Development and qualification of S53 ultrahigh-strength corrosion resistant steel for cadmium replacement

JCAT
January 26, 2006 – San Diego, CA
Charlie Kuehmann
**4. TITLE AND SUBTITLE**

Development and qualification of S53 ultrahigh-strength corrosion resistant steel for cadmium replacement

**5. AUTHOR(S)**

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**9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)**

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Approved for public release; distribution unlimited

**15. SUBJECT TERMS**

**16. SECURITY CLASSIFICATION OF:**

<table>
<thead>
<tr>
<th>a. REPORT</th>
<th>b. ABSTRACT</th>
<th>c. THIS PAGE</th>
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</thead>
<tbody>
<tr>
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</tr>
</tbody>
</table>

**17. LIMITATION OF ABSTRACT**

Same as Report (SAR)

**18. NUMBER OF PAGES**

28
Drivers

Issues:
- Over $200 million spent in LG per year
  - 80% corrosion related
- SCC failures
- Cad plating used to protect current steel known carcinogen (Hill AFB ~ 2000 lbs/yr)

Benefits:
- Dramatic reduction in LG cost (60%)
- Savings of $120 million per year
- Significant reduction in SCC failures
- Cadmium plating not required
- General corrosion mitigated
- 80% of Steel Condemnations Avoided

Materials By Design®
Milestones and Plans

December 1999 - SERDP SEED START

Nov 00 - Series 1 & 2 designs demonstrate tensile properties in 300 lb heats.

Jan 2000 - Series 1 & 2 designs demonstrate tensile properties in 300 lb heats.

June 2001 - SERDP PHASE I START

June 02 - Series 6 designs demonstrate mechanical property goals

March 2003 - ESTCP PROGRAM START

Jan 01 - Series 3 designs determine melt practice specifications

June 03 - S53A demonstrates scale-up of tensile properties.

Jan 02 - Series 3 designs determine melt practice specifications

June 03 - S53A demonstrates scale-up of tensile properties.

March 2003 - ESTCP PROGRAM START

Feb 06 - Forging demonstration complete.

Dec 05 - 1st component completed.

Feb 06 - 3rd S53D heat complete.

Jan 05 - 1st production S53D meets specification and quality tests. 2nd heat ships Feb 05.

March 2003 - ESTCP PROGRAM START

Feb 06 - 3rd S53D heat complete.

Nov 06 - 2nd component qualification complete.

June 07 - JTP testing complete.

Jan 2007 - ESTCP Program Complete

Materials By Design®
S53 System Flow-Block Diagram

**PROCESSING**
- TEMPERING
- SOLUTION TREATMENT
- HOT WORKING
- SOLIDIFICATION
- DEOXIDATION
- REFINING

**STRUCTURE**
- **Matrix**
  - Lath Martensite: M₆ ≥ 200°C
  - Ni: Cleavage Resistance
  - Co: SRO Recovery Resistance
  - Cr: Corrosion Resistance
- **Strengthening Dispersion**
  - (Cr,Mo,V,Fe)₂C
  - Avoid Fe₃C, M₆C, M₇C₃, M₂₃C₆
- **Passive Film Formation**
  - Cr partitioning into oxide film
  - Eₚp and iₘᵢᵣ
- **Microsegregation**
  - Cr, Mo, V
- **Grain Refining Dispersion**
  - d/f
  - Microvoid Nucleation Resistance
- **Grain Boundary Chemistry**
  - Cohesion Enhancement: B, Re
  - Impurity Gettering: La, Ce

**PROPERTIES**
- **Strength**
  - σₜₚ ≥ 280 ksi
  - σₚ ≥ 230 ksi
- **Aqueous Corrosion Resistance**
  - ≥15-5PH
- **Stress Corrosion Resistance**
  - KᵢSCC ≥ 30 ksi/√in
- **Fatigue Resistance**
  - ≥ 300M
- **Core Toughness**
  - K₁C ≥ 50 ksi/√in

