Fundamental Understanding of Creep-Fatigue Interactions in 9Cr-1MoV Steel Welds

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Fundamental Understanding of Creep-Fatigue Interactions in 9Cr-1MoV Steel Welds

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a. Abstract

Ferritic-martensitic steels constitute a vital class of materials for current and future generation of nuclear reactors as well as ultra supercritical coal fired power plants due to their superior creep resistant properties. One particular ferritic-martensitic steel, 9Cr-1MoV (Grade 91) is one of five materials approved for high temperature use in nuclear service by the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code (BPVC). Grade 91 is also a candidate material for proposed fission and fusion reactors such as the sodium cooled fast reactor (SFR).

In service, Grade 91 components can be subjected to elevated temperatures and cyclic stresses, leading to the accumulation of creep-fatigue (CF) damage. While the monotonic creep properties of Grade 91 steels have been studied extensively, there is less knowledge concerning the mechanisms governing CF damage of this steel in the creep-dominated failure region. Moreover, the creep failure is especially pronounced in welded Grade 91 components, which are commonly joined using conventional arc welding techniques such as flux-cored arc welding (FCAW). In a Grade 91 weld, the creep failure typically occurs in the outer region of the heat affected zone (HAZ), specifically in the intercritical or fine-grained heat-affected zone (ICHAZ or FGHAZ), a phenomenon known as type IV failure. Currently, there exists a limited knowledge of CF damage mechanisms in Grade 91 welds.

Improving the mechanistic understanding of the CF damage and failure of Grade 91 steel and its welds with inhomogeneous microstructure is crucial for the safety and design of key structural components in advanced, high-temperature reactors. The overall objectives of the project were to advance the state of knowledge and understanding of CF damage in both Grade 91 base metal and its welds, especially under loading conditions where creep is the dominant damage mechanism.

The key apparatus developed or utilized in the project included:

- Developed a cost-effective dwell CF testing system based on a standard lever-arm creep-testing frame with a load train featuring a pneumatic actuator. The system was equipped with high-resolution Linear Variable Differential Transformer (LVDT) extensometer as well as a digital acquisition system to record strain, temperature, dwell time and loading cycles.
- Developed a high-temperature digital image correlation (DIC) based measurement system to map the strain localization in the Grade 91 weld in-situ during CF testing.
- Developed the welding procedure for joining Grade 91 steel plates using a low heat input arc welding process based on cold metal transfer (CMT).
- Utilized Gleeble®, a thermal-mechanical testing system, to produce specimens with simulated ICHAZ microstructure, which were subsequently used in CF testing to isolate the effect of HAZ microstructure on CF properties.
- Utilized high-resolution electron microscopies for characterizing subgrain size, dislocation density, and precipitate size distribution of Grade 91 steel and welds.
The overall results obtained in the project included:

- Fabricated welded joints of Grade 91 plates using the conventional FCAW and low heat input CMT processes.
- Large amount of CF data of Grade 91 base metal and cross-weld specimens tested under various load-controlled testing conditions. Testing conditions included:
  - **Specimens:** Base metal, cross-weld of FCAW, cross-weld of CMT, and Gleeble-simulated ICHAZ
  - **Testing temperature:** 600 or 650°C
  - **Maximum load at 650°C:** 85, 100 or 150 MPa
  - **Maximum load at 600 °C:** 175 and 200 MPa
  - **Dwell time at max. load:** 300, 900, 3600 and 5400 seconds
- Extensive microstructure characterization of the evolution of subgrain size, dislocation density, and precipitate size distribution during CF testing of Grade 91 base metal.
- Anelastic backflow of strain at minimum load.
- Evolution of local HAZ strain in cross-weld specimens using high-temperature DIC.
- Characterization of location-specific ICHAZ microstructure for subgrain size, dislocation density, and precipitate size distribution before and after CF testing.
- Computational simulation of carbon diffusion during welding and subsequent CF testing.

By studying the mechanical response to CF loading coupled with advanced characterization, the following key conclusions were drawn:

**Base metal of Grade 91 steel:**

- In the load-controlled tests, adding fatigue cycles had a deleterious effect on the rupture life. Moreover, the CF damage was caused by creep voids formed around coarsened precipitates, a mechanism similar to that in monotonic creep damage.
- Dislocation density decreased logarithmically as a function of creep-fatigue cycling.
- Subgrain coarsening occurred at the onset of creep-fatigue and was followed by a decrease in subgrain diameter after the primary transient, and then rapid coarsening in the tertiary creep-fatigue region.
- Stress exponents remained constant for CF specimens tested at 600 and 650°C, indicating no change in CF damage mechanisms over this temperature range.

**Weldments of Grade 91 steel:**

- Creep-fatigue failure occurred near the outer edge of HAZ in the ICHAZ/FCHAZ, indicative of type IV failure.
- The weldments joined by low heat input CMT process significantly outperformed those joined by the conventional FCAW in CF testing.
- Microstructural results indicated that the $M_{23}C_6$ precipitate stability was increased in the CMT weldments, impeding subgrain coarsening and increasing CF properties.
- Based on the results of diffusion simulations and CF testing data of Gleeble-simulated ICHAZ, the improved CF properties in CMT was likely due to a combination of carbon diffusion and HAZ width.

