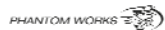


SERDP Hydrogen Re-Embrittlement DoE Test Plan Status

By:
Stephen Gaydos
Boeing – St. Louis

May 17, 2007
DoD Metal Finishing Workshop – Chromate Alternatives for
Metal Treatment and Sealing
Hilton Garden Inn – Layton, UT

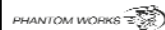


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DoD Metal Finishing Workshop May 2006 – Washington D.C.

- Workshop Identified Barriers for Implementing Cadmium Plating Alternatives
- Hydrogen Embrittlement Testing Was Identified as a Major Barrier
 - Need to Verify that Alternative Coating Process is Non-Embrittling to HSS (Called Embrittlement Testing)
 - Need to Verify that Maintenance Chemicals on HSS with Alternative Coating Do Not Embrittle the HSS (Called Re-Embrittlement Testing)



2

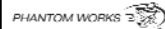
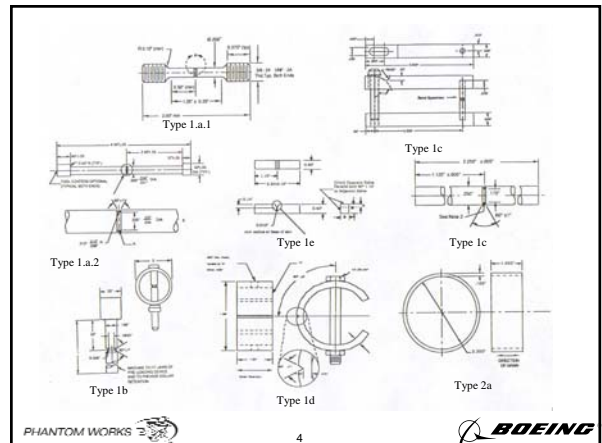


ASTM Embrittlement Test

- Process Control Test Conducted per ASTM F519 on Plated/Coated (and Baked) Specimens
 - 7 Specimens to Choose From
 - 1a.1, 1a.2, 1b, 1c, 1d, 1e, 2a (Evaluated by Boeing)
 - 1b Rarely Used – Not Tested
 - 1d Most Sensitive
 - 2a Least Sensitive
 - 1e Loses Sensitivity at Low H Levels
 - Sustained Load (SL) Method is Typical Process
 - Specimens Typically Loaded at 75% NFS for **200 Hours**
 - Incremental SL (ISL) Methods Being Evaluated But Not Widely Accepted
 - Cadmium Replacement Barrier Identified Test Time
 - **Need a 24 Hour Test Method**



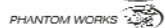
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ASTM F 519 Specimens

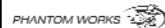


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ASTM Re-Embrittlement Test

- ASTM F 519 – Annex A5
 - Annex A5 Describes Different Test Method Guidelines
 - "...the testing protocols described in this section (Annex A5) should only be used as a guideline. . ."
 - 7 Specimen Types
 - Aggressive Chemicals
 - » Hold at 45% NFS while Wet for ?? Time, Then Rinse and Dry and Hold at 75% NFS for 200 hrs
 - Passive Chemicals
 - » Hold at 45% or 65% NFS while Wet for 150 hrs
 - Solution Volume, Temperature and Concentration are Undefined
 - Cadmium Replacement Barrier Identified
 - **Need a Simplified and Standard Test Method to Re-Qualify All Maintenance Chemicals That are Currently Qualified with Cadmium Plated HSS Using the Alternative Coating on HSS**
 - Potential Cost Savings is Significant



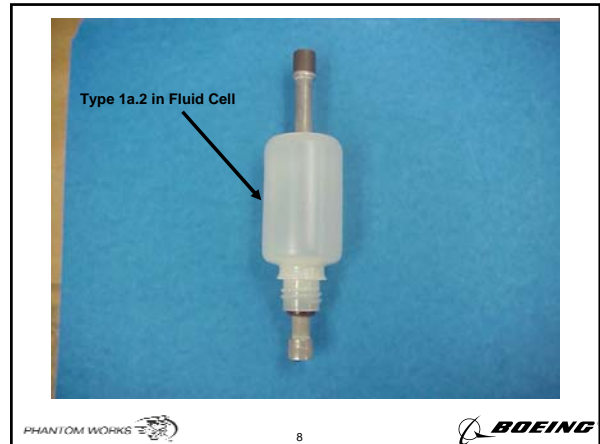
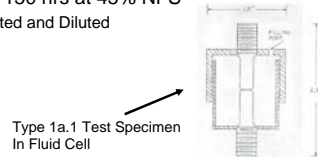
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Maintenance Chemical Qualification