*Materials By Design®*
S53 Processing Schematic

- **Temperature, °C**
- **Carbon, wt. %**

- **fcc**
- **fcc + MC**
- **fcc + MC + M₁₃C₃**
- **bcc + fcc + MC**
- **bcc + fcc + MC + M₁₃C₃ + M₂₃C₆**
- **bcc + MC + M₁₃C₃ + M₂₃C₆**

- **Room Temperature**
- **Homogenize**
- **MC**
- **Normalized**
- **Solution Heat Treatment**
- **Anneal**
- **Air Cool**
- **Air Cool**
- **Air Cool**
- **Cryogenic Treatment**

**Materials**
- Fine grain material with predominantly MC grain refining carbides
- Machinable residual stress free stock
- Lath martensite with nanoscale M₂₃C strengthening carbides
ESTCP Program Objectives

- 3 Commercial scale heats
- Identify initial implementation components
- Qualification testing for AMS (S-basis) allowables
  - Execution of Joint Test Protocol (JTP)
  - Estimate MMPDS A & B-basis allowables by AIM
- Specifications for manufacturing process
  - Alloy Production
  - Forging
  - Rough Machining
  - Heat Treatment
  - Finish Machining/Surface Preparation
- Cost/Benefit Analysis
- Future Implementation Plan
ESTCP Production Line-up

Primary VIM
- 20" ingot (7 tons)

Secondary VAR – 24 in.

Homogenization
- 24" ingot (7 tons)

Press Forge
- ~ 16" DOC (2.5 tons)

Bar Forging
- ~ 20" ingot (7 tons)

Bar Rolling
- ~ 24" ingot (7 tons)

JTP
- ~ 16" DOC (2.5 tons)

Demo Plan
- 4" RCS (~2000 lbs)
- 8" RND (~2000 lbs)
- ~ 8.5" RND (2500 lbs)
- ~ 16" DOC (2.5 tons)
S53A Scale-up Properties

<table>
<thead>
<tr>
<th>Property</th>
<th>S53A (300 pound heat)</th>
<th>S53A (3,000 pound heat)</th>
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<tbody>
<tr>
<td>YS [ksi]</td>
<td>225</td>
<td>225</td>
</tr>
<tr>
<td>UTS [ksi]</td>
<td>285 285</td>
<td></td>
</tr>
<tr>
<td>El. [%]</td>
<td>16 15</td>
<td></td>
</tr>
<tr>
<td>RA [%]</td>
<td>63 59</td>
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</tr>
</tbody>
</table>
S53 Nanostructured UHS Stainless Results

Typical Fracture Toughness $K_{IC}$, L-T (KSI/N$^{1/2}$) vs. Ultimate Tensile Strength (KSI)

- 15-5PH
- 13-8Mo
- Custom 465
- AF 1410
- AerMet 100
- Ferrium S53
- 4340/300M
## Baseline Data

### Ideal Heat Treatment Condition:

1100°C 70 min + OQ + -78°C 1 hr + AW(RT) + 505°C 3 hrs + WQ  
+ -78°C 1 hr + AW(RT) + 492°C 12 hrs + AC

<table>
<thead>
<tr>
<th>Material Code</th>
<th>Configuration</th>
<th>0.2% YS</th>
<th>UTS</th>
<th>CVN</th>
<th>K\textsubscript{IC}</th>
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</thead>
<tbody>
<tr>
<td>209126</td>
<td>8&quot; round</td>
<td>229.4</td>
<td>288.5</td>
<td>21</td>
<td>67</td>
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<tr>
<td></td>
<td>Transverse</td>
<td>230.6</td>
<td>285.0</td>
<td>20</td>
<td>74</td>
</tr>
<tr>
<td></td>
<td>4&quot; RCS</td>
<td>229.8</td>
<td>285.3</td>
<td>19</td>
<td></td>
</tr>
<tr>
<td>209193</td>
<td>8&quot; round</td>
<td>222.3</td>
<td>287</td>
<td>23</td>
<td></td>
</tr>
<tr>
<td></td>
<td>4&quot; RCS</td>
<td>223.7</td>
<td>287.2</td>
<td>24</td>
<td></td>
</tr>
</tbody>
</table>
Heat Treatment Conclusions

