Optimization of Advanced Steels for Cyclic Operation through an Integration of Material Testing, Modeling and Novel Component Test Validation
DE-FE002620

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Key Collaborator, Task 2: Wyman Gordon, Ian Dempster and Tom Armstrong
Key Collaborator, Task 6: Babcock & Wilcox, Dave Dewees
Key Collaborator, Task 7: Oak Ridge National Laboratory, Amit Shyam

Crosscutting Research and Rare Earth Elements Portfolios Review
April 21, 2016
Setting the Stage for World Power Market

- **USA**
  - There is immense commercial pressure to increase efficiency in state-of-the-art combined cycle gas turbine (CCGT) plants.
  - Within the immediate future, steam outlet temperatures in heat recovery steam generators (HRSGs) will achieve >600°C.

- **Asia**
  - There is immense commercial pressure to increase efficiency in coal fired power plants (i.e. today’s ultra-supercritical power plants are operating with steam outlet temperatures approaching 625°C).
  - Steam outlet temperatures in advanced ultra-supercritical power plants are being planned within the range of 700 to 760°C.

Regardless of end-use application there is an increasing need for materials with optimized thermal/creep properties and with a minimized cost impact ➔ CSEF steels.
Background, A Summary of >5 Years of EPRI Research in Grade 92 Steel
Grade 92 ➝ 9CrWNbVNB CSEF Steel

Not all Grade 92 is the same

<table>
<thead>
<tr>
<th>Gr92 Heat</th>
<th>Co</th>
<th>Cr</th>
<th>Ni</th>
<th>P</th>
<th>Mn</th>
<th>Mo</th>
<th>Si</th>
</tr>
</thead>
<tbody>
<tr>
<td>BM A</td>
<td>0.015</td>
<td>8.797</td>
<td>0.38</td>
<td>0.009</td>
<td>0.49</td>
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<tr>
<td>BM B</td>
<td>0.016</td>
<td>8.776</td>
<td>0.25</td>
<td>0.012</td>
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<td>0.33</td>
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<tr>
<td>BM C</td>
<td>&lt;0.001</td>
<td>8.939</td>
<td>0.19</td>
<td>0.009</td>
<td>0.40</td>
<td>0.43</td>
<td>0.252</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Gr92 Heat</th>
<th>B</th>
<th>C</th>
<th>N</th>
<th>Nb</th>
<th>V</th>
<th>W</th>
</tr>
</thead>
<tbody>
<tr>
<td>BM A</td>
<td>0.0041</td>
<td>0.113</td>
<td>0.045</td>
<td>0.062</td>
<td>0.188</td>
<td>1.836</td>
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<tr>
<td>BM B</td>
<td>0.0041</td>
<td>0.131</td>
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<td>0.056</td>
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<td>1.617</td>
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<tr>
<td>BM C</td>
<td>0.0042</td>
<td>0.093</td>
<td>0.0508</td>
<td>0.054</td>
<td>0.189</td>
<td>1.794</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Gr92 Heat</th>
<th>Al</th>
<th>As</th>
<th>Cu</th>
<th>O</th>
<th>S</th>
<th>Sb</th>
<th>Sn</th>
</tr>
</thead>
<tbody>
<tr>
<td>BM A</td>
<td>0.002</td>
<td>0.0064</td>
<td>0.189</td>
<td>0.0053</td>
<td>0.008</td>
<td>0.0016</td>
<td>0.016</td>
</tr>
<tr>
<td>BM B</td>
<td>0.015</td>
<td>0.0082</td>
<td>0.135</td>
<td>0.0022</td>
<td>0.001</td>
<td>0.001</td>
<td>0.008</td>
</tr>
<tr>
<td>BM C</td>
<td>0.001</td>
<td>&lt;0.0001</td>
<td>0.001</td>
<td>0.0043</td>
<td>0.001</td>
<td>&lt;0.0001</td>
<td>&lt;0.001</td>
</tr>
</tbody>
</table>

Also analyzed, with no detected amount: Pb, Bi, La, Nd, Ta, Ti, Zr and Ca ≤0.002
The Trends in Controlled Composition is Observed in Grade 92 \([650^\circ C/90 \text{ MPa}]\)

- **BM A**
  - 3,945 hours
  - 26.0% ROA

- **BM B**
  - 8,593 hours
  - 16.3% ROA

- **BM C**
  - 14,190 hours
  - 42.4% ROA

3.6X increase in strength; Observable increase in ductility
Comparison of Creep Ductility for a Damage Susceptible Heat of Grade 92