Uncovering the mechanisms governing the creep-fatigue deformation and damage in Grade 91 base metal in creep-dominated region as well as type IV failure in Grade 91 welds contributes to a new knowledge that may lead to more accurate life prediction models than those currently in use. Moreover, the superior CF properties obtained by low heat input CMT has a high potential to develop practical solutions to the premature failure of Grade 91 weld.
b. Statement of objective and description of the effort performed, and the accomplishments achieved

b.1. Introduction

Grade 91 (or 9Cr-1MoV) steel was developed in the 1970s/1980s by Oak Ridge National Laboratory to increase operating parameters for future generations of power plants.\[8\] Typical in-core applications include cladding wrappers and ducts while out-of-core applications consist of pressure vessels and piping.\[9\] Grade 91’s complex chemistry produces a complex microstructure, which takes advantage of many high-temperature strengthening mechanisms. In the normalized and tempered condition, Grade 91 contains a substructure of dislocation networks, forming subgrain boundaries with substantial dislocation content within subgrain interiors. The high chromium content provides corrosion resistance and formation of $\text{M}_{23}\text{C}_6$ type carbides which nucleate and grow at prior austenite and subgrain boundaries, resulting in a boundary pinning, high temperature strengthening effect. Molybdenum is a ferrite stabilizer and is distributed between the solid solution and $\text{M}_{23}\text{C}_6$ and MX type carbides. Vanadium and Niobium precipitate in the form of MX carbides, nitrides and carbonitrides. MX precipitates nucleate within subgrain interiors, inhibiting dislocation movement during elevated temperature deformation.\[10\]

In service, Grade 91 components can be subjected to elevated temperatures and cyclic stresses, leading to the accumulation of creep-fatigue (CF) damage. The monotonic creep properties of Grade 91 base metal have been studied extensively, and additionally CF data of Grade 91 steel, tested in strain-controlled condition, is becoming available. On the other hand, there is less knowledge concerning the mechanisms governing CF damage in Grade 91 base metal in the creep-dominated failure region.\[3–5\]

Moreover, the creep failure is especially pronounced in welded Grade 91 components, which are commonly welded using conventional arc welding techniques such as flux-cored arc welding (FCAW).\[6,7\] In a Grade 91 weld, the creep failure typically occurs in the outer region of the heat affected zone (HAZ), specifically in the ICHAZ or FGHAZ, a phenomenon known as type IV failure. ICHAZ is a region where the local peak temperature experienced by the steel during welding is between $\text{Ac}_1$ and $\text{Ac}_3$, resulting in an incomplete transformation to austenite and thus fine-grained structure. The temperature in the FGHAZ during welding is above $\text{Ac}_3$ but not high enough to fully dissolve the $\text{M}_{23}\text{C}_6$ carbides existed in the base metal. During welding, these carbides prevent growth of new austenite grains formed, resulting in a fine-grained structure.\[10,11\] Kimmins and Smith suggested that the relative ease of grain boundary sliding in the FGHAZ leads to premature failure in Grade 91 weldments.\[12\] Another mechanism proposed by Hirata and Ogawa suggested the loss in creep strength in the FGHAZ could be attributed to the coarsening of creep-strengthening MX and $\text{M}_{23}\text{C}_6$ carbides during the welding process.\[13\] Although these mechanisms have provided an improved understanding of monotonic creep properties, there exists a limited knowledge of CF damage mechanisms in Grade 91 welds.

The overall objectives of the project were to advance the state of knowledge and understanding of CF damage in both Grade 91 base metal and its welds, especially under loading conditions where creep is the dominant damage mechanism. The specific objectives are:
• Creep-fatigue damage in Grade 91 base metal in creep-dominated region
• Type IV failure in creep-fatigue loading of Grade 91 welds

A description of the effort performed and the accomplishments achieved are provided in the following sections.

b.2. Objective 1 - Creep-fatigue damage in Grade 91 base metal in creep-dominated region

b.2.1. Creep-fatigue testing equipment

The present study utilizes a cost-effective, load-controlled CF testing apparatus, which can be used to extend tests to longer hold times, simulating a creep-dominated creep-fatigue testing environment. The CF frame developed at OSU consists of a standard lever-arm creep-testing frame with a load train featuring a pneumatic actuator. The pneumatic actuator provides the ability to conduct positive R-ratio CF testing while controlling minimum and maximum loads, dwell time, and loading rate. The actuator is controlled with an electro-pneumatic regulator and LabVIEW programming software. LabVIEW records the load on the specimen, cyclic loading information, and specimen extension via linear variable differential transformer (LVDT) extensometry. Using this design, loading rates between 20 and 100 MPa/s are achievable. Current specimen geometry limits the minimum load to 10 MPa or higher, with no practical limit on the maximum tensile load. Specimens were tested in atmosphere at temperatures between 600°C and 650°C, however, temperatures between 25°C and 1100°C are achievable with the current experimental setup. Figure 1 below shows a schematic for the CF frame used in this study along with a schematic stress-strain hysteresis produced by this frame.