- Cryo not necessary to go all the way to liquid nitrogen
  - 0.2% YS  |  UTS  |  CVN
  -78°C  |  229.4 |  288.5 |  21
  -196°C |  227.1 |  288.1 |  21

- Up to 8 hours after quench is acceptable before cryo-treatment
  - 0.2% YS  |  UTS  |  CVN
  1 hour    |  225.1 |  287.9 |  22
  5 hours   |  221.6 |  289.1 |  22
  8 hours   |  231.3 |  284.4 |  20
  24 hours  |  214.7 |  285.4 |  23
Successful scale-up: Fatigue of Production S53 alloy product forms from large Heat meet or exceed the 300M alloy fatigue trendline (L & T) of MIL-HDBK-5, and Longitudinal data matches L data of SERDP program’s S53 alloy R&D small Heat.

ESTCP Program on Ferrium S53 Corrosion Resistant Steel: $R = -0.33$ Fatigue Data: for 300M ($Su = 284$ ksi) & S53-6F ($Su = 291$ ksi) (from 300 lbs Heat) of SERDP/2003 and production grade S53 ($Su = 284$ ksi) (from 10,000 lbs Heats).

Compare with S/N Curve Trendline for unnotched unpeened 300M ($Ftu=280$ ksi) of MIL-HDBK-5.
Sensitivity Analysis

- +/- 7°C in temperature (solution and tempering)
- +/- 30 minutes in time (solution and tempering)
- Represents 28 samples
Corrosion Results from Anodic Polarization

Overall Corrosion Rate (mpy)

- 300M: 7.0
- S53-2A: 0.52
- S53-2C: 0.40
- S53-4A: 0.45
- S53-4B: 1.05
- S53-4D: 1.12
- S53-4F: 0.62
- 15-5 PH: 0.26
- S53A: 0.56
- S53-6F: 0.33
- S53-3A Peak: 0.51
- S53-3A Stage I: 0.38
- S53-4A Overage: 0.88
$K_{ISCC}$ Results of Ferrium™ S53 vs 4340
Weld Microstructure & Mechanical Properties

<table>
<thead>
<tr>
<th></th>
<th>Base Metal</th>
<th>Weld Metal</th>
</tr>
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<tbody>
<tr>
<td>UTS (ksi)</td>
<td>276</td>
<td>275</td>
</tr>
<tr>
<td>YS (ksi)</td>
<td>226</td>
<td>220</td>
</tr>
<tr>
<td>Elong.%</td>
<td>15</td>
<td>9</td>
</tr>
<tr>
<td>CVN (ft-lbs.)</td>
<td>7</td>
<td>6</td>
</tr>
</tbody>
</table>

Study completed on S53A heat HC56 rejected for high N content.
Annealed S53 Machinability Evaluations

- Turning annealed Ferrium S53 at 38 to 40 HRC is harder than turning 300M and AerMet 100 in the normalized and annealed condition.
- Ferrium S53 hardens during turning inducing an unusual wear of the inserts.
- Lower speed is needed to have a reasonable inserts wear.
- A very important deformation (TIR) was noted on a 7.75" bar (0.040"), even with a low speed, which is not acceptable.
- Feed is found to be the most critical parameter to decrease the deformation (TIR) of the bars. Feeds as low as 0.006” are needed (compared with 0.012” for Aermet 100).
- Very good finishes after turning could be reached (34 Ra) with the most performing inserts.
- S53D Spec. incorporates a cryo treatment to address high annealed hardness and high work hardening rate – initial results are positive.