Hydrogen Re-Embrittlement -

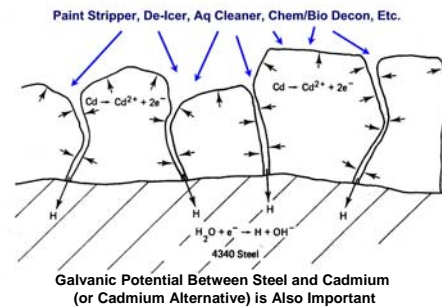
- ASTM F519, Test Specimens
 - LHE Cadmium Plated High Strength Steel (HSS)
- Wet Notch - 150 hrs at 45% NFS
 - Concentrated and Diluted



Hydrogen Re-Embrittlement

- Hydrogen Re-Embrittlement Mechanism
 - It's a Form of Corrosion
 - Cathodic Reaction Occurs on Steel
 - $2H^+ + 2e^- = H_2$ (gas)
 - Anodic Reaction Occurs on Sacrificial Cadmium Coating
 - $Cd = Cd^{2+} + 2e^-$
 - Parts Affected by Hydrogen Re-Embrittlement Typically Cannot be Baked to Remove Hydrogen
 - Parts are typically assembled on equipment
 - Baking is 375°F x 24 hrs.
- Test Data Scatter is Common
 - Appears to be Testing Coating Integrity or Test Set-Up and Not The Cleaner

Basic Mechanism in Hydrogen Re-Embrittlement



Re-Embrittlement Test Issues

- Not Standardized Across the Industry – Variations of ASTM Annex A5 Used
 - Various ASTM F519 Specimens Used
 - Type 1.a.1, 1.a.2, 1b, 1c, 1d, 1e, and 2a
 - Various Specimen Immersion Methods
 - Wet for 150 hrs, Wet Than Dry, Concentrated or Diluted Chemicals
 - Volume of Fluid, Temperature
 - Various Loading Methods
 - Tension, Bending, Sustained Load, Incremental Step Loading (24 hours)
 - 45%, 65%, 75% NFS, 80%YS for 150 or 200 hrs

Re-Embrittlement Testing Is Our Tower of Babel



The Confusion of Tongues by Gustave Doré (1865)

History of Re-Embrittlement Test

- Sustained Load and Time to Pass H-RE Test in Water Determined in '70s by Lockheed, DAC and Boeing
 - Specimens Tested: 4340 Steel 260-280 ksi, LHE Cd Plate
- Results:
 - 1a.2 Passed at 40% NFS (> 200 hrs No Fracture)
 - 1c Passed at 45% NFS (> 300 hrs No Fracture)
 - 2a Passed at 80% YS (> 1300 hrs No Fracture)
- Conclusions in ASTM F 519 (Current Standard):
 - 1a and 1c = 45% NFS for 150 hrs is a pass
 - 2a = 80% Yield Strength for 150 hrs is a pass
- Current Standard Test is Questionable
 - 1a.1, 1b, 1d, 1e Added Without Testing
 - Passing This H-RE Test With Water is Difficult

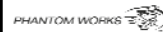


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Proposed Solution to Problem

- Modify Re-Embrittlement Test Parameters in ASTM F 519 to Insure that Baseline Cadmium Can Pass in Water
 - Conduct "Round-Robin" DoE Tests
 - Use LHE Cadmium as Baseline
 - Look at Cadmium Alternatives After Modifying Test Parameters for LHE Cadmium
 - Need to Determine What is Required to Have Cadmium Pass Water Tests?
- ARL Received \$100K SERDP Funding in January 2007 to Start DoE Testing
 - Boeing and ARL Also Contributing Labor to Conduct DoE
 - Richard Green Co. Fabricating Test Specimens
 - Omega Research To Assist in Conducting Tests

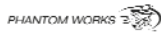


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DoE Test Plan Background Info