**Figure 1:** Schematic representation of cost effective CF testing apparatus. Inset shown is a schematic stress-strain hysteresis generated by this load-frame[15]
b.2.2. High-resolution microstructure characterization

Samples for Scanning Electron Microscopy (SEM) and Electron Backscattered Diffraction (EBSD) were polished initially using SiC polishing pads from 600 to 1200 grit followed by a diamond compound polish, and approximately four hours in a colloidal silica vibratory (0.02μm). SEM imaging was performed using 5kV and 1.6nA beam settings on a ThermoFisher Apreo FEG SEM. Due to the ferromagnetic properties of ferritic-martensitic steels, samples for Transmission Electron Microscopy (TEM) were prepared using a FEI Helios Dual Beam FIB/SEM (Focused Ion Beam). TEM imaging was performed using a FEI Tecnai F20 S/TEM microscope at 200keV in Scanning (S)TEM mode.

b.2.3. Effect on creep life of Grade 91 due to fatigue cycling

Figure 2 shows the max strain vs time curves for four Grade 91 base metal specimens creep-fatigue tested at various temperatures and max loads; all conducted with a 15 minute (900 s) max load dwell time. Table 1 compares the CF rupture time with the time to rupture for an equivalent monotonic creep specimen (i.e., tested at the same temperature and max load) taken from Tabuchi et al.\textsuperscript{[14]} From Table 1 it is clear that the time to rupture for a Grade 91 base metal due to combined CF loading is significantly shorter than the monotonic equivalent. Specifically, the $t_r$ ratios indicate that the CF specimens fail 80% to 90% sooner than the monotonic specimens, indicating a marked deleterious effect on creep life of Grade 91 due to fatigue cycling.

Figure 2: CF testing conducted on base metal specimens at various loads and temperatures
**Table 1:** Time to rupture ratios for CF tests compared to monotonic creep specimens at equivalent max loads and temperatures

<table>
<thead>
<tr>
<th>Max. Load (MPa)/Temp (°C)</th>
<th>Cycles to failure</th>
<th>Time to failure (hr)</th>
<th>Est. Creep Rupture Time (hr)</th>
<th>t_r-CF/t_r-C</th>
</tr>
</thead>
<tbody>
<tr>
<td>85/650</td>
<td>1221</td>
<td>311.38</td>
<td>2960</td>
<td>11%</td>
</tr>
<tr>
<td>100/650</td>
<td>607</td>
<td>155.15</td>
<td>693</td>
<td>22%</td>
</tr>
<tr>
<td>175/600</td>
<td>127</td>
<td>32.95</td>
<td>270</td>
<td>12%</td>
</tr>
<tr>
<td>200/600</td>
<td>28</td>
<td>7.33</td>
<td>40</td>
<td>18%</td>
</tr>
</tbody>
</table>

b.2.4. Microstructural evolution during creep-fatigue deformation

To improve the understanding of damage processes in Grade 91 steel subjected to creep-dominated, “longer-term” creep-fatigue loading, a series of interrupted tests were conducted to observe the microstructural evolution. **Figure 3** below shows the creep-fatigue curve with annotations showing the cycle at each interruption. Interrupted testing was conducted at 650°C with a maximum load of 85 MPa (R=0.12) and a dwell time of 900s. Test interruptions occurred at cycle 0, 26, 451, and 1000. Microstructural investigations were also conducted for a failed specimen, which ruptured at cycle 1654. Two of the primary defect structures for Grade 91 are subgrain size and dislocation density. Kostka et al, Cerri et al, and Ennis et al have shown that these microstructural features are critical for describing monotonic creep response of ferritic-martensitic steels.[7,8,14]

![Figure 3: Creep-fatigue curve from interrupted tests showing cycles chosen for microstructural investigations](image-url)
In order to measure dislocation density as a function of CF strain accumulation, a series of zone-axis STEM images were taken from each specimen. Zone-axis imaging allows for the activation of many diffraction conditions at once, giving the best possible representation of defect content within the specimens. Figure 4 shows zone-axis images from specimens with varying amounts of creep-fatigue strain. These images clearly show that the dislocation density decreases significantly as a function of CF strain accumulation. To quantitatively measure dislocation density, a commonly accepted stereological approach was used, employing equation 1.\textsuperscript{16,17} Using this equation, a grid of intersecting lines was overlaid on zone-axis images and the intersections of lines and dislocations were counted and normalized by specimen thickness.

\[
\rho_{\text{dis}} = \frac{1}{t} \left( \frac{N_L}{L_L} + \frac{N_T}{L_T} \right)
\]

Equation 1

\(\rho_{\text{dis}}\) = dislocation density (m\(^{-2}\))

\(t\) = specimen thickness (m)

\(N_L, N_T\) = Longitudinal and transverse dislocation intersections, respectively

\(L_L, L_T\) = Longitudinal and transverse line lengths (m), respectively

\textbf{Figure 4}: Zone axis STEM images, showing the dislocation content as a function of cycle number under creep-fatigue loading conditions
Figure 5 shows the dislocation density as a function of cycle number for the specimens examined in Figure 4. It shows that the dislocation density decreases logarithmically as a function of CF strain accumulation. Over the span of more than 1600 cycles the dislocation density decreases by approximately three orders of magnitude. The apparent immediate decrease in dislocation density exhibited by the specimen tested after 26 cycles of CF damage is especially interesting. The CF strain curve shown in Figure 3 exhibits a significant primary transient, indicating a hardening effect. However, dislocation density measurements show that dislocation density is continuously decreasing, indicating that the hardening shown during primary creep-fatigue is not due to an increase in dislocation content.