<table>
<thead>
<tr>
<th>P/N</th>
<th>Dimension</th>
<th>Number of parts</th>
<th>Interrupted turning</th>
<th>Continuous turning</th>
<th>Drilling</th>
<th>Tapping</th>
</tr>
</thead>
<tbody>
<tr>
<td>SK-0110</td>
<td>3.5” x 3.5” x 7.75”</td>
<td>6</td>
<td>√</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SK-0112</td>
<td>3.5” x 3.5” x 7.75”</td>
<td>5</td>
<td>√</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SK-0113</td>
<td>3.5” x 3.5” x 23”</td>
<td>1</td>
<td>√</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SK-0114</td>
<td>3.5” x 3.5” x 7.75”</td>
<td>2</td>
<td>√</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SK-0115</td>
<td>3.5” x 3.5” x 7.75”</td>
<td>2</td>
<td>√</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SK-0116</td>
<td>3.5” x 3.5” x 17.3”</td>
<td>1</td>
<td></td>
<td></td>
<td>√</td>
<td></td>
</tr>
<tr>
<td>SK-0117</td>
<td>0.7” x 6” x 24”</td>
<td>2</td>
<td></td>
<td></td>
<td></td>
<td>√</td>
</tr>
</tbody>
</table>
Production S53 Annealed Machinability
S53 Fully Hardened Machining Evaluations

Threaded S53 sample piece

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Insert</th>
<th>Speed (SFM) (1)</th>
<th>Infeed (Inch per Revolution)</th>
<th>Depth of cut (inch)</th>
<th>BNI/Finish (2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>300M</td>
<td>Carbide KC5010</td>
<td>160R</td>
<td>0.010</td>
<td>0.075</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>180F</td>
<td>0.008</td>
<td>0.030</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Ceramics</td>
<td>550R/F</td>
<td>0.006</td>
<td>0.030</td>
<td></td>
</tr>
<tr>
<td>Aermet 100</td>
<td>Carbide</td>
<td>N/A</td>
<td>N/A</td>
<td>N/A</td>
<td></td>
</tr>
<tr>
<td></td>
<td>160F</td>
<td>0.010</td>
<td>0.010</td>
<td>A/58-64Ra</td>
<td></td>
</tr>
<tr>
<td>Ferrium S53</td>
<td>Carbide KC5010/positive</td>
<td>180F</td>
<td>0.008</td>
<td>0.015</td>
<td>A/55-71Ra</td>
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<tr>
<td></td>
<td>Carbide KC7310/positive</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td>Carbide KC5010 OR EH510Z/positive</td>
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<tr>
<td></td>
<td>Carbide KC5010/positive</td>
<td>90F</td>
<td>0.006</td>
<td>0.015</td>
<td>A/15-16Ra</td>
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Grinding Results
- S53 is very difficult to induce grind damage
- Grind burns could not be detected with a standard nital etch
- New etchant needs to be developed

<table>
<thead>
<tr>
<th>P/N – Dia (inch)</th>
<th>Number of parts</th>
<th>Turning</th>
<th>Threading</th>
<th>Drilling</th>
</tr>
</thead>
<tbody>
<tr>
<td>SK-0110 – 1.50</td>
<td>6</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SK-0111 – 1.75</td>
<td>1</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SK-0112 – 3.00</td>
<td>5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SK-0113 – 3.00</td>
<td>1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SK-0114</td>
<td>2, three diam</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SK-0115 – 3.80</td>
<td>2</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SK-0117 – plate</td>
<td>2</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

-Parts prepared for the grinding trials

Turning Results

<table>
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<td>1</td>
<td>√</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SK-0112 – 3.00</td>
<td>5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SK-0113 – 3.00</td>
<td>1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>SK-0114</td>
<td>2, three diam</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
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<td>2</td>
<td></td>
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<tr>
<td>SK-0117 – plate</td>
<td>2</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

-Parts prepared for the grinding trials
Forging Study

A10 MLG Piston
Kropp Forge
5/17/05

Takeaways:
• Forges easily
• Forges better than AerMet
• Minimal (if any) change in mechanical properties
A10 Forging Characterization

