Effect of Post-Weld Heat Treatment on Creep Rupture Properties of Grade 91 Steel Heavy Section Welds

Reactor Concepts RD&D

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Abstract

This report summarizes the findings from a systematic study of the effect of post-weld heat treatment on the creep rupture properties of heavy section Grade 91 welds. The overall research approach has been quantitative measurements of thermal, mechanical, and microstructural parameters, and verifications by analytical and numerical models.

Type IV rupture in the inter-critical heat-affected zone has been reproduced from the experiment welds made from flux-cored arc welding of Grade 91 piping and heavy section plates. In the range of 600 to 840 °C, 2 and 8 hour post-weld heat treatment, longer creep life is resulted from Grade 91 welds heat treated with a lower temperature and shorter time (600 °C, 2 hours). However, the room temperature toughness of the heat-affected zone only recovers after a 720 °C, 2 hour post-weld heat treatment. Following a post-weld heat treatment at temperatures above the 820 °C, the creep strain rate and creep ductility significantly increase, the rupture life is shorter, and the rupture location switches to the weld fusion zone, resulting in Type I failure. Post-weld heat-treatment changes the resultant grain size and degree of tempering of martensite, or leads to formation of new martensite if the \( A_1 \) is exceeded. The number and sizes of carbide precipitates are proportional to the post-weld heat treating temperature and time. The precipitation and growth of carbide particles and fine-grained ferrite are believed to be a microstructure with decreased creep resistance.
# Contents

1 Introduction 3

2 Experimental Procedure 5
   2.1 Base and filler materials 6
   2.2 Welding procedure 6
   2.3 Heat treatment 7
   2.4 Microstructural characterization 8
   2.5 Ductility and toughness 8
   2.6 Creep test 9

3 Results 9
   3.1 Ductility characterization 9
   3.2 Microhardness 10
   3.3 Toughness of the heat-affected zone 10
   3.4 Thermal measurements 12
   3.5 Creep results 13

4 Discussion 14
   4.1 Thermal models and verification 14
   4.2 Microstructure evolution during welding and PWHT 15
   4.3 Creep damaged microstructure 17
   4.4 Stress relaxation 18
   4.5 A model for cross-weld creep 19

5 Conclusion 21

6 Project Deliverables 22
   6.1 Reports and publications 22
   6.2 Students and educational activities 23

7 Acknowledgment 23

8 References 24

9 Tables and Figures 26
1. Introduction

Grade 91 and creep rupture properties – The fabrication and use of Grade 91 steel in high temperature steam pressure vessels has become a subject of particular interest to the nuclear and fossil fuel power generation industries. This is because Grade 91 exhibits a superior level of creep strength and oxidation resistance at elevated temperatures [1]. However, after being welded, Grade 91 exhibits a severe loss in creep performance and must be heat treated in order to improve creep strength and meet design requirements. In addition to this, other mechanical properties such as hardness, ductility, and toughness must be taken under consideration before and after post-weld heat treatment (PWHT).

Because of their substantially large dimensions, the process for heat treating steam pressure vessels used in energy generation involves the use of ceramic pad heaters applied to the welded surface. In many cases, the interior of the pressure vessel is not accessible and heaters must be applied to the exterior surface only. The industry recognized standard for PWHT parameters of Grade 91 steel currently dictates an ideal temperature of 760°C for two hours but the feasibility of maintaining these precise conditions during actual fabrication is questionable. Therefore, a quantifiable understanding of the allowances for variation in the PWHT parameters must be developed in order to ensure that the final mechanical properties of the steam pressure vessel will meet design requirements.

Grade 91 steel, more formally known as modified 9Cr-1Mo is a martensitic Cr-Mo steel that has been microalloyed with vanadium and niobium and has a controlled nitrogen content. After welding, brittle martensite with unfavorable material properties is formed in the weld metal. Thus, heat treatment is required in order to produce tempered martensite with precipitated carbides and vanadium/niobium-rich carbo-nitrides which provide for acceptable material properties [2].

