180 ksi Peened



# Phase II Fatigue Testing

Means Compared @ 95% limits: 160 ksi Peened



- 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



# of steel Panels	Plating	Scribed	Prime/Paint
3	LHE Zn-Ni	Yes	No
3	LHE Zn-Ni	No	No
3	Cd	Yes	No
3	Cd	No	No
3	LHE Zn-Ni	Yes	Yes
3	Cd	Yes	Yes

# Corrosion Performance



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

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

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

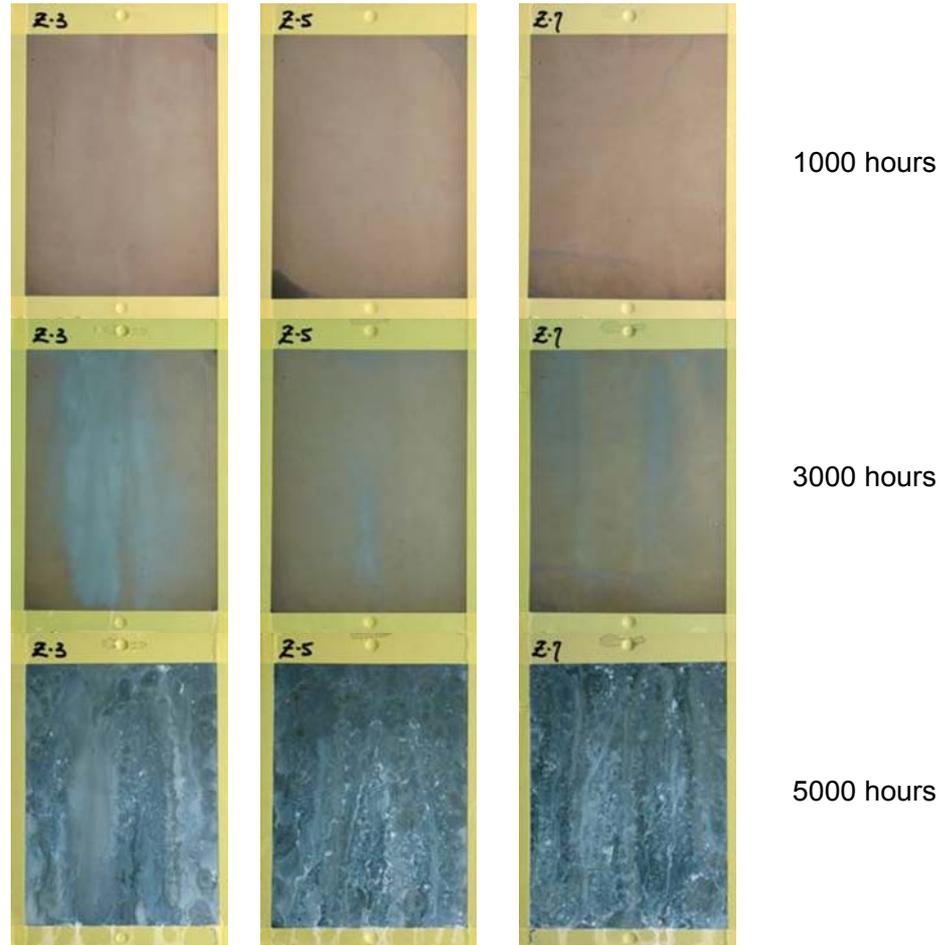
Test Panel  
 Removed From  
 Salt Spray Cabinet –  
 Excess Amount  
 of Red Rust  
 Detected

5000 hours

## Cadmium Coatings – Phase II ASTM B 117 Panels @ Boeing Unscribed)

# Corrosion Performance

IZ-C17+ Zn-Ni with Trivalent Chrome Conversion Coating  
Unscribed – ASTM B 117  
Figure 4



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

# Corrosion Performance



Cadmium with Hexavalent Chrome Conversion Coating  
 Scribed – ASTM B 117  
 Figure 5