Time to Rupture for all Specimens ~26k hours

State-of-the-art designs will operate in temperature regimes ≥600°C
Recent EPRI Database Development

- EPRI has evaluated performance of Grade 92 steel for three unique base materials (the previously mentioned “BM A”, “BM B” and “BM C”). Testing included
  - Uniaxial creep
  - **Notch bar creep (two notch designs), and interrupted**
  - Creep-fatigue to ASTM E2714-09 with various hold times, strain rates, test temperatures, etc.
  - Cyclic stress relaxation
  - Fatigue
  - Cross-weld tests including feature tests in selected heats

- >500k hours of testing and ~$3 million in leverage from industry and collaborative partners
Research has shown the value in notch bar creep testing as a feature test which is relevant to the development of damage in power plant components. There is a clear transition to principal stress controlled damage (i.e., initiation and linking of creep cavities).
Research has shown the value in notch bar creep testing as a feature test which is relevant to the development of damage in power plant components.

An upper-bound strength, super-clean heat (BM C) shows a distinct offset to principal stress controlled cavitation damage.
The Concern Regarding Low Ductility

- Traditionally in material development for the power industry, creep ductility (i.e. resistance to damage) has been given little consideration.

- However, the risk to catastrophic failure due to low damage tolerance is a reality where:
  - Mechanical notches exist due to poor design considerations
  - Metallurgical notches exist to fabrication (i.e. weldments)

- The realities regarding creep ductility, even when investigated, are often improperly identified:
  - Uniaxial creep versus notch tests
  - “Small sample” cross-weld versus feature cross-weld tests

Where CSEF steels possess high susceptibility to damage, these grades or heats trend to NOTCH WEAKENING behavior where proper testing produces relevant results.
The macro-level assessment is critical to validating models to apply to in-plant components. Since the model predicts a trend to cavitation-dominated damage, it is important to evaluate damage from a micro-level using advanced electron optics and confirm this observation.
Micro-level Evaluation of Grade 92 Base Material Samples indicates that Damage is NOT Isolated to Prior Austenite Grain Boundaries in these Steels

- Damage must be forming in or on multiple features since damage is clearly within the grain and on grain boundary
- This contradicts much of the literature suggesting:
  - Poor characterization in most assessments
  - Potential for multiple damage modes where the reported results are from a pedigreed researcher
Micro-level Evaluation of Grade 92 Base Metal Damage shows a Strong Correlation with Boron-nitride (BN) and a Weaker Correlation with MnS and Alumina
Confirmation of Cavitation was Performed for Each Evaluated Heat of Grade 92 Steel

Subsurface cavity identified in SEM using high accelerating voltage

To confirm the link to BN, sub-surface cavities such as in (a) were removed by focused-ion beam milling. BN is confirmed in (b) by EDS.
All Heats of Grade 92 are Susceptible to the formation of BN (hence the development of MARBN-type steels such as SAVE 12 AD and others) – From Abe Diagram
Quick Summary

- Eliminating the presence of BN in Grade 92 steel is important to reduce the “easy” nucleation sites and delay the transition to principal stress controlled damage
  - Accomplished by an unconventional, high temperature normalization ~1150°C
  - And “sufficiently fast” cooling rate to avoid BN reformation

- Optimizing an existing, Code-approved material is desirable from a practical and database standpoint
  - Currently, Grade 92 is the highest strength approved CSEF steel in ASME Code (Code Case 2179)
  - Material is no longer exclusive to a single manufacturer and the supply chain already exists for manufacturing the needed components:
    - Castings
    - Fittings
    - Plate
    - Pipe
    - Tube
On the Reduction/Removal of BN from the Matrix

- All of the evaluated Grade 92 steels show damage associated with BN
- Dissolution of BN is accomplished through a sufficient peak temperature (see table on right)
- Preventing the re-formation of BN is believed to be a cooling rate dependent issue (i.e. may need to impose water-, oil- or accelerated air-cooling)
- Additionally, an “unconventionally” high normalization and cooling can lead to optimized precipitate structure

<table>
<thead>
<tr>
<th>Temperature (Time = 2 hours for all conditions)</th>
<th>Result¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>1200</td>
<td>BN Fully Dissolved</td>
</tr>
<tr>
<td>1175</td>
<td></td>
</tr>
<tr>
<td>1150</td>
<td></td>
</tr>
<tr>
<td>1125</td>
<td></td>
</tr>
<tr>
<td>1100</td>
<td>Not Dissolved</td>
</tr>
</tbody>
</table>

