Improving the Performance of Creep-Strength-Enhanced Ferritic (CSEF) Steels

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• Dr. Fujio Abe, NIMS, Japan
• Informal collaborations continue with ASME, boiler manufacturers, and EPRI
Estimated CSEF needs for construction of a High-Efficiency Boiler

- Headers & piping
  - P91/P92 – 1,000,000 lbs
- Boiler tubing
  - T23, T91, T92 Alloy Grades – 2,600,000 lbs

Purpose is to build fundamental understanding needed to maximize performance of CSEF steels

- Activities combine basic & applied R&D with strong power industry interactions
- Specific goals include:
  - Improving the structural performance of (9-12)Cr-Mo steels
  - Provide science-based guidelines for maximizing safe operating temperatures
  - Understand the fundamental causes of current temperature limitations
    - Causes of Type IV failures
    - Possible ways of minimizing/eliminating Type IV failures
Long-time weldment properties may not meet projections from short-time data

- **Type IV failure** is due to weakened microstructures in HAZs
- Weld Strength Factors (WSF = $\sigma_{\text{weld}} / \sigma_{\text{base metal}}$) for CSFE steels can be as low as 0.5 at ~600°C.
- Unpredictable behavior that causes unplanned outages, concerns about reliability & safety, more aggressive inspection procedures
Type IV failures depend on gradients of microstructures/properties in weld HAZs

- Post Weld Heat Treatment (PWHT) is applied to temper HAZ/weld metal.
- Type IV failures take place at FG/ICCHAZ, even after PWHT.
Approach to improved CSEF steels relies on two strategies

1. **Modified heat treatments:**
   - Could be effective with existing alloys
   - Implementation could be straightforward
     - ASME Code approval required

2. **Modified alloys:**
   - Newly developed alloys appear more resistant to Type IV behavior
   - Limited experience with welding
     - Behavior is not understood

- Martensitic matrix with $\text{M}_{23}\text{C}_6$ and MX
- Prior austenite grain size is from 15 to 30 microns
Contents of this presentation

1. **Modified heat treatments (Gr 91):**
   - Characterization/creep test results of PWHT samples (ORNL/OSU)
   - *In-situ* diffraction study of HAZ simulated samples (OSU)

2. **Modified alloys (Gr 92):**
   - Creep test results of Experimental 9Cr steel (ORNL/NIMS)

### Table: Chemical composition of the alloy studies

<table>
<thead>
<tr>
<th></th>
<th>Fe</th>
<th>C</th>
<th>Mn</th>
<th>Si</th>
<th>Cr</th>
<th>W</th>
<th>Mo</th>
<th>Ni</th>
<th>Co</th>
<th>V</th>
<th>Nb</th>
<th>N</th>
<th>B</th>
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<tbody>
<tr>
<td>Gr 91</td>
<td>Bal.</td>
<td>0.08</td>
<td>0.27</td>
<td>0.11</td>
<td>8.61</td>
<td>-</td>
<td>0.89</td>
<td>0.09</td>
<td>-</td>
<td>0.21</td>
<td>0.07</td>
<td>0.06</td>
<td>&lt;0.001</td>
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<tr>
<td>Gr 92</td>
<td>Bal.</td>
<td>0.09</td>
<td>0.47</td>
<td>0.16</td>
<td>8.72</td>
<td>1.87</td>
<td>0.45</td>
<td>-</td>
<td>-</td>
<td>0.21</td>
<td>0.06</td>
<td>0.05</td>
<td>0.002</td>
</tr>
<tr>
<td>N130B</td>
<td>Bal.</td>
<td>0.08</td>
<td>0.49</td>
<td>0.30</td>
<td>8.97</td>
<td>2.87</td>
<td>-</td>
<td>-</td>
<td>2.91</td>
<td>0.18</td>
<td>0.05</td>
<td>0.002</td>
<td>0.013</td>
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</tbody>
</table>
**Sample Preparation Sequence**

<table>
<thead>
<tr>
<th>Pre-weld Temper (1.5h), °C</th>
<th>PWHT (4h), °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>800</td>
<td>800</td>
</tr>
<tr>
<td>760</td>
<td>760</td>
</tr>
<tr>
<td>700</td>
<td>700</td>
</tr>
<tr>
<td>650</td>
<td>760</td>
</tr>
<tr>
<td>600</td>
<td>700</td>
</tr>
</tbody>
</table>

*(in ASME: 730-800°C) (in ASME: 730-775°C)*

**Sample IDs are described such as 650T/760 or 760T/760**

- ✓ Mechanical property screening (tensile, hardness, and creep testing)
- ✓ Metallography
Improved Tensile Properties for Lower Pre-weld Tempering Temperature

- $650T/760$ showed higher strength and better ductility than $760T/760$. 
Different Hardness Distribution in HAZ after PWHT

- Distinct soft zone and wide hardness range in 760T/760 specimen
Coarsening of Carbides Trigger Softening

- Coarser $M_{23}C_6$/MX were observed in 760T/760.
Lower tempering temperature shifted fracture locations to base metal

- Fracture behavior transition between 700/650°C
Creep-rupture lives also showed transition

- At 650°C/70MPa: Rupture life for “650T/760” ≈ 5X life for “760T/760”
- At 600°C/120MPa: need further considerations
**In-situ** diffraction study of HAZ simulated samples (OSU)

Motivation: To understand the mechanism of tempering temperature dependence of softening after PWHT.