According to Newell [3], a number of different parameters must be considered during the implementation of a PWHT process. First, the lower critical transformation temperature must not be exceeded. This requires that the chemical composition of both the base and filler metals be known so that the lower critical temperature ($A_1$) can be estimated. Next, the required duration of time at temperature is dependent upon the thickness of the material. With data suggesting that thin sections (thinner than 13mm, or 0.50 inch) can be tempered in 15 to 30 minutes while thicker sections should receive a minimum of 2 hours at temperature. Finally, the time between welding and PWHT should be kept to a minimum in order to reduce the possibility of cold cracking. These guidelines produce a framework for industry procedures but do not fully quantify the effects of variance from these guidelines.

In regard to creep-rupture properties, the fine grain heat affected zone (FGHAZ) is considered to be the weakest region. Type IV cracking occurs in the FGHAZ and is a major cause of premature creep failure at lower stress levels and higher temperatures [2, 4]. While it is believed that the risk of Type IV cracking can be reduced by conducting a thoroughly controlled PWHT process, experimental results have not clearly quantified this phenomenon [3, 5, 6]. Further testing must be conducted in order to correlate the effect of PWHT on Type IV creep rupture.

In a study conducted by Smet [2], material hardness and impact toughness were correlated with PWHT time and temperature. The Larson Miller parameter (LMP) was used in the study in order to combine the effects of PWHT time and temperature and compare them to hardness and
toughness values. A range of 750°C to 770°C with an LMP value near 21 was suggested in order to provide acceptable high-temperature properties. Emphasis was placed by previous studies on hardness testing as a reasonable means for verification of proper PWHT.

**Weldability of Grade 91** – Modified 9%Cr-1%Mo martensitic steel Grade 91 (Grade 91 for plate/piping and T91 for tubing) is increasingly used for components subjected to temperatures up to 1120°F in the power generation and nuclear industries, due to Grade 91’s favorable physical properties, superior creep, and oxidation resistance [7]. Welding is an indispensable process in the fabrication of nuclear pressure vessels. Welded Grade 91, however, generally has a lower creep and creep-rupture performance than the base material. For optimum joint properties, the welding procedure of Grade 91 calls for AWS E90XX, ER90, or EB90 series low-hydrogen filler metals, along with a 450-550°F preheat and a post-weld heat treatment (PWHT) at 1350-1400°F for a minimum of 2 hours [3]. Deviations from the optimum welding and PWHT procedures have resulted in serious loss of weld strength and creep rupture properties [1, 8, 9].

For heavy sections and assembly joints made in the field, the PWHT usually cannot be conducted using the optimal procedure. As a result, the creep and creep rupture properties of the joints may be compromised. Currently, a quantitative understanding is not available to correlate the specific thermal history, microstructure, and groove geometry of a Grade 91 weld between its expected creep and creep rupture properties.

Air-cooled and tempered Grade 91 is comprised of a tempered martensite matrix with dispersed precipitates of vanadium nitride, niobium carbonitride ($M_X$-type), and $M_23C_6$-type carbide particles [10]. Welding produces a weld fusion zone and a heat-affected zone (HAZ) in the resultant joint. The weld fusion zone has a chemical composition that is determined by the filler metal and the amount of dilution of the base metal. The heat-affected zone is an unmelted region adjacent to the weld fusion zone that has experienced microstructural and mechanical property changes from the welding process. Since each point in the heat-affected zone experiences a different thermal cycle, its microstructure is highly heterogeneous. A weld joint can therefore be considered as a “composite” consisting of layered materials, each with different properties, sandwiched between two pieces of base metal.

Creep-rupture testing of cross weld “composite” specimens under lower stress levels fails predominantly in the so called Type IV fracture. The softening zone and fine-grained zone in the HAZ of the martensitic-type alloy has a lower creep strength than other parts of the joint, leading to a premature creep failure [4]. While it is widely believed that a properly conducted post-weld heat treatment (PWHT) helps address Type IV fracture in creep, conflicting results have been reported on the effectiveness of heat treatments [3, 5].