![Dislocation Density vs Cycle Number](image)

**Figure 5:** Dislocation density as a function of creep-fatigue cycle for a series of interrupted creep tests.

Figure 6 shows a series of STEM images illustrating subgrain size as a function of CF cycling. Using the line intercept grain size measurement method outlined in ASTM E112, subgrain size was quantified and the result is summarized in Figure 7. Subgrain size is shown to increase at the beginning of testing, followed by a decrease in size after the primary transient, and a significant increase in coarsening at the onset of the tertiary transient. Especially interesting to note from Figure 7 is the decrease in subgrain size after the primary transient, which is most likely due to the rearrangement of dislocations into low energy configurations, effectively increasing low angle grain boundary area and decreasing subgrain size. The hardening behavior observed in Figure 3 during the primary transient may be attributable to the increase in low angle boundaries observed in Figure 6.
Figure 6: STEM Images showing subgrain coarsening with creep-fatigue strain accumulation

Figure 7: Subgrain size as a function of creep-fatigue cycling
b.2.5. Effect of dwell time on creep-fatigue deformation

The effect of dwell time on creep-fatigue deformation has been systematically studied. By observing the minimum strain accumulation per cycle it is evident that as the dwell time is increased the strain accumulation per cycle is increased. Preliminary results suggest that microstructural damage is most severe for intermediate dwell times. This is illustrated by the STEM images shown in Figure 9, where the subgrains exhibit the greatest coarsening for the 900s followed by the 5400s and 300s dwell times. Future work is needed to understand the effect of dwell time on microstructural damage.

Figure 8: Minimum strain accumulation per cycle, as a function of dwell time

Figure 9: STEM images illustrating the effect of dwell time on subgrain coarsening
b.2.6. Dependence of creep-fatigue strain rate on stress

The CF tests on Grade 91 base metal specimens at various temperatures and maximum load times allowed for stress dependence correlations analogous to those used to classically describe the monotonic creep behavior. Figure 10 shows $\frac{d\varepsilon_{\text{min}}}{dt}$ vs. maximum dwell load for a series of tests conducted at 650 °C and 600 °C. It can be found that the minimum strain rate vs. load curves follow a power law relationship, similar to the monotonic creep testing.

Using the classic Norton power law relationship shown in equation 2 it is possible to extract creep-fatigue power law relationships. Fitting equation 2 to the creep-fatigue data collected at 600°C and 650°C yields stress exponents of $n=7.06$ and 7.07, respectively. The stability of the stress exponent indicates that the same mechanisms are governing CF damage accumulation over this temperature range (i.e., 600-650°C).

\[
\dot{\varepsilon}_{ss} = \frac{A}{kT} \left( \frac{\sigma}{G} \right)^n
\]

Equation 2

$\dot{\varepsilon}_{ss}$ = Steady state strain rate (s$^{-1}$)  
$A, n$ = Constants  
$K$ = Boltzmann’s constant (J/K)  
$T$ = Temperature (K)  
$\sigma$ = Stress (MPa)  
$G$ = Bulk modulus (GPa)

Tests conducted with 125 MPa max load at 600°C and 650°C (also shown in Figure 10) allow for a calculation of activation energy for CF, using equation 3 below. Activation energy for CF from equation 3 yields $Q_c = 494$ kJ/mol. In the literature, Cerri et al. calculated an activation energy for monotonic creep to be 661 kJ/mol, for a similar temperature range. [7] Therefore, it can be
concluded that CF is a more deleterious type of damage than monotonic creep over this temperature range. Additional literature data is being collected to compare the activation energy determined in this study and will be reported in future journal publications.

\[
Q_c = -R \frac{\partial (ln \dot{\varepsilon})}{\partial \left(\frac{1}{T}\right)}
\]

Equation 3

\(Q_c = \text{Creep-fatigue activation energy (kJ/mol)}\)
\(R = \text{Universal gas constant (J/k mol)}\)
\(\dot{\varepsilon} = \text{Minimum strain rate (s}^{-1}\text{)}\)
\(T = \text{Temperature (K)}\)

b.2.7. Anelastic backflow of strain at minimum load

An interesting behavior observed in this study, which is unique to the load-controlled test, is the anelastic backflow of strain at minimum load. Figure 11 below shows a stress-strain hysteresis from the Grade 91 base metal for three of the cycles; one in each primary, secondary and tertiary stage in Figure 3. As shown in Figure 11, while the creep strain changes dramatically as a function of cycle number, the anelastic strain at minimum load changes very little. However, as the time spent at minimum load increases, the amount of “recovered” strain increases as well, as supported by additional tests summarized in Table 2.

\[\varepsilon_{anelastic}\]

\[\varepsilon_c\]

\[\varepsilon_{min}\]

**Figure 11**: Hysteresis from different CF cycles in a Grade 91 base metal sample, showing anelastic backflow of strain, during primary, secondary and tertiary stages.
Table 2: Effect of time spent at minimum load on anelastic backflow of strain

