Design-of-Experiment Approach to Hydrogen Re-Embrittlement Evaluation

WP - 2152

Prepared by
Scott M. Grendahl
U.S. Army Research Laboratory, APG, MD

Presented by:
Steve Gaydos
The Boeing Company, St. Louis, MO

ASETS DEFENSE February 7-10, 2011
**Report Documentation Page**

<table>
<thead>
<tr>
<th>1. REPORT DATE</th>
<th>2. REPORT TYPE</th>
<th>3. DATES COVERED</th>
</tr>
</thead>
<tbody>
<tr>
<td>FEB 2011</td>
<td></td>
<td>00-00-2011 to 00-00-2011</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>4. TITLE AND SUBTITLE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Design-of-Experiment Approach to Hydrogen Re-Embrittlement Evaluation</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>5a. CONTRACT NUMBER</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>5b. GRANT NUMBER</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>5c. PROGRAM ELEMENT NUMBER</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>5d. PROJECT NUMBER</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>5e. TASK NUMBER</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>5f. WORK UNIT NUMBER</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>6. AUTHOR(S)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)</th>
</tr>
</thead>
<tbody>
<tr>
<td>U. S. Army Research Laboratory, Aberdeen Proving Ground, MD, 21005</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>8. PERFORMING ORGANIZATION REPORT NUMBER</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>10. SPONSOR/MONITOR’S ACRONYM(S)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>11. SPONSOR/MONITOR’S REPORT NUMBER</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>12. DISTRIBUTION/AVAILABILITY STATEMENT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Approved for public release; distribution unlimited</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>13. SUPPLEMENTARY NOTES</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASETSDFense 2011: Sustainable Surface Engineering for Aerospace and Defense Workshop, February 7 - 10, 2011, New Orleans, LA. Sponsored by SERDP/ESTCP.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>14. ABSTRACT</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>15. SUBJECT TERMS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>16. SECURITY CLASSIFICATION OF:</th>
</tr>
</thead>
<tbody>
<tr>
<td>a. REPORT: unclassified</td>
</tr>
<tr>
<td>b. ABSTRACT: unclassified</td>
</tr>
<tr>
<td>c. THIS PAGE: unclassified</td>
</tr>
</tbody>
</table>

| 17. LIMITATION OF ABSTRACT:|
| Same as Report (SAR) |

<table>
<thead>
<tr>
<th>18. NUMBER OF PAGES:</th>
</tr>
</thead>
<tbody>
<tr>
<td>29</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>19a. NAME OF RESPONSIBLE PERSON</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
</tr>
</tbody>
</table>

Standard Form 298 (Rev. 8-98)
Proscribed by ANSI Std Z39-18
Re-Embrittlement Test Issues

1a.1
1a.2
1b
1c
1d
2a
1e
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 Then 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
  - Various Strength Levels to Bake or No-Bake
    - 160 ksi, 180 ksi, 200 ksi, 220 ksi
Re-Embrittlement Testing Is Our Tower of Babel

*The Confusion of Tongues* by Gustave Doré (1865)
Typical ASTM F07.04 Hydrogen Embrittlement Meeting

Steve Gaydos
F07.04 Subcommittee Chairman

F07.04 Subcommittee Members
Project Team

- Lead PI – S. Grendahl, ARL
- C. Willan – Omega Research Inc.
- J. Green – NAVAIR Pax River
- C. Hogan – Hill AFB
- R. Green – Green Specialty Service
- D. Kelly – ASKO Processing Inc.
Technical Objective

- Increase the implementation and utilization of environmentally friendly maintenance chemicals and cadmium alternatives by alleviating the HE obstacle.

  - Year 1 – Life models for aerospace grade 4340 steel
  - Year 2 – Life models for prospective maintenance chemicals
  - Year 3 – Life models for prospective alternative coatings
Technical Approach

- He testing has traditionally been done pass/fail on worst case material.

- DoE approach develops life prediction models over a range of material strength, applied stress, and environment:
  - 280 ksi
  - Stress varies with geometry
  - Cad plated steel

  - 140 - 280 ksi
  - 10 - 95% NFS
  - % of NaCl, or Concentration or Plating

- Statistical analysis allows a reasonable matrix size while accounting for full spectrum of variables with prediction:
  - 5x5x5x5 (625) Vs. 400
Technical Approach

- Linear with Center points
- Quadratic
- Confirmation Runs

- Base model is developed from Linear and Quadratic portions
  - $Y = \ln X = 19.01 - 11.67 \times \text{strength} - 9.93 \times \text{test_load} - 0.88 \times \text{NaCl} + \text{error}$
  - Run confirmations, re-compute, and refine model
Technical Approach

- Material Strength (140 - 280 ksi)
- Applied Stress (% of NFS)
- Environment (wt% of NaCl, or Conc. of chemical or thickness of coating)
- Model Yields - TTF (Time to Failure)
Technical Approach
Technical Approach

- Year 1 models will be created for aerospace 4340 steel
- Assess results via team to determine best geometry for program
- 1 geometry then used to assess applicable maintenance chemicals or platings
- Results will provide the airworthiness authority data to assess which processes (chemicals or platings) and applications are safe zones.
Seed Funding Results

- Air-melted 4340 steel, 10 - 95% NFS, 0 - 3.5% NaCl
- 5 geometries, 5 heat treatments
- Separate models for each heat treatment (or material strength)
  - T1 140 ksi
  - T2 158 ksi
  - T3 210 ksi
  - T4 262 ksi
  - T5 280 ksi
1a1 Results