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

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

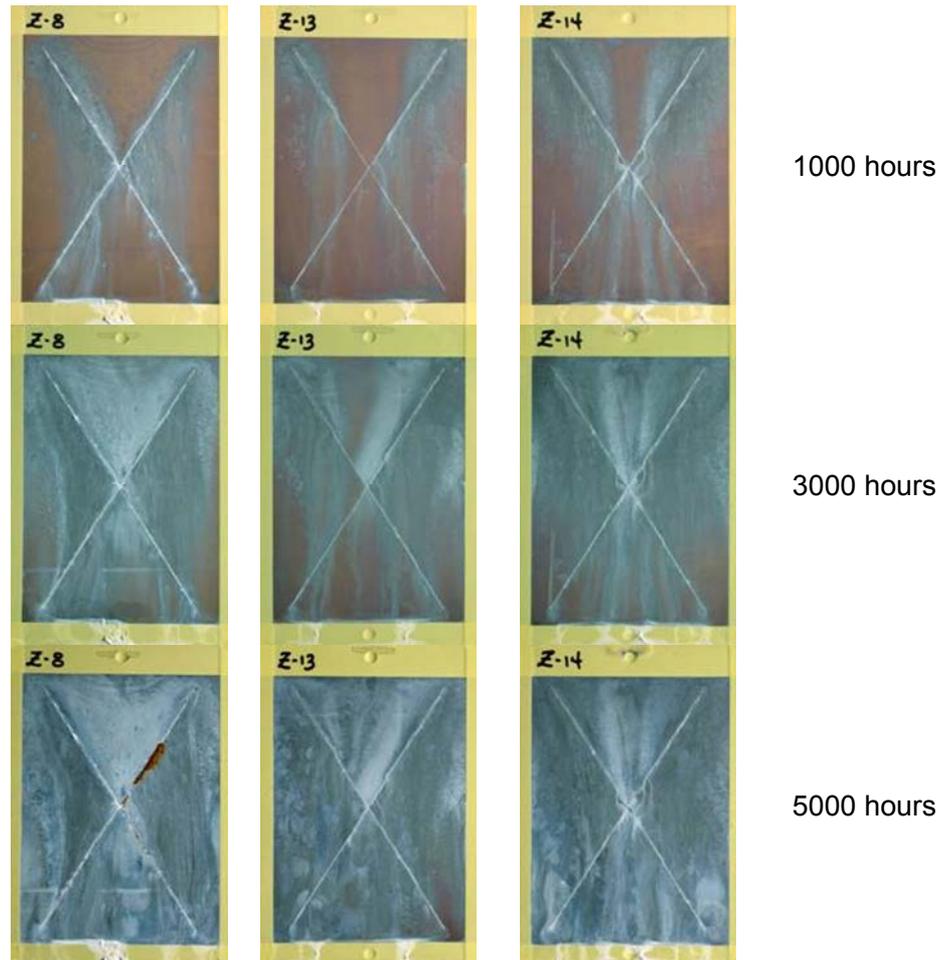
Test Panel  
 Removed From  
 Salt Spray Cabinet –  
 Excess Amount  
 of Red Rust  
 Detected

5000 hours

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

# Corrosion Performance

IZ-C17+ Zn-Ni with Trivalent Chrome Conversion Coating  
Scribed – ASTM B 117  
Figure 6

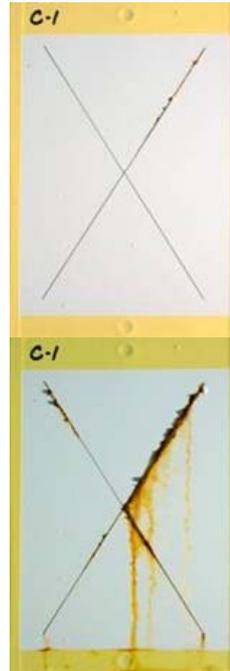


## Zinc Nickel Coatings – Phase II ASTM B 117 Panels @ Boeing (Scribed)

# Corrosion Performance



Cadmium with Hexavalent Chrome Conversion Coating  
 Scribed Painted – ASTM B 117  
 Figure 9



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

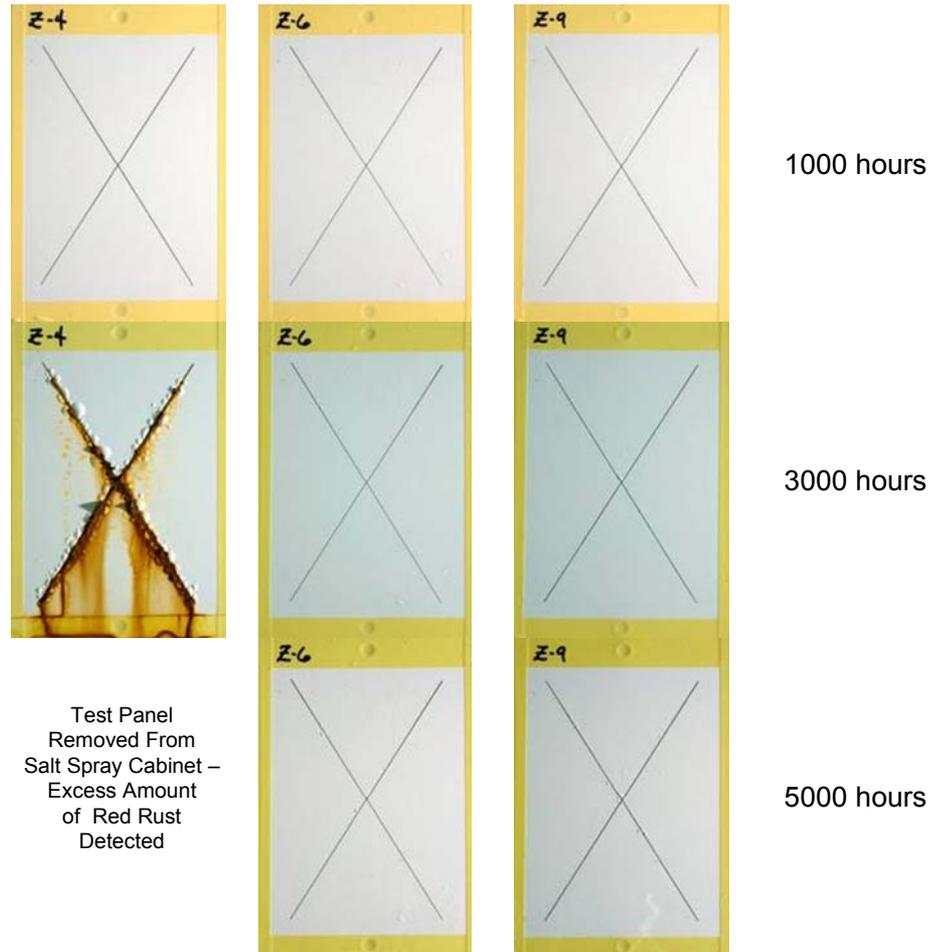
Test Panel  
 Removed From  
 Salt Spray Cabinet –  
 Excess Amount  
 of Red Rust  
 Detected