<table>
<thead>
<tr>
<th>Time at Minimum Load</th>
<th>Strain Recovery</th>
</tr>
</thead>
<tbody>
<tr>
<td>5s (100 MPa max)</td>
<td>0.010%</td>
</tr>
<tr>
<td>10s (85 MPa max)</td>
<td>0.017%</td>
</tr>
<tr>
<td>30s (85 MPa max)</td>
<td>0.035%</td>
</tr>
<tr>
<td>60s (85 MPa max)</td>
<td>0.040%</td>
</tr>
</tbody>
</table>

The anelastic backflow of strain exhibited by the Grade 91 specimens is present in all CF tests conducted on base metal and welded specimens. This anelastic flow of strain may be due to back stresses that are greater than the minimum load applied. Back stresses may be caused by load shedding from the highly inhomogeneous microstructure present in Grade 91 microstructure after tempering. Figure 12 shows a zone-axis, diffraction contrast STEM image, showing subgrains with very similar orientations but highly inhomogeneous dislocation content.

Figure 12: Left: zone-axis STEM image illustrating varying dislocation densities within subgrains of similar orientation. Middle: TKD IPF map illustrating similar orientations between adjacent subgrains. Right: Grain Reference Orientation Deviation (GROD) map illustrating large deviation in lattice misorientation between subgrains with varying dislocation densities.

b.3. Objective 2 – Type IV failure in creep-fatigue loading of Grade 91 welds

b.3.1. Materials and welding

As-received Grade 91 plates, provided in a normalized and tempered condition, has a 100% tempered martensite microstructure. Butt joints were welded using a conventional flux-cored arc welding (FCAW) and a low heat input cold metal transfer (CMT) welding process. The filler metal compositions are summarized in Table 3. As illustrated in Table 4, the heat input is significantly lower in the CMT weldment than in the FCAW weldment. This is manifested macroscopically in a 40% narrower HAZ in the CMT weldment when compared to the FCAW
weldment, as shown by the optical micrographs in Figure 13.

Following welding, specimens were subjected to post-weld heat treatment (PWHT) at 760°C (1400°F) for two hours to temper any fresh martensite produced during welding and to relieve some of the microstructural gradient present in the HAZ. Both welding and PWTH were performed in compliance with the relevant procedure in ASTM BPVC.

### Table 3: Grade 91 base metal and filler metal compositions (in wt%)

<table>
<thead>
<tr>
<th></th>
<th>C</th>
<th>Cr</th>
<th>Mo</th>
<th>V</th>
<th>Ni</th>
<th>Mn</th>
<th>N</th>
<th>Nb</th>
<th>B</th>
</tr>
</thead>
<tbody>
<tr>
<td>Plate</td>
<td>0.08</td>
<td>8.40</td>
<td>0.92</td>
<td>0.23</td>
<td>0.09</td>
<td>0.45</td>
<td>0.041</td>
<td>0.075</td>
<td>10⁻⁴</td>
</tr>
<tr>
<td>Solid Filler</td>
<td>0.106</td>
<td>8.92</td>
<td>0.99</td>
<td>0.194</td>
<td>0.47</td>
<td>0.75</td>
<td>0.044</td>
<td>0.063</td>
<td>3x10⁻⁴</td>
</tr>
<tr>
<td>FC Filler</td>
<td>0.096</td>
<td>8.73</td>
<td>1.026</td>
<td>0.219</td>
<td>0.648</td>
<td>0.610</td>
<td>0.022</td>
<td>0.051</td>
<td>1.4x10⁻⁴</td>
</tr>
</tbody>
</table>

### Table 4: Welding parameters for FCAW and CMT

<table>
<thead>
<tr>
<th>Process</th>
<th>Wire Feed Speed (m/s)</th>
<th>Travel Speed (mm/s)</th>
<th>Travel Angle</th>
<th>Voltage (V)</th>
<th>Current (A)</th>
<th>Heat input (kJ/mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CMT</td>
<td>0.0846</td>
<td>3.37</td>
<td>10° push</td>
<td>13.4</td>
<td>168</td>
<td>0.67</td>
</tr>
<tr>
<td>FCAW</td>
<td>0.269</td>
<td>4.23</td>
<td>10° drag</td>
<td>25.0</td>
<td>238</td>
<td>1.40</td>
</tr>
</tbody>
</table>

**Figure 13:** Butt joint schematic and optical micrographs showing HAZ width in each welding process FCAW (right) and CMT (left)

b.3.2. Effect of low heat input welding on creep-fatigue performance

Albert et al. have shown that by reducing the heat input used during welding of ferritic-
martensitic steels, the monotonic creep rupture life can be increased.\cite{15} The current study attempts to evaluate the creep-fatigue performance of a field applicable low-heat input arc welding process, CMT welding.

Figure 14 shows a comparison of creep-fatigue performance for FCAW and CMT transverse weld specimens at 85 and 100 MPa max load and 900s dwell time at 650°C test temperature. As shown in this figure, CMT specimens exhibit approximately one order of magnitude decrease in minimum strain rate compared to FCAW creep-fatigue specimens tested with the same CF loading parameters. In addition to drastically decreased minimum strain rates, the rupture times for the CMT specimens are approximately 5 times greater at 100 MPa max load and 10 times greater at 85 MPa max loads.