¹Simulated experiments for BMA using a Gleeble Thermomechanical Simulator

For Grade 92, proper normalization needs to be conducted at 1125°C minimum and 1150°C target
An Interesting and Relevant Point on Normalization from Code Case Data Package for SAVE 12AD
Note: SAVE 12AD is a 9Cr-3W-3Co-Nd-B (Nb, V, N)

<table>
<thead>
<tr>
<th>Steel</th>
<th>Heat</th>
<th>Product Form</th>
<th>Dimensions (mm)</th>
<th>Heat Treatment</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>Heat1</td>
<td>Plate</td>
<td>t15</td>
<td>1150°C x1h AC → 780°C x 1h AC</td>
</tr>
<tr>
<td>S2</td>
<td>Heat2</td>
<td>Plate</td>
<td>t15</td>
<td>1150°C x1h AC → 780°C x 4h AC</td>
</tr>
<tr>
<td>S3</td>
<td>Heat2</td>
<td>Plate</td>
<td>t15</td>
<td>1150°C x2h AC → 780°C x 4h AC</td>
</tr>
<tr>
<td>S4</td>
<td>Heat3</td>
<td>Plate</td>
<td>t15</td>
<td>1150°C x1h AC → 780°C x 4h AC</td>
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<tr>
<td>S5</td>
<td>Heat3</td>
<td>Plate</td>
<td>t15</td>
<td>1150°C x2h AC → 780°C x 4h AC</td>
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<td>S6</td>
<td>Heat4</td>
<td>Plate</td>
<td>t15</td>
<td>1150°C x1h AC → 780°C x 4h AC</td>
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<tr>
<td>S7</td>
<td>Heat4</td>
<td>Plate</td>
<td>t15</td>
<td>1150°C x2h AC → 780°C x 4h AC</td>
</tr>
<tr>
<td>S8</td>
<td>Heat5</td>
<td>Plate</td>
<td>t15</td>
<td>1150°C x1h AC → 780°C x 4h AC</td>
</tr>
<tr>
<td>S9</td>
<td>Heat5</td>
<td>Plate</td>
<td>t15</td>
<td>1150°C x2h AC → 780°C x 4h AC</td>
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<td>S10</td>
<td>Heat6</td>
<td>Plate</td>
<td>t15</td>
<td>1150°C x1h AC → 780°C x 4h AC</td>
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<tr>
<td>S11</td>
<td>Heat6</td>
<td>Plate</td>
<td>t15</td>
<td>1150°C x2h AC → 780°C x 4h AC</td>
</tr>
<tr>
<td>S12</td>
<td>Heat7</td>
<td>Plate</td>
<td>t25</td>
<td>1150°C x1h AC → 780°C x 4h AC</td>
</tr>
<tr>
<td>S13</td>
<td>Heat8</td>
<td>Plate</td>
<td>t25</td>
<td>1150°C x1h AC → 780°C x 4h AC</td>
</tr>
<tr>
<td>T1</td>
<td>Heat9</td>
<td>Tube</td>
<td>38OD × 8.8WT</td>
<td>1150°C x10min AC → 780°C x 2h AC</td>
</tr>
<tr>
<td>T2</td>
<td>Heat10</td>
<td>Tube</td>
<td>80OD × 20WT</td>
<td>1150°C x1h AC → 780°C x 4h AC</td>
</tr>
<tr>
<td>T3</td>
<td>Heat11</td>
<td>Tube</td>
<td>45OD × 8.5WT</td>
<td>1150°C x10min AC → 780°C x 3h AC</td>
</tr>
<tr>
<td>P1</td>
<td>Heat12</td>
<td>Pipe</td>
<td>350OD × 50WT</td>
<td>1150°C x1h AC → 780°C x 3h AC</td>
</tr>
<tr>
<td>P2</td>
<td>Heat13</td>
<td>Pipe</td>
<td>350OD × 40WT</td>
<td>1150°C x30min AC → 780°C x 6h AC</td>
</tr>
<tr>
<td>P3</td>
<td>Heat14</td>
<td>Pipe</td>
<td>350OD × 40WT</td>
<td>1150°C x30min AC → 780°C x 6h AC</td>
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# Grade 92 Uniaxial Creep Specimens