Albert et al. [6] studied a PWHT for different durations at 1013K with furnace cooling and its effect on creep life. It was found that longer soaking times at temperature increased the creep life only slightly. Since the authors did not test other PWHT temperatures, no conclusion can be drawn on the effectiveness of PWHT. They did point out that factors other than microstructure might have influenced the creep rate. Specifically, the stress state due to heterogeneous heat-affected zone microstructure seemed to be the key for the stress peak at the fine-grained zone, where Type IV fracture occurred.

Abd El-Azim et al. [11] identified the reasons for the low creep life of the fine grained region as (1) the finer prior austenite grain size increased the rate of softening and creep cavitation, and (2) the lower peak temperature in the fine grained region produced a softer martensite matrix.
Watanabe et al. [12] found that the rupture locations for welded Grade 91 joints shifted from the weld metal at the higher stress condition to the fine grained HAZ adjacent to the base metal at lower stress conditions. A remarkable decrease of dislocation density and growth of precipitates of $M_{23}C_6$ and Laves phase during creep were observed in the fractured fine-grained HAZ.

Cross-weld creep tests at lower temperatures and higher stress levels have similar creep resistance as the base metal. Failure randomly happens in base metal, weld metal, or HAZ. At lower stress levels and higher temperatures, the HAZ appears to be the weakest link, decreasing the weldment creep strength to 50% of base metal. Failure formation and propagation is mostly located in the fine grained (FGHAZ) and intercritically heat-affected (ICHAZ) regions of the HAZ. Strict differentiation between ICHAZ and FGHAZ is difficult due to similar microstructural features [13]. The method to eliminate or control the fine grains through welding and heat treatment thermal parameters seems to be a critical question.

**Project objectives** – In the NEUP 2009 “G4A-1 Graphite Recycle Issues” Scope Area, there are two relevant objectives: (1) Characterization of creep and creep rupture properties of Grade 91 in suboptimal heat treatment conditions that might be anticipated due to heavy sections and heavy section welds, and (2) Mechanistic understanding of stress and temperature conditions, bounding negligible creep regime for Grade 91 steel, in optimal and suboptimal heat treatments of heavy section welds.

Responding to the first objective of the workscope, this project conducts a systematic metallurgical study on the effect of PWHT on the creep rupture properties of Grade 91 heavy section welds. The project consists of four interdependent tasks: (1) Characterize, experimentally and numerically, the temperature fields of typical post-weld heat treatment procedures for joint configurations to be used in Gen IV; (2) Characterize the microstructure of various regions, including the weld fusion zone, coarse-grain heat affected zone, and fine-grain heat affected zone, in the welds that underwent the various welding and post-weld heat treating thermal histories; (3) Conduct creep and creep-rupture testing of coupons extracted from both actual welds and physically simulated welds; and (4) Establish the relationship among PWHT parameters, thermal histories, microstructure, creep and creep-rupture properties.

### 2. Experimental Procedure

A survey of industry and study of Gen IV weld plans identified the target thickness of heavy section Grade 91 to be at least 5 inches. To facilitate the experimental study of welding processes and procedures, 1 inch thick Grade 91 pipe was used first. The optimized welding procedure was then used on the 5 inch thick heavy section weldment. During all the experimental welding, thermal histories at different locations of the joint, including the fine-grained heat-affected zone, were recorded with an array of thermocouples. The as-welded and heat-treated microstructure and hardness of the welds were characterized with quantitative metallography. The impact toughness of the as-welded and heat treated HAZ was also measured. The creep specimens were characterized before and after testing. Microstructure evolution during the post-weld heat treatment were characterized and correlated with creep performance of cross-weld specimens. The cross-weld creep behavior was modeled using a finite-element method in which the measured minimum creep rates of individual weld regions were used as inputs.
2.1. Base and filler materials

The seamless steel pipe ASTM/ASME A/SA335 Grade 91 used in this study had an 8 inch length, 8.625” diameter, and 1.143” wall thickness. The heavy section Grade 91 material for creep rupture study were custom made according to ASTM A182F9 Grade 91. These forged blocks have dimensions of 5” x 9” x 12”. The chemical composition and heat-treatment of the as-received pipe and block materials are shown in Table 1.