Predicted Median Lifetime
Strength=T1 (140 KSI)

Predicted Median Lifetime
Strength=T2 (158 KSI)

Predicted Median Lifetime
Strength=T3 (210 KSI)

Predicted Median Lifetime
Strength=T4 (262 KSI)

Predicted Median Lifetime
Strength=T5 (280 KSI)

T1 140 ksi
T2 158 ksi
T3 210 ksi
T4 262 ksi
T5 280 ksi
1a2 Results

Predicted Median Lifetime
Strength=T1 (140 KSI)

Predicted Median Lifetime
Strength=T2 (158 KSI)

Predicted Median Lifetime
Strength=T3 (210 KSI)

Predicted Median Lifetime
Strength=T4 (262 KSI)

Predicted Median Lifetime
Strength=T5 (280 KSI)

T1 140 ksi
T2 158 ksi
T3 210 ksi
T4 262 ksi
T5 280 ksi
1c Results

Predicted Median Lifetime
Strength=T1 (140 KSI)

Predicted Median Lifetime
Strength=T2 (158 KSI)

Predicted Median Lifetime
Strength=T3 (210 KSI)

Predicted Median Lifetime
Strength=T4 (262 KSI)

Predicted Median Lifetime
Strength=T5 (280 KSI)

T1 140 ksi
T2 158 ksi
T3 210 ksi
T4 262 ksi
T5 280 ksi
1d Results

Predicted Median Lifetime
Strength=T1 (140 KSI)

Predicted Median Lifetime
Strength=T2 (158 KSI)

Predicted Median Lifetime
Strength=T3 (210 KSI)

Predicted Median Lifetime
Strength=T4 (262 KSI)

Predicted Median Lifetime
Strength=T5 (280 KSI)

T1 140 ksi
T2 158 ksi
T3 210 ksi
T4 262 ksi
T5 280 ksi
1e Results

Predicted Median Lifetime
Strength=T1 (140 KSI)

Predicted Median Lifetime
Strength=T2 (158 KSI)

Predicted Median Lifetime
Strength=T3 (210 KSI)

Predicted Median Lifetime
Strength=T4 (262 KSI)

Predicted Median Lifetime
Strength=T5 (280 KSI)

T1 140 ksi
T2 158 ksi
T3 210 ksi
T4 262 ksi
T5 280 ksi
T2 (158 ksi) Results

Predicted Median Lifetime
Strength=T2 (158 KSI)

1a2

1d

1a1

Predicted Median Lifetime
Strength=T2 (158 KSI)

1e

1c

Predicted Median Lifetime
Strength=T2 (158 KSI)
T3 (210 ksi) Results

Predicted Median Lifetime
Strength=T3 (210 KSI)

1a2

Predicted Median Lifetime
Strength=T3 (210 KSI)

1d

Predicted Median Lifetime
Strength=T3 (210 KSI)

1a1

Predicted Median Lifetime
Strength=T3 (210 KSI)

1e

Predicted Median Lifetime
Strength=T3 (210 KSI)

1c
T4 (262 ksi) Results

Predicted Median Lifetime
Strength=T4 (262 KSI)

1d

Predicted Median Lifetime
Strength=T4 (262 KSI)

1a2

Predicted Median Lifetime
Strength=T4 (262 KSI)

1a1

Predicted Median Lifetime
Strength=T4 (262 KSI)

1e

Predicted Median Lifetime
Strength=T4 (262 KSI)

1c
T5 (280 ksi) Results

Predicted Median Lifetime
Strength=T5 (280 KSI)

1d

1a2

1a1

Predicted Median Lifetime
Strength=T5 (280 KSI)

1e

1c
Transition Plan

- Work has been briefed and discussed by ASTM committee F07 on Aerospace and Aircraft, and in detail within subcommittee F07.04 on hydrogen embrittlement
- Most active participants of the committee are directly involved
- Changes to F-519 are likely upon completion and data review
- Lifetime prediction models for the targeted maintenance chemicals will be utilized by AMCOM/AMRDEC to alleviate the presently existing requirement of bake relief treatments for processes that have failed HE testing
  - Material applications below susceptibility threshold (e.g. 180 ksi)
  - Service stress applications below threshold (e.g. below 50% UTS)

- Lifetime prediction models for cadmium alternatives will be transitioned to service use for applications shown to be below the HE susceptibility threshold (e.g. ZnNi below 1.5 mils on 200 ksi steel)
- Commercial partners will follow guidance from the aviation authority in implementing targeted applications deemed safe.
BACKUP MATERIAL
Acronyms and Symbols

- HE - Hydrogen Embrittlement
- NFS - Notch Fracture Strength
Prior and Leveraged Work

- Boeing Ruggedness Study
  - Aimed at establishing which factors were most important
    - Surface condition plated or bare
    - Notch condition plated or bare
    - Solution Volume
    - Solution Temperature
    - Solution Concentration
    - Exposure Time
    - Exposure Temperature

- Boeing Risk Reduction Study
  - 1a1 and 1d geometries at 519 strength and load levels
  - Assessment of NaCl solution merit, low strength material procedure

- SPOTA/ARL for re-machining and Aerospace Grade material purchase

- ASTM Committee and coordination work - unfunded

- ASKO Plating for developmental work

- Boeing and ARL labor to date
Publications