5000 hours

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

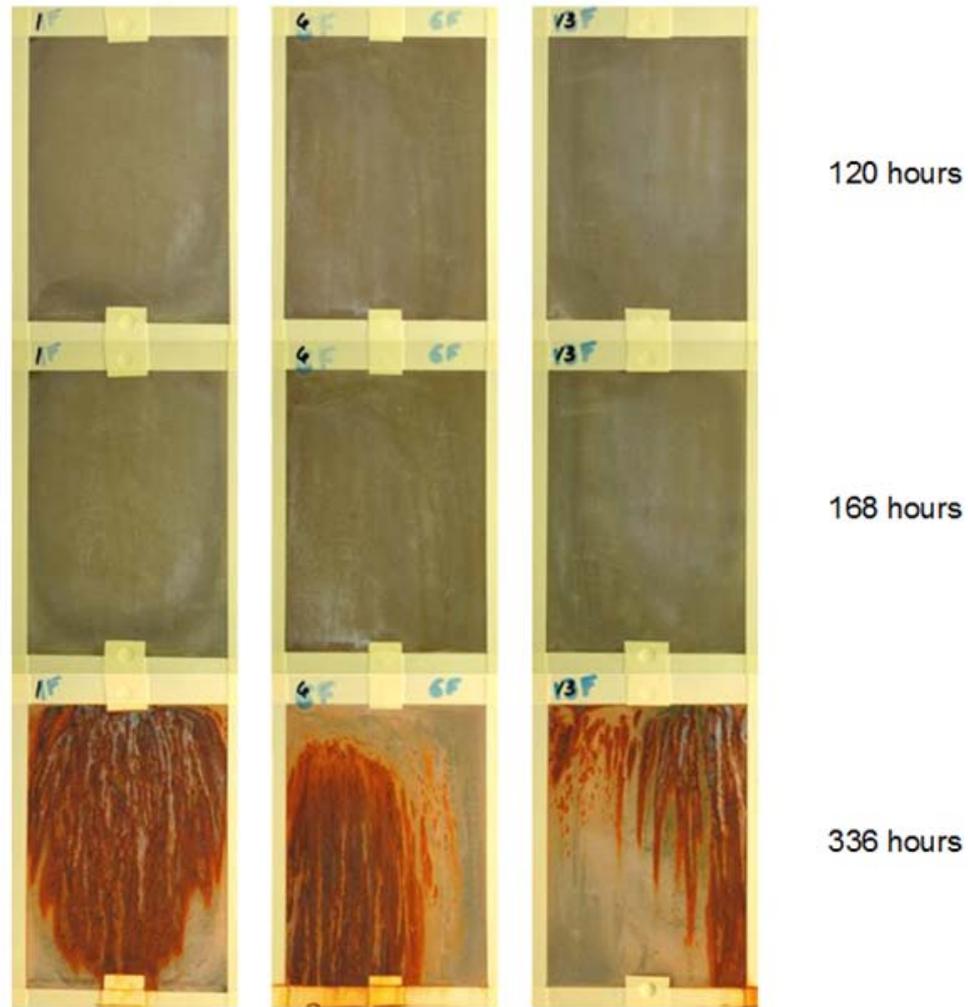
# Corrosion Performance

IZ-C17+ Zn-Ni with Trivalent Chrome Conversion Coating  
 Scribed Painted – ASTM B 117  
 Figure 10