While the CMT specimens exhibit significantly enhanced creep-fatigue performance, failure in all FCAW and CMT specimens occurred in the outer edge of HAZ, indicative of Type IV failure. Figure 15 shows an example of fractured CMT specimen. Given the small width of ICHAZ and FCHAZ, it is difficult to determine the exact location of fracture. Hence, the failure location is designated as ICHAZ/FCHAZ.

![Figure 14](image1.png)

**Figure 14:** Creep-fatigue performance comparison for FCAW and CMT welded specimens tested at max. load of 85 and 100 MPa. Dwell time = 900s, and test temperature = 650°C.

![Figure 15](image2.png)

**Figure 15:** Optical micrograph of a ruptured CMT specimen, illustrating failure in the outer edge of HAZ (i.e., ICHAZ/FCHAZ).
b.3.3. Local strain accumulation in HAZ

As a cross-weld specimen comprises microstructure gradient and thus property gradient along its length direction, the local deformation (as opposite to the total deformation over the gauge section) is essential to study the CF damage evolution. A high-temperature DIC technique was developed to observe the local strain distribution in-situ during CF testing. The max load, used high-temperature DIC experiment was 150 MPa. Due to equipment limitation, the total testing duration was limited to 10 hours. Testing at the lower max. loads (e.g., 100 and 85 MPa) did not result in fracture of cross-weld specimens.

High-temperature DIC results for a cross-weld specimen of FCAW are shown in Figure 16, where the boundaries of weld metal (WM), heat-affected zone (HAZ), and base metal (BM) are marked. As shown in this figure, strain rapidly accumulated in the HAZ even after the first two cycles. The strain continued to accumulate in the HAZ, and after the last cycle of the test (cycle #29), the highest strain was found in the HAZ adjacent to the BM. This HAZ region is expected to be ICHAZ/FCHAZ. It is noted that the test was interrupted after cycle #29 due to equipment limitation, and the cross-weld specimen was not fractured.

Figure 16: Distribution of longitudinal strain in a cross-weld specimen of FCAW, which shows a significant strain accumulation in the ICHAZ. Strain map was measured using digital image correlation. Testing temperature = 650 °C, peak stress = 100 MPa and dwell time = 900 s

A comparison of the local strain in HAZ as a function of time for FCAW versus CMT cross-weld specimens is shown in Figure 17. Due to the extremely large volume of data generated by the DIC camera, two cycles were measured and analyzed for FCAW specimen; four cycles for CMT specimen. The actually measured data are plotted as solid lines whereas the data between the recording cycles are extrapolated as dashed lines. Similar to that shown in Figure 14, the CMT specimen fractured at a much longer time (7.5 hours) than the FCAW specimen (2 hours). The steady-state strain rate is also much lower for CMT than FCAW. The local HAZ strain at fracture is about 50% for CMT. For FCAW, it is noted that the final fracture strain was not determined as the DIC recording was not done for the cycle during which the specimen failed.
To better quantify the strain accumulation and failure location, the strain field measured at the cycle closest to the final failure for FCAW vs. CMT was compared in Figure 18. This strain field was superimposed on a “un-deformed” reference image prior to CF testing. As shown in this figure, the location with highest local strain and failure measured on the un-deformed image was at a distance of 1.95 mm and 1.04 mm away from the fusion boundary for the FCAW and CMT specimens, respectively. These distances are fairly consistent with the HAZ widths shown in Figure 13. Combining with the post-test optical microscopy, the failure was determined to occur in the ICHAZ/FGHAZ for both CMT and FCAW cross-weld specimens.

Figure 18: Distribution of longitudinal strain prior to failure in cross-weld specimens of (a) FCAW and (b) CMT measured by DIC. Testing temperature = 650 °C, peak stress = 150 MPa and dwell time = 900 s.

b.3.4. Isolated effect of HAZ microstructure on CF properties

The much improved CF properties for CMT weld over the conventional FCAW is interesting in
that both welds were post-weld heat treated and a similar microstructure in ICHAZ is thus expected. One possible factor is the different extent of alteration to the base metal microstructure in the ICHAZ/FGHAZ due to different welding heat input. In order to isolate the effect of HAZ microstructure, simulated ICHAZ samples were created using a Gleeble thermo-mechanical simulator. The experimentally-measured thermal profile used to make these specimens is shown in Figure 19. Compared to a cross-weld specimen with a microstructure gradient, the simulated ICHAZ specimen is expected to contain a homogenous microstructure within its gauge section.

![Figure 19: Thermal profiles measured in the ICHAZ for FCAW and CMT weldments during welding.](image)

The Gleeble-simulated ICHAZ specimens were tested under the same conditions as the specimens reported in Figure 14. The results for the simulated Gleeble specimens are found in Figure 20. When comparing the results of Figure 20 to Figure 14, it is clear that the simulated Gleeble specimens do not exhibit the large improvement in CF properties of CMT over FCAW. If the hypothesis concerning carbon diffusion from the FZ to the ICHAZ is correct, these results are to be expected. In other words, the ICHAZ microstructure is likely a major factor for the improved CF properties in CMT.
Figure 20: CF results of Gleeble-simulated ICHAZ specimens at 650°C, 100MPa and 85 MPa max loads

b.3.5. HAZ microstructure

As the failure occurred in the ICHAZ/FCHAZ, it is essential to study the microstructure evolution in this region due to CF loading. A prerequisite for such characterization is to produce location-specific specimens, which was done by focused ion beam milling. For an untested sample, a combination of microhardness indentation and chemical etching were used to distinguish the specific regions in the HAZ and to locate the ICHAZ on the weld transverse section. An example for FCAW before CF loading is shown Figure 21 below. Using the idents as reference markers, FIB specimens were then extracted from the ICHAZ.