• **Mechanical**

<table>
<thead>
<tr>
<th></th>
<th>0.2% YS</th>
<th>UTS</th>
<th>CVN</th>
</tr>
</thead>
<tbody>
<tr>
<td>8&quot; Bar Stock</td>
<td>229.4</td>
<td>288.5</td>
<td>21</td>
</tr>
<tr>
<td>A10 Forging</td>
<td>233.7</td>
<td>284.2</td>
<td>18</td>
</tr>
</tbody>
</table>

Decarb approximately 0.060” (1500 μm)

Decarb approximately 0.060” (1500 μm)
**Demonstration Target Components**

### A-10 Main Landing Gear
- A-10 main landing gear piston (4330 – 240 ksi)
  - More complex loading
  - Forged component
  - Currently in production for spares

### A-10 Nose Gear
- A-10 drag brace (300M - 270 ksi)
  - Simple tension loading
  - No forging required
  - Corrosion related failures
Accelerating the Materials Development Cycle

Materials by Design™

- Concept
  - Design
    - Prototype
      - Meet Objectives?
        - Yes: Specify Processing
        - No: A

- Process Optimization
  - Full Scale Heat
  - Meet Objectives?
    - Yes: Production Heat
    - No: A

- Design Data
  - Meet Objectives?
    - Yes: Sample Production
    - No: A

- Application & Process Design
  - Meet Objectives?
    - Yes: Implementation
    - No: A

AIM Methodologies

Materials By Design®
Probabilistic-Property-Prediction ($P^3$) Roadmap

- **Legacy Data**
  - Supplier
  - Heat Treaters
  - $\Delta x_i$, $\Delta T_s$, $\Delta T_t$, $\Delta t$

- **Prototype Data**
  - $\sigma(X_i, T_s, T_t, t)$

- **Initial Models**
  - Trajectory Model

- **Revised Modeling**

- **Focused Testing**

- **Monte Carlo Estimate $P(\sigma)$**

- **Qualification Testing**

- **H-BayesCalc**

- **Handbook Design Allowables Prediction**

- **iSIGHT Prediction**
S53 Robust/Sensitivity Analysis with Compositional Variations

**Compositional Variations** (wt%, ±6σ):
- C ± 0.01
- Cr ± 0.2
- Mo ± 0.1
- W ± 0.1
- Co ± 0.3
- Ni ± 0.1
- V ± 0.02

**Variations of:**
- **Structure** — carbide solvus Ts, martensite Ms, precipitation control ΔG’s
- **Property** — hardness HRC, toughness CVN

Results of 1000 runs (12 minutes on a Pentium IV 2.2GHz CPU)

- Probability Distribution for CVN
  - mean val = 46.7653
  - std dev = 1.1687

- Probability Distribution for DG(M2C)
  - mean val = 15.6608
  - std dev = 0.0310322

- Probability Distribution for DG(Intermetallic)
  - mean val = 3.61054
  - std dev = 0.3549247

- Probability Distribution for MS
  - mean val = 182.145
  - std dev = 1.114147

- Probability Distribution for HRc
  - mean val = 53.66986
  - std dev = 0.164583

- Probability Distribution for TS
  - mean val = 1020.15
  - std dev = 1.21442
ESTCP AIM Objectives

- Objective is to predict MIL-HBK 5 “A”- Allowables with only 3 heats available.
- Designers can design new LG components with confidence 3-5 years earlier.
- Testing costs are 70% lower, overall costs are 50% lower.

<table>
<thead>
<tr>
<th>Property</th>
<th>Mean value</th>
<th>+3σ</th>
<th>-3σ</th>
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</thead>
<tbody>
<tr>
<td>AMS Specification</td>
<td>AIM Predictions</td>
<td>MIL-HBK 5 “A”- Allowables</td>
<td></td>
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</table>

3 10
Summary and Takeaways

- S53 has demonstrated property goals in multiple production scale heats.
- Primary manufacturing evaluations have been completed for machining, surface treatments, and welding.
- Yield stress is the property most sensitive to process variation.
- AIM methods will predict MMPDS (MIL-HNBK-5) A-allowables with 3 heats completed.
- First applications to be completed for Air Force replacement requirements. A-10, 2007-2008