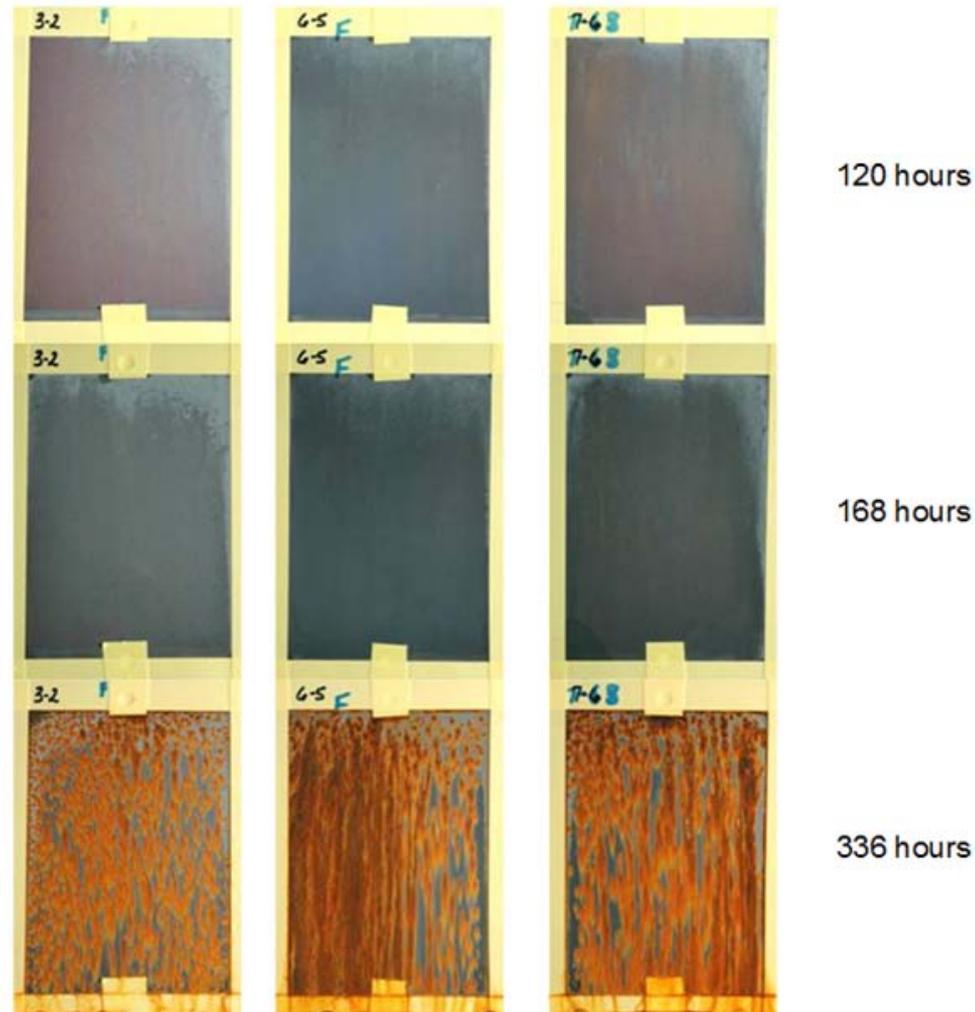


## Zinc Nickel Coatings – Phase II ASTM B 117 Panels @ Boeing (Painted/Scribed)

# G 85 SO<sub>2</sub> Corrosion-Cad



# G 85 SO<sub>2</sub> Corrosion-LHE Zn-Ni



# Brush Plating Repair



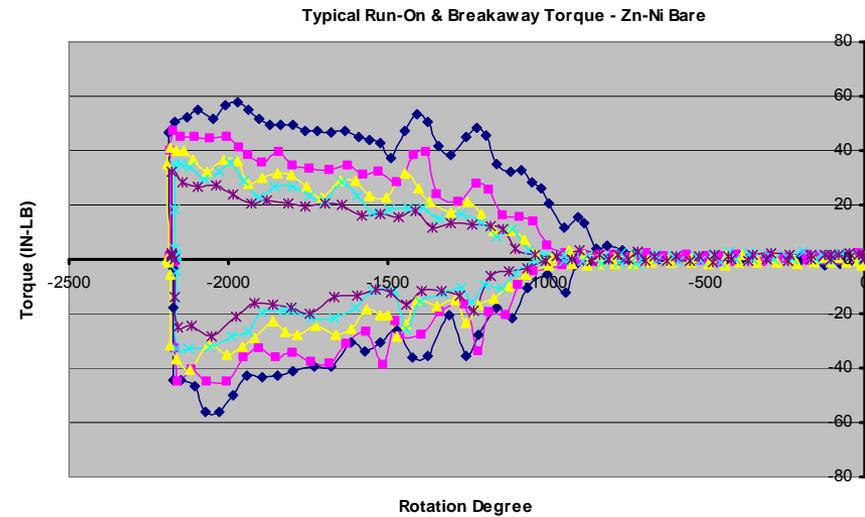
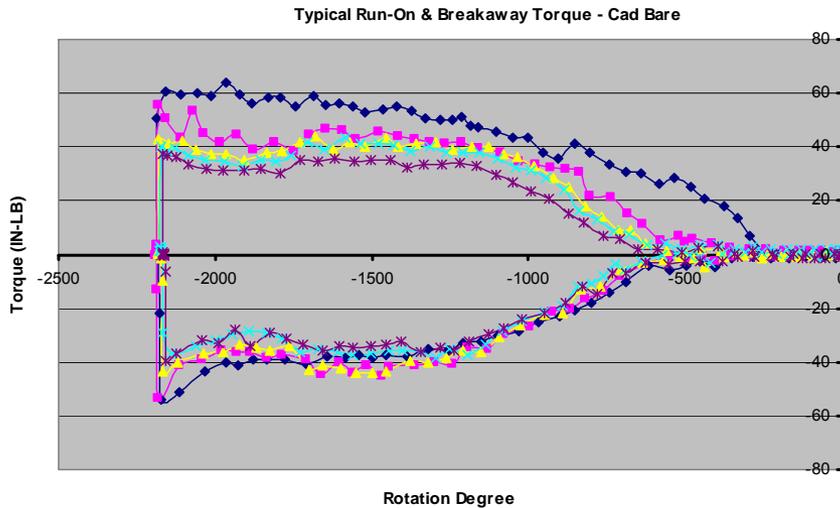
- In order for a brush LHE Zn-Ni plating to qualify it must pass the following tests:
  - Hydrogen Embrittlement (HE) testing per ASTM F519