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Base Metal</th>
<th>Test Conditions</th>
<th>Life (hrs)</th>
<th>Elong. (%)</th>
<th>ROA (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>G92-33</td>
<td>BM A</td>
<td></td>
<td>3,945</td>
<td>11.2</td>
<td>26.0</td>
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<tr>
<td>G92M-21</td>
<td>BM B</td>
<td></td>
<td>8,593</td>
<td>8.1</td>
<td>16.3</td>
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<tr>
<td>G92J-23</td>
<td>BM C</td>
<td></td>
<td>14,190</td>
<td>11.2</td>
<td>42.4</td>
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<tr>
<td>10766-1CRP</td>
<td>BM A (ReN+T)</td>
<td></td>
<td>&gt;14,190 (on-going)</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- As a simple experiment, the poorest performing material (in terms of strength and ductility, BM A) was re-normalized at 1200°C/1h/WQ and tempered (777°C/1h/AC)
- To date, this material exceeds performance [strength] as compared to BM C. Ductility/damage will be confirmed upon failure
Comparison of Grade 92 Uniaxial Creep Specimens

BM A

BM B

BM C

Time to Rupture (hours)

Strain (%)

650°C, 90 MPa

BM A, ReN+T (on-going)
1200°C/1h/WQ + 777°C/1h/AC
Based on this information and promising approaches for modeling and material heat treatment optimization, EPRI was awarded a project sponsored by DOE.
Motivation for EPRI Project

- Increase the resistance to damage [i.e. creep ductility]
  - We can potentially increase the creep ductility in Grade 92 steel by:
    - Reducing the void nucleation sites (i.e. remove BN)
    - Delay the transition to principal stress controlled damage (i.e. very clean composition)

- Increase the deformation resistance [i.e. creep strength]
  - We can potentially increase strength in Grade 92 steel by:
    - Optimizing the type of precipitate
    - Optimizing the precipitate composition

- Phase I – optimized heat treatment of a commercial heat of Grade 92
- Phase II – optimized heat treatment of a “super clean” commercial heat of Grade 92
If both the Composition and Processing are Optimized for Grade 92, we may reasonably Expect Performance within the Scatter-band for SAVE12AD

Heat Treatment may also be effective in delaying the transition to principal stress controlled damage
Tasks

- Task 2.0 – P92 Alloy Procurement and Processing [Wyman]
- Task 4.0 – Laboratory Scale Creep, Creep and Thermal Cycling Testing of P92 Samples
- Task 5.0 – Microstructural Evaluation of Initial Material, Heat Treatments and as-Tested Samples
- Task 6.0 – Development of Constitutive Equations, Creep-Fatigue Models and Design of a Phase II Pressure Vessel Component Test [Babcock & Wilcox]
- Task 7.0 – Design and Fabrication of a Structural Feature Scale Creep-Fatigue Test [ORNL]
Task 2.0 – P92 Alloy Procurement and Processing

- Donated pipe measures 20” OD X 5.27” WT X 4 feet long
- One section left in the as-manufactured state
  - Normalization = 1065°C for 2.75 hours + Fan Cool
  - Tempering = 775°C for 5.5 hours
- A second section given an optimized heat treatment
  - Normalization = 1125°C (2055°F) for 2 hours minimum (Target 1150°C) + Oil Quench
  - Tempering = 775°C (1425°F) for 5 hours

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>Ti</th>
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<tbody>
<tr>
<td>0.084</td>
<td>0.47</td>
<td>0.008</td>
<td>0.0013</td>
<td>0.238</td>
<td>0.17</td>
<td>8.693</td>
<td>0.43</td>
<td>0.192</td>
<td>&lt;0.002</td>
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<table>
<thead>
<tr>
<th>Co</th>
<th>W</th>
<th>Nb</th>
<th>B</th>
<th>N</th>
<th>Al</th>
<th>As</th>
<th>Cu</th>
<th>Sb</th>
<th>Sn</th>
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<tbody>
<tr>
<td>0.014</td>
<td>1.86</td>
<td>0.064</td>
<td>0.0023</td>
<td>0.0480</td>
<td>0.002</td>
<td>0.004</td>
<td>0.152</td>
<td>0.0012</td>
<td>0.007</td>
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</tbody>
</table>

*Also analyzed, with no detected amount: Pb, Bi, La, Ta, Zr and Ca ≤0.002*
Task 4.0 – Planned Testing is Underway