The cross-weld CF sample had a diameter of 0.16" (4.06 mm) and a gauge length of 0.64" (16.26 mm). The gauge section was carefully machine to cover the entire HAZ and a portion of the adjacent weld metal and base metal. After CF testing to rupture, the failed region was cut longitudinally (i.e., along the weld transverse direction). The aforementioned combination of microhardness indentation and chemical etching were also applied to the rupture sample to confirm the failure location in ICHAZ. FIB specimens were then extracted from region near the fracture surface.

STEM investigations of ICHAZ specimens show that a major difference microstructurally between the FCAW and CMT is the $M_{23}C_6$ precipitate size and distribution as well as subgrain size. Figure 22 shows STEM images from FCAW and CMT specimens before and after CF deformation, showing $M_{23}C_6$ and subgrain coarsening after CF testing.

Table 5 shows the average size and area fraction of MX precipitates before and after CF deformation for FCAW and CMT specimens, in addition to subgrain size and dislocation density measurements. As shown in this table, the dislocation density remains fairly constant in the
FCAW and CMT specimens before and after CF deformation. However, the dislocation density in the CMT specimens is considerably larger than the dislocation density in the FCAW specimens after PWHT and CF rupture. Also important to note is the significant subgrain coarsening in the FCAW specimens compared to relatively stable substructure present in the CMT ICHAZ.

![Image of micrographs](Image)

**Figure 21:** Optical micrograph (left) and micro-hardness profile (right) used to distinguish the specific regions in the HAZ and to locate ICHAZ. Example is for FCAW.

![Image of STEM images](Image)

**Figure 22:** STEM images, showing M$_{23}$C$_6$ precipitate coarsening in FCAW weldments before and after CF testing

**Table 5:** Microstructural characteristics of FCAW and CMT specimens before and after CF testing

<table>
<thead>
<tr>
<th>Sample</th>
<th>MX Diameter (nm)</th>
<th>MX Area Fraction (TEM)</th>
<th>Dislocation Density (m$^{-2}$)</th>
<th>Subgrain Size (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CMT_PWHT</td>
<td>45.7±15.6</td>
<td>0.87%</td>
<td>1.19x10$^{14}$ ± 1.04x10$^{14}$</td>
<td>581±121</td>
</tr>
<tr>
<td>CMT_CF</td>
<td>33.3±15.9</td>
<td>0.78%</td>
<td>1.99x10$^{14}$ ± 1.61x10$^{14}$</td>
<td>517±76</td>
</tr>
<tr>
<td>FCAW-PWHT</td>
<td>40.1±16.3</td>
<td>0.44%</td>
<td>3.988x10$^{13}$ ± 1.83x10$^{13}$</td>
<td>691±76</td>
</tr>
<tr>
<td>FCAW-CF</td>
<td>50.8±18</td>
<td>0.46%</td>
<td>5.20x10$^{13}$ ± 3.11x10$^{13}$</td>
<td>1088±480</td>
</tr>
</tbody>
</table>
In addition to STEM diffraction contrast imaging to analyze defect content and MX precipitate characteristics, low kV SEM imaging was utilized to study M$_{23}$C$_6$ precipitate characteristics before and after PWHT and CF deformation. The BSE images were segmented to extract M$_{23}$C$_6$ precipitate volume fractions and other relevant precipitate characteristics. An example of BSE images from PWHT and CF specimens is shown below in Figure 23.

![BSE images showing M$_{23}$C$_6$ particles at boundaries](image)

**Figure 23**: BSE images showing M$_{23}$C$_6$ particles at boundaries (a) FCAW-PWHT, (b) FCAW-CF, (c) CMT-PWHT, and (d) CMT-CF

Table 6 below shows M$_{23}$C$_6$ precipitate statistics determined via segmentation of the low kV BSE images. From this table, it is clear that the stability of the M$_{23}$C$_6$ precipitates is significantly better in the CMT specimens than in the FCAW specimens. The CMT specimens exhibit higher area fraction of M$_{23}$C$_6$ and less coarsening than the FCAW weldments. Correspondingly, the nearest neighbor distance is much lower in the CMT weldments after CF deformation than in the FCAW weldments.