  - Bend to break adhesion test per ASTM B571
  - Corrosion testing per ASTM B117
- SIFCO recommended procedures were used to plate several sets of HE type 1a.1 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



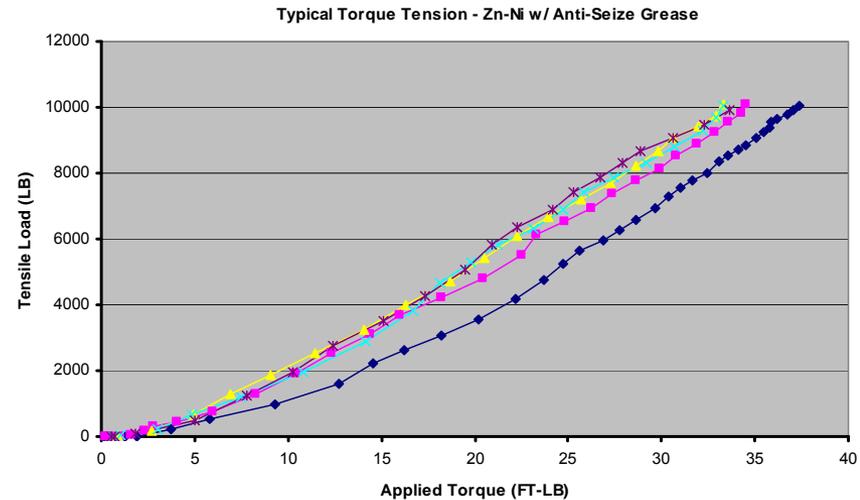
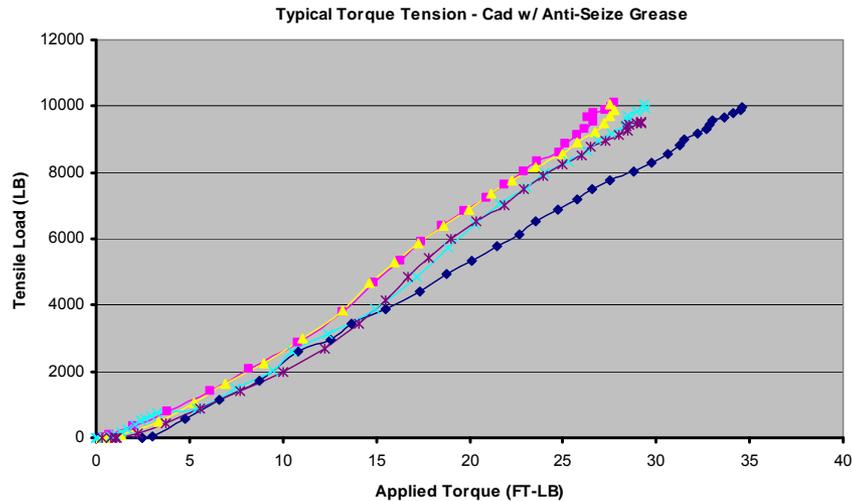
- Robins AFB Cad plating replacement on threaded fastener and components
  - Typical chart for run on – break away test showing Cad vs. LHE Alkaline Zn-Ni