- Plain bar creep tests on optimized Grade 92
- Notch bar creep tests on optimized Grade 92
- Sequential Fatigue-Creep tests
  - Low cycle fatigue + Creep
  - Creep + Tensile
- Simulated HAZ plain bar creep tests on conventional and optimized Grade 92
- Feature Cross-weld Creep tests
- [ORNL Task 7.0] Pressurized and pressurized + end load tests
Comparison of Feature, Cross-weld Tests for Task 4.5 to Conventional Uniaxial Round Bar Sample

Feature Type Tests

Full Tube Test

Standard Round Bar Test

Full tube test 85X
Large, feature tests 20X
Standard round bar test 1X*

*Indicates the increase in gauge cross-sectional area
Cross-weld Creep Feature Test Samples

- Weldment tests made in the oil quenched produced pipe
- Single vee weld (30° included angle) SMAW process + Grade 92-type filler metal and subcritical PWHT at 750°C/2h
Task 5.0 – Microstructural Evaluation of Initial Material, Heat Treatments and as-Tested Samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>1150°C/1h/WQ</th>
<th>750°C/1h/AC</th>
<th>900°C/1m/AC</th>
<th>750°C/1h/AC</th>
</tr>
</thead>
<tbody>
<tr>
<td>M1</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>M2</td>
<td></td>
<td></td>
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</tr>
<tr>
<td>M3</td>
<td></td>
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</tr>
<tr>
<td>M4</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>M5</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>M6</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>M7</td>
<td>As-received (1065°C/2.75h/FAC + 775°C/5.5h/AC)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>COMM-1</td>
<td>Optimized (1150°C/2h/OQ + 775°C/5h/AC)</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- Initial characterization to include:
  - Macro hardness measurements
  - PAGB and substructure
  - $M_{23}C_6$ and MX
  - BN dissolution
  - Cooling rate experiments

- Post-test characterization once samples begin to fail using similar methods for direct comparison
Summary

- Resistance to deformation (i.e. creep strength) can be realized by an optimized heat treatment in 9Cr CSEF steels
  - It is not yet clear the role heat treatment will play in the resistance to damage, although the intent of the optimized heat treatment is to simultaneously modify both properties
  - In the minimum, it may be possible to delay the onset to principal stress controlled damage through heat treatment alone

- Demonstration of improved performance must be demonstrated by a combination of critical tests:
  - Notch bar creep tests
  - Feature tests in cross-weld creep
  - Multi-axial tube tests with pressure and end load

- The development of suitable CDM approaches for Grade 92 (conventional and optimized) are vital for potential Phase II component testing and design of components from optimized materials
Looking into the Future (Potential Phase II Examinations)

- The performance of Grade 92 (and by extension Grade 91) can be further optimized through:
  - Careful control of composition (i.e. working towards a “superclean” composition)
  - Optimized tempering heat treatment (such as 750°C versus 775°C)
  - Potential for homogenization step (such as during pipe manufacturing)

- Ultimately, it may be possible to realize very high creep strength and acceptable creep ductility through existing materials as opposed to resource-extensive material property development for new alloy concepts
Together...Shaping the Future of Electricity
Tests under Uniaxial Creep, Notch Bar Creep, Creep-Fatigue, Cyclic Stress Relaxation have been Modeled to Exercise Developed Material Descriptions to Validate Continuum Damage Mechanics Approach

Continuum Damage Mechanics Predicts Maximum Damage will be Subsurface
Detailed Macro-Assessment of Post-test Samples Includes Highly Accurate Laser Microscopy

- Area highlighted by yellow was analyzed
  - 141 Total Images
  - 20X Objective (~400X magnification)
  - Void density calculated for each image and reported in voids/mm²
  - “Black area” for some images at notch was removed

- Data reported as:
  - “Heat Map” – Grid pattern
  - “Contour Map” Overlay onto the image
Comparison of Model versus Actual Macro-
Measurements shows Excellent Agreement

The Continuum Damage Mechanics Model can be
Applied to Predictive, Component Behavior
The Effect of Normalization on Precipitate Structure
Comparison of As-received Materials – through Optimized Heat Treatment we can make BM A more like BM C

As-received

<table>
<thead>
<tr>
<th>BM A</th>
<th>BM B</th>
<th>BM C</th>
</tr>
</thead>
<tbody>
<tr>
<td>[Mean -20%]</td>
<td>[Mean]</td>
<td>[Mean +20%]</td>
</tr>
</tbody>
</table>

1200°C Normalization