- Boeing Conducted Ruggedness Test to Determine Precision of Hydrogen Re-Embrittlement Test Method
- Experimental Factors (ASTM E 1169)
 - A. surface treatment (LHE cadmium plated versus bare)
 - B. notch condition (bare notch root versus Cd plated notch root)
 - C. ratio of fluid volume to surface area of specimen exposed as ml/cm²
 - D. fluid concentration in volume percent
 - E. fluid temperature in degrees Fahrenheit
 - F. fluid exposure time (hrs)
 - G. load (% NFS)
- Tested 1a.1 and 1d in Sustained Load (Water and Aqueous Cleaners)
 - *RED = Significant Effect



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DoE Proposed Test Plans

Phase I

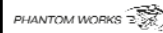
	-a	-	0	+	+a	Desired Tolerance
Strength (ksi)	140	168	210	252	280	± 7% K _{av}
Test Load (%NFS)	35	45	60	75	85	± 7% NFS
Water Conductivity (µS/cm)	0.056	0	0.02775	0		± 7% µS/cm
	I	IV	Soft Water	Hard Water	3.5% NaCl	

Phase II

	-a	-	0	+	+a	Desired Tolerance
Strength (ksi)	140	168	210	252	280	± 7% K _{av}
Test Load (%NFS)	40	50	65	80	90	± 7% NFS
Cleaner Concentration (wt%)	1	17.5	50.5	83.5	100	± 7% Wt%

Phase III

	-a	-	0	+	+a	Desired Tolerance
Strength (ksi)	140	163	210	257	280	± 7% K _{av}
Bake Delay (hrs)	0	12	36	60	72	± 7% NFS
Bake Time (hrs)	0	4	12	20	24	± 7% µS/cm



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Phase I – DoE Test Plan

- 5 Levels – 3 Factors
 - Tensile Strength
 - 140, 160, 210, 250 and 260 ksi
 - Sustained Test Load
 - 35, 45, 60, 75, 85% NFS
 - Water Purity
 - Ty I, Ty IV, Soft, Hard, 3.5% NaCl
- 5 Specimen Types
 - Repeat Test 3 Times

	A	B	C	Run Order	
Repeat entire matrix 3x for 1a.1, 1a.2, 1c, 1d and 1d					
	RUN ID	Strength (ksi)	Test Load (%NFS)	Water Conductivity (µS/cm)	
Linear Portion	L1	-	-	-	TBD
	L2	-	-	+	
	L3	-	+	-	
	L4	-	+	+	
	L5	+	-	-	
	L6	+	-	+	
Center Points	CP1	0	0	0	TBD
	CP2	0	0	0	
	CP3	0	0	0	
Center Points	CP4	0	0	0	TBD
	CP5	0	0	0	
	CP6	0	0	0	
Quadratic Portion	Q1	0	0	0	TBD
	Q2	0	0	0	
	Q3	0	0	0	
	Q4	0	0	0	
	Q5	0	0	0	
	Q6	0	0	0	



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Phase 1 DoE Test Plan (Cont.)

	A	B	C	Run Order
Repeat entire matrix 3x for 1a.1, 1a.2, 1c, 1e and 1d				
	RUN ID	Strength (ksi)	Test Load (%NFS)	Water Conductivity (µS/cm)
	1	TBD	TBD	TBD
	2			
	3			
	4			
	5			
	6			

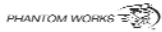


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Status

- \$50K Spent to Order 390 Test Specimens
- Plating, Inspecting and Testing by Outside Labs Will Easily Consume the Remaining \$50K of SERDP Funds
 - ARL and Boeing Providing Labor to Make-Up Difference
 - Additional Support Would Be Beneficial
- Cadmium Plating of Test Specimens to Begin by July 2007
- Phase I DoE Testing Begins August 2007

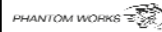


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Expected Outcome from Phase I

- Determine Which Specimen Type Gives Reproducible and Meaningful Results
- Determine What Strength Level of Steel is Non-Embrittling
- Determine The Load That Gives Reproducible and Meaningful Results
- Identify the Test Parameters That Allows Cadmium Plated Steel to Pass Test in Water
- Use Information from Phase I to Conduct Phase II DoE Tests

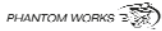


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Round-Robin DoE Test Coordinators

- Scott Grendahl – ARL, Aberdeen
 - ASTM Task Group Leader for Revising ASTM F 519 Annex A5
- Ed Babcock – Boeing, Mesa
 - ASTM Task Group Leader for Revising ASTM F 519 Main Specification
- Steve Gaydos – Boeing, St. Louis
 - ASTM F07.04 Subcommittee Chairman for Hydrogen Embrittlement Test Methods



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