# 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

- 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  $P_{th}$
    - $\%NFS = 100 \times P_{th} / NFS_{F1624}$
    - If  $\%NFS \geq 90\%$  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

# Process Validation Plan



- 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

# Process Validation Plan



- 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
    - Requires quarterly chemical analysis for trace contamination metals for both tanks
  - Agitation requirements
    - Agitation should be sufficient enough to re-circulate the tanks plating solution a minimum of 3 times per hour
    - No air agitation

# Process Validation Plan



- 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

# Process Validation Plan



- 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)

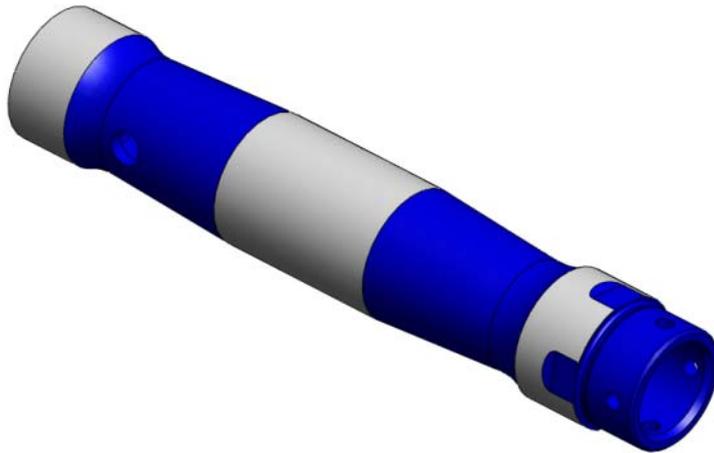
# Phase III Effort Prototype Process Line



## Prototype Part Matrix

Component	Part #
C-5 MLG Stop Plate	4G11453-101B
F-15 MLG Outer Cylinder	68A412702-1001/1002
B-1 MLG Axle	1881B85
F-15 MLG Lower Drag Brace	68A410792-2001
A-10 MLG Torque Arm	19046-1
F-16 NLG Inner Cylinder	2007644-103
C-5 MLG Rotation Collar	4G13565-101A/-101B
A-10 NLG Axle	18800-3