2.2. Welding procedure

A comprehensive review of welding procedures was conducted for heavy section Grade 91. Based on the review, gas-tungsten arc welding (GTAW) root pass and flux-cored arc welding (FCAW) filling passes were selected in this study. Both GTAW and FCAW are readily automated for high-production deposition of heavy section welding; but the more important factor in both welding processes is the readily available high quality weld metal. Specifically, GTAW can retain the filler metal composition by argon shielding, and FCAW can guarantee the chemical composition of the weld metal through the use of flux, while bare wire gas metal-arc welding (GMAW) cannot [3]. Compared with submerged arc welding (SAW), the other frequently used welding process for heavy sections, both GTAW and FCAW use much less heat input, and therefore produce a much narrower heat-affected zone.

During the entire preheating, welding, and post-weld baking process, temperature profiles of a region close to the weld on the ID of the pipe were measured using 16 type-K thermocouples. Two 8-channel data loggers were used to record the temperature measurements. A sampling frequency of 5 Hz was used. A surface temperature probe was also used to determine the inter-pass temperatures. The temperature measurements served not only as the in-process quality control of preheat and inter-pass temperatures, but also used as verification and calibration data for the welding thermal models.

Actual welding, including selection of fixtures, filler metals, welding process, preheating, welding parameters, inter-pass temperature, and post-weld cooling, was conducted in the Welding Laboratory at USU, using a factorial design-of-experiments method. The laboratory was set up to represent a standard manufacturing facility. Figure 1 shows the pipe welding setup.

The test weld groove had a 60° included angle with 1.5 mm root face. Mill scale near the weld zone was removed before welding. The root pass was welded by GTAW with 300A DC, with 1.27 m/min wire feed speed. The electrode was φ1/8 inch 2% thoriated tungsten with 90° tip angle. Pure Argon shielding was used. The filler metal was φ1.2 mm ER90S-B9. The 0.14 m/min linear travel speed was maintained by a stepper motor controlled fixture. Flux-cored arc welding (FCAW) was used to fill the groove following the GTAW root pass. The filler wire was φ1.2 mm ER91T1-B9. The wire feed speed was 6.35 and 7.62 m/min, with 26.1 and 27 Volts arc voltage. The linear travel speed was 0.292 m/min. A mixed 75/25 Ar/CO2 shielding was used. For FCAW, slag removal was necessary. A pneumatic descaler and wire brushing were used. Each of the pipe sections were initially prepared with a 60° included angle V-groove joint. They were then pre-heated to a temperature between 150 and 250°C.

For the 5” thick plate sections, two different weld joint configurations were used. The first of which was a dual groove with a 60° included angle 7/8” high at the bottom of the joint and a 30° included angle maintained through the rest of the joint. The second weld joint employed
the same 60° included angle 7/8" high at the bottom of the joint but reduced the included angle throughout the rest of the joint to 20° in order to reduce the total amount of weld passes needed to fill the joint. The plate sections were pre-heated to a temperature between 150 and 250°C and welded solely using FCAW with φ1.2 mm ER91T1-B9 filler wire and a 75/25 Ar/CO₂ mix for shielding gas. Figure 2 shows the block welding setup and a welded specimen.

Following the welding procedures for the pipes and plate sections, a post-weld bakeout was performed. For the pipes and the second plate section, an electric furnace was used to heat the weldments to 250°C for 4 hours, after which they were left in the furnace to cool. Because the specimen was too large to fit into the electric furnace, the first welded plate section was subjected to a post-weld bakeout using ceramic pad heaters enclosed by ceramic bricks. For this reason, the precise temperature of the weldment during the bakeout was maintained between 150 and 250°C.