**Table 6**: M$_{23}$C$_6$ precipitate characteristics before and after CF deformation in CMT and FCAW weldments

<table>
<thead>
<tr>
<th>Sample</th>
<th>Area Fraction M$_{23}$C$_6$ (%)</th>
<th>Equivalent Diameter (nm)</th>
<th>Coarsening Rate (nm/hr)</th>
<th>Nearest Neighbor Distance (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>BM-PWHT</td>
<td>1.96±0.10</td>
<td>102.3±4</td>
<td>N/A</td>
<td>343.6±22.4</td>
</tr>
<tr>
<td>FCAW PWHT</td>
<td>1.83±0.16</td>
<td>114±4</td>
<td>1.2</td>
<td>359±34</td>
</tr>
<tr>
<td>FCAW CF</td>
<td>1.71±0.27</td>
<td>149±6</td>
<td></td>
<td>541±27</td>
</tr>
<tr>
<td>CMT PWHT</td>
<td>2.1±0.29</td>
<td>111±10</td>
<td>0.09</td>
<td>359±25</td>
</tr>
<tr>
<td>CMT CF</td>
<td>2.19±0.38</td>
<td>126±9</td>
<td></td>
<td>427±58</td>
</tr>
</tbody>
</table>
The precipitate size distribution after PWHT is fairly constant for the FCAW and CMT weldments. However, after CF deformation, the distribution for the FCAW weldments show a pronounced shift to larger sizes when compared to the CMT weldments after CF deformation. This is best illustrated by the equivalent diameter cumulative distribution curves shown in Figure 24.

![Cumulative distribution curves](image)

**Figure 24:** Cumulative distribution curves for the equivalent diameter measurements of $M_{23}C_6$ precipitates in CMT and FCAW weldments after PWHT and CF deformation.

The similar microstructure characteristics shown in Table 5 and Table 6 are consistent with the comparable CF properties of Gleeble-simulated ICHAZ specimens shown in Figure 20. Hence, it is very intriguing that the cross-weld of CMT specimen markedly outperformed that of FCAW specimen and had much lower coarsening of $M_{23}C_6$ precipitates and subgrains. One possible explanation for the increased volume fraction of $M_{23}C_6$ precipitates and refined distribution of precipitates in the CMT specimens after CF deformation is carbon diffusion from the FZ to the ICHAZ after CF deformation. The filler metal compositions in Table 3 indicate that the carbon content is much higher in the CMT filler wire than in the FCAW filler wire. In addition, due to the low-heat-input achieved by the CMT welding process, the diffusion distance for carbon to travel to nucleate new $M_{23}C_6$ precipitates in ICHAZ is much lower. Figure 25 shows diffusion simulations using Dictra tracking carbon diffusion from the FZ to the ICHAZ during welding, PWHT and aging at the CF test temperature of 650 °C. These simulations were performed assuming single phase BCC diffusion couples with the BM composition on the left and the WM composition on the right. Only a single pass weld is modeled, and the effect of dilution is ignored.
Figure 25 shows that the amount of carbon is much higher after PWHT in the CMT weldments. This is due to the combination of higher carbon content and a much shorter diffusion distance in the CMT weldments. It has been well documented that $\text{M}_2\text{3C}_6$ nucleates during PWHT in the HAZ of P91 weldments. With a higher carbon concentration which is continuously increasing throughout PWHT, it is thus expected that the CMT weldments exhibit a higher area fraction of $\text{M}_2\text{3C}_6$ precipitates, which in turn reduces subgrain coarsening and improved CF properties.

**Figure 25**: Dictra simulations of carbon diffusion during welding using the FCAW process (top) and CMT process (bottom)
b.4. Conclusions

By studying the mechanical response to CF loading coupled with advanced characterization, the following key conclusions were drawn:

Base metal of Grade 91 steel:

- In the load-controlled tests, adding fatigue cycles had a deleterious effect on the rupture life. Moreover, the CF damage was caused by creep voids formed around coarsened precipitates, a mechanism similar to that in monotonic creep damage.
- Dislocation density decreased logarithmically as a function of creep-fatigue cycling.
- Subgrain coarsening occurred at the onset of creep-fatigue and was followed by a decrease in subgrain diameter after the primary transient, and then rapid coarsening in the tertiary creep-fatigue region.
- Stress exponents remained constant for CF specimens tested at 600 and 650°C, indicating no change in CF damage mechanisms over this temperature range.

Weldments of Grade 91 steel:

- Creep-fatigue failure occurred near the outer edge of HAZ in the ICHAZ/FCHAZ, indicative of type IV failure.
- The weldments joined by low heat input CMT process significantly outperformed those joined by the conventional FCAW in CF testing.
- Microstructural results indicated that the $M_{23}C_6$ precipitate stability was increased in the CMT weldments, impeding subgrain coarsening and increasing CF properties.
- Based on the results of diffusion simulations and CF testing data of Gleeble-simulated ICHAZ, the improved CF properties in CMT was likely due to a combination of carbon diffusion and HAZ width.

Uncovering the mechanisms governing the creep-fatigue deformation and damage in Grade 91 base metal in creep-dominated region as well as type IV failure in Grade 91 welds contributes to a new knowledge that may lead to more accurate life prediction models than those currently in use. Moreover, the superior CF properties obtained by low heat input CMT has a high potential to develop practical solutions to the premature failure of Grade 91 weld.

c. List of publications

Thesis:

Journal papers:
- T. Mukherjee, J. S. Zuback, W. Zhang and T. DebRoy, “Residual stresses and distortion in

Conference presentations:


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References

10. Klueh, R. L. Elevated temperature ferritic and martensitic steels and their application to future