# Phase III Effort Prototype Parts

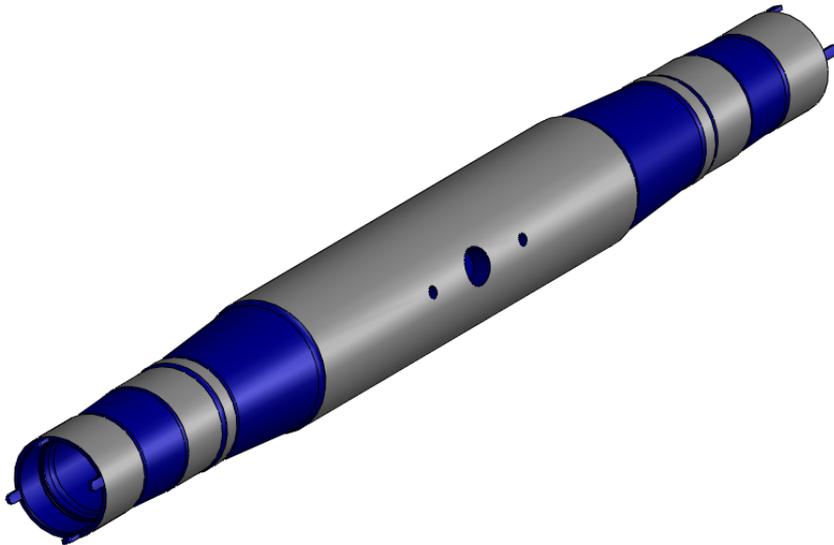


**A-10 NLG Axle**

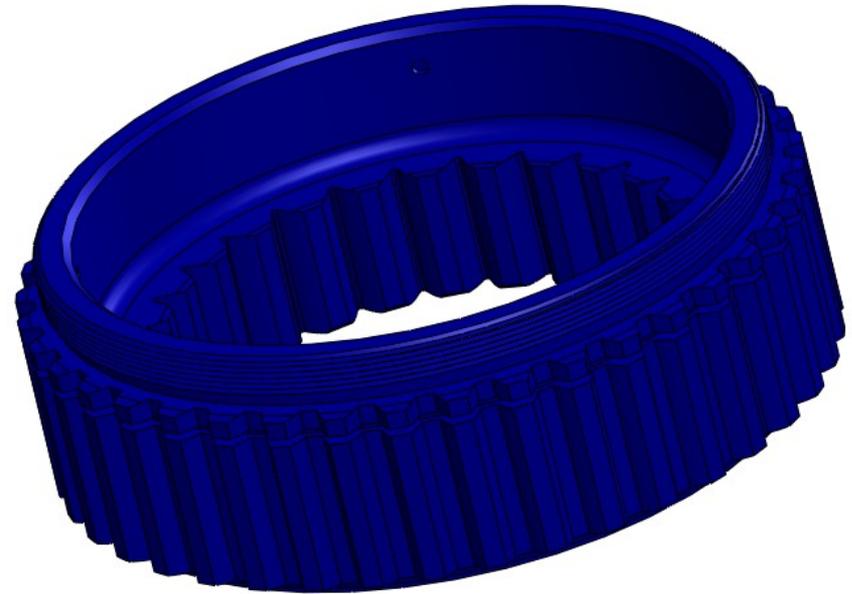


**A-10 MLG Torque Arm**

# Phase III Effort Prototype Parts

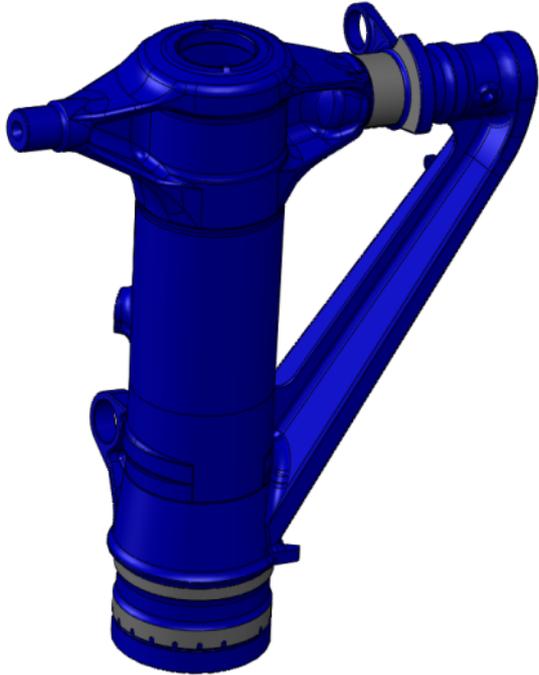


**B-1B MLG Axle**



**C-5 MLG Rotation Collar**

# Phase III Effort Prototype Parts



**F-15 MLG Cylinder**

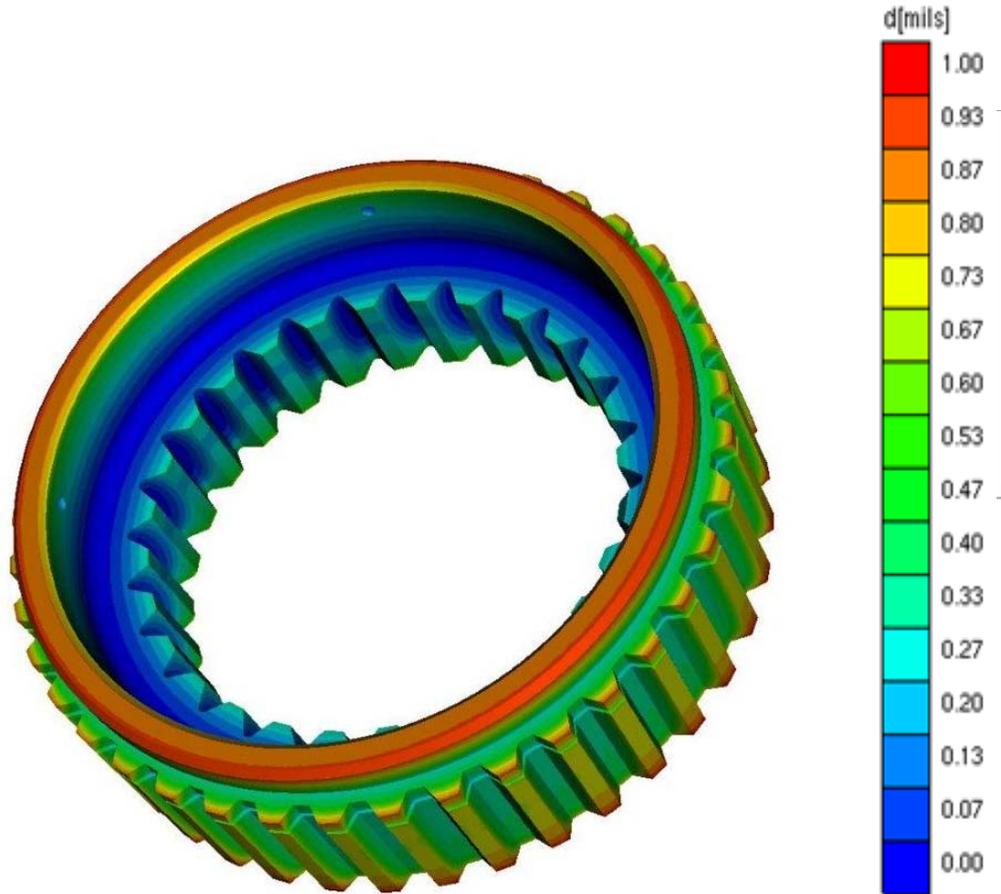