Output: Dissolution, Nucleation, and Growth of $M_{23}C_6$ during heating and cooling process explain the variety of microstructure (and properties).
Synchrotron diffraction experiments can capture the transformation dynamics

- Tested two different tempered samples (at 650 and 760°C), at SP8, Japan (by X. Yu and S. Babu, OSU)
- Much higher time resolution than conventional XRD
**M\textsubscript{23}C\textsubscript{6} dissolved above A\textsubscript{C3} temperature, but MX remained after cooling**

(Tempered at \textbf{760°C}, Peak temperature = \textbf{1050°C})

- Contrast of \textit{M}\textsubscript{23}C\textsubscript{6} after peak temperature is very weak.
Both $M_{23}C_6$ and MX remained after cooling

(Tempered at 760°C, Peak temperature = 950°C)

- Lower peak temperature formed residual $M_{23}C_6$. 

No obvious M$_{23}$C$_6$ observed before and after testing

(Tempered at 650°C, Peak temperature = 950°C)

- The amount of M$_{23}$C$_6$ is lower than the detection limit.
Residual $\text{M}_{23}\text{C}_6$ due to insufficient heating

(Temperd at $760^\circ\text{C}$, Peak temperature = $950^\circ\text{C}$)

The $\text{M}_{23}\text{C}_6$ formation mode during PWHT at $760^\circ\text{C}$:
- in $T760^\circ\text{C}$: coarsening of residual $\text{M}_{23}\text{C}_6$
- in $T650^\circ\text{C}$: nucleation and growth (fine precipitate)
Low temperature pre-weld tempering can minimize the formation of coarse M$_{23}$C$_6$

Table: Microstructure evolution at fine grain heat affected zone

<table>
<thead>
<tr>
<th></th>
<th>Pre-weld temper</th>
<th>Weld (at FGHAZ)</th>
<th>PWHT</th>
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<tr>
<td><strong>High temperature</strong></td>
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<tr>
<td>(e.g. 760T/760)</td>
<td><img src="image1.png" alt="Image" /></td>
<td><img src="image2.png" alt="Image" /></td>
<td><img src="image3.png" alt="Image" /></td>
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<tr>
<td><strong>Low temperature</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>pre-weld tempering</strong></td>
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<td></td>
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<td><img src="image5.png" alt="Image" /></td>
<td><img src="image6.png" alt="Image" /></td>
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- : M$_{23}$C$_6$  : MX

(during welding) ➔ (after cooling)
Creep test of Experimental 9Cr steel (ORNL/NIMS)

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Improved HAZ behavior in modified 9Cr steel

- The B addition resulted in sluggish austenitization (from diffraction study at APS, ORNL).
- No fine grain formation was due to stabilization of $M_{23}C_6$ (NIMS).
Specimens simulated HAZ (P92/N130B)

- Specimens with $T_{\text{peak}} = 900$ C (simulated inter-critical HAZ)

Base metal

HAZ

~3 inch
**Improved creep properties in N130B**

Creep curves of HAZ simulated specimens

![Graph showing creep curves](image)

- **P92:** ~10500h
- **N130B:** >14,000h (still running)

Creep-rupture lives of weldments

![Graph showing creep-rupture lives](image)

- Microstructure characterization is required for better understanding.
Summary

1. **Modified heat treatments (Gr 91):**
   - Lower pre-weld tempering temperature can improve mechanical properties
     *(Better tensile strength/ductility, 5x longer rupture life at 650°C/70MPa)*
   - Control of $M_{23}C_6$ dissolution/precipitation is the key to improve the mechanical properties of weld 9Cr steels

2. **Modified alloys:**
   - Eliminating FGHAZ has a potential to avoid type IV failure
     *(Improved creep properties of the N and B modified steel)*

**Future plan:**
- Complete characterization of creep-rupture specimens
- Propose new processing route/ alloy compositions based on the current results
  - Higher strength, better oxidation resistance, and type IV failure resistance
FY11 Milestones & Status:

- Complete tensile testing of 'best' plates
  - Status: Met
- Initiate long-term creep-tests of welded joints
  - Status: Met
- Evaluate aged microstructures and issue a technical paper/report on current state of studies
  - Status: Delayed until FY12 (scheduled July 31, 2012).
- Evaluate initial creep-test results, determine progress
  - Status: Met
FY12 Milestones & Status:

• Characterize cross-weld specimens of 9Cr steel weldments subjected to non-standard heat treatments
  – Status: Met
• Evaluate creep-test results of synchrotron diffraction specimens
  – Planned May 31, 2012
• Produce a publication on initial results of microstructure characterization of creep specimens from modified heat treatment study (in collaboration with OSU)
  – Planned July 31, 2012
• Initiate production of experimental heats of new, advanced creep strength enhanced ferritic steels with resistance to type IV cracking
  – Planned September 30, 2012