**F-16 NLG Inner Cylinder**



**F-15 MLG Lower Drag Brace**

# Phase III Effort Prototype Anode Design



**C-5 MLG Rotation Collar**

# Phase III Effort Prototype B-1 Bushing

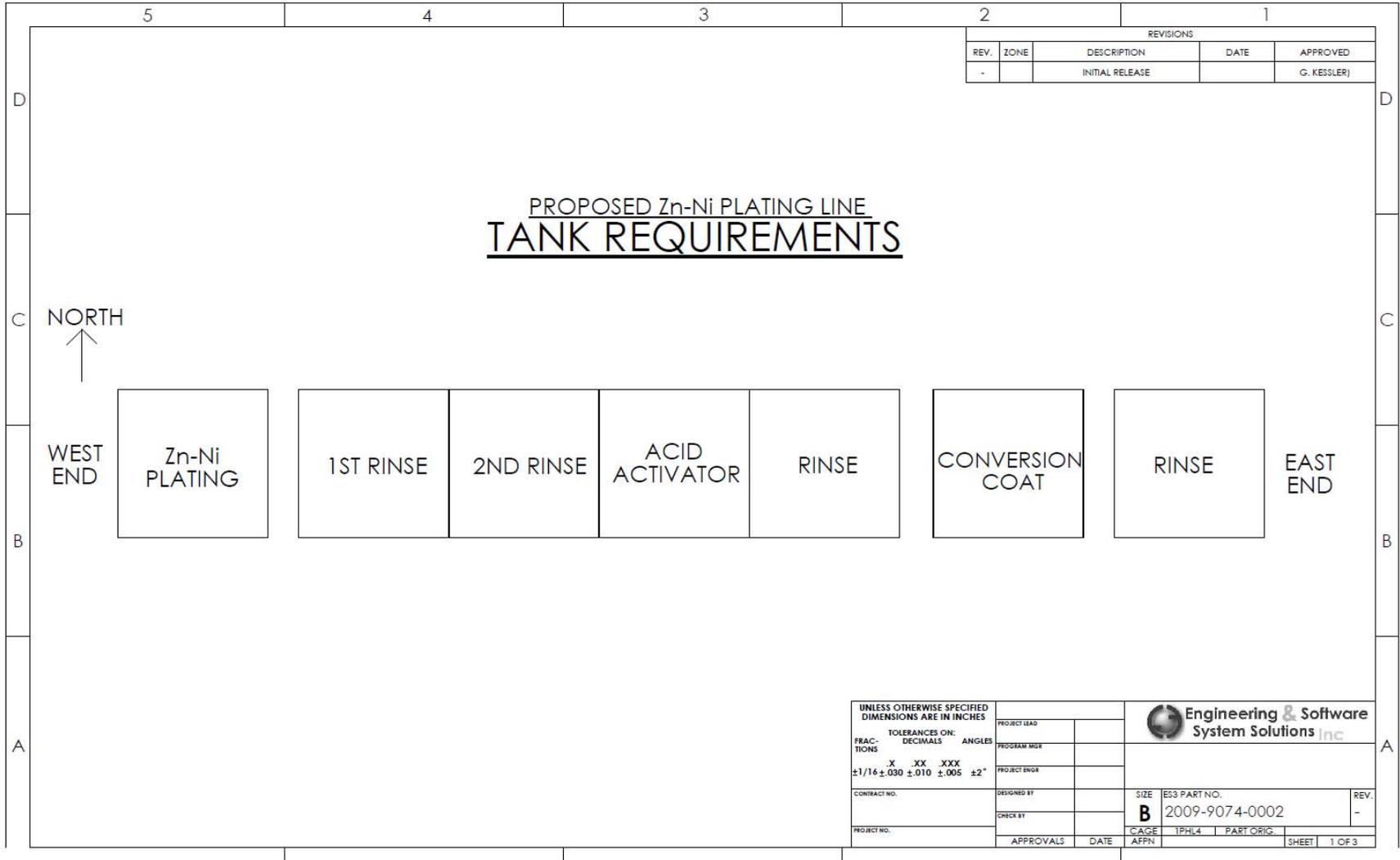


**Plating B-1 Bushings LHE Zn-Ni**



**Plated LHE Zn-Ni B-1 Bushings**

# Phase III Effort Proposed Prototype Plating Line







# Questions



*www.ES3inc.com • 1669 E. 1400 S • Clearfield, UT 84015  
(801) 926-1150 • fax (801) 926-1155*