The first eight pipe sections were welded using various pre-heat, heat input, and interpass parameters with the objective being to find a set of optimum welding parameters. For each parameter, a high and a low setting was chosen and a matrix was created to test every combination. For pre-heat, the high and low temperatures chosen were 250°C and 150°C. For interpass, temperatures of 300°C and 200°C were chosen for the high and low. With wire feed speed at a given voltage being the means of controlling the heat input, a high and a low setting of 7,620 mm/min at 27V and 6,350 mm/min at 26V were selected. Figure 3 summarizes the test matrix of preheat, inter-pass temperature, and heat input for the welding experiments.

After analyzing the data collected from testing on the first eight welded pipe sections, a pre-heat temperature of 150°C, an interpass temperature of 300°C, and a wire feed speed of 7,620 mm/min at 27V were selected. These parameters were then used to produce two more pipe section welds. Finally, two 5" thick plate section welding procedures were conducted using the selected heat input parameters. However, because the preheat and interpass temperatures were maintained using the ceramic pad heaters, the preheat and interpass temperatures experienced a variance of no more than +/-25°C from the target values.

2.3. Heat treatment

Nearly all heat treating operations were performed using a Cress electric furnace Model C162012 /SD. The furnace temperature was verified using high temperature type-K thermocouples with Pico USB TC-08 data loggers to ensure precise conditions were maintained. All heat treating procedures were performed in a systematic and repeatable manner that would produce consistency amongst the results.

The post weld heat treatment operations were conducted based on the industry recommended 760°C for 2 hours. For the first set of eight pipe section welds, the post weld heat treatment was carried out at this temperature and time. However, after optimization of the welding parameters, various heat treatment temperatures and times, ranging from 650 to 840°C and 2 to 8 hours, were selected in order to better quantify the effects that variance will have on the mechanical properties of the Grade 91 weldments. All heat treated specimens were air cooled. The temperature and time response of the welds under various heat treatment parameters were acquired. Numerical thermal simulations were conducted to understand the temperature fields as influenced by the weld geometry and heat treatment conditions.
2.4. **Microstructural characterization**

The microstructure of all welded and heat treated welds was characterized using optical microscopy and SEM and EDS, following a standard procedure of sample preparation. The metallography samples were extracted from each of the welds. These samples were mounted in epoxy, polished, and then acid etched with a 10% nital solution to reveal the microstructure. A traverse of Vickers hardness data points was then taken starting in the base material, spanning across the HAZ, and ending in the weld metal. To differentiate martensite, tempered martensite, and ferrite phases, the LePera etching [14] (4% picric acid in ethanol mixed with a 1% sodium metabisulfite in distilled water in an 1:1 volume ratio) was also used. The phases in various weld zones were further verified by XRD.

In order to experimentally estimate the critical transformation temperatures of the P-91 material used, a dilatometry sample was manufactured for the weld metal, pipe, and thick plate sections. These samples were first rough cut on the band saw and then machined into \(10\) mm by \(100\) mm long round bars. The round bars were then hand sanded to produce a smooth surface finish. The dilatometry procedure was performed on a Gleeble mechanical testing system.

2.5. **Ductility and toughness**

To characterize the effect of varying heat treatment parameters on the ductility of welded samples, a series of three point side bend samples was created. The samples were were cut to approximately \(10\) mm thick on the band saw with the weld metal in the center of the sample. They were then exposed to a heat treatment with parameters varying from \(400\) °C to \(842\) °C for anywhere from \(30\) minutes to \(8\) hours. After heat treatment, the samples were machined flat for consistency. Finally, the samples were subjected to a three point bend test according to the AWS B4.0 standard which produced either a complete fracture or a \(180\)° U-shaped bend.

The first set of samples was a series of \(80\) charpy impact coupons that were extracted from the first eight pipe welds (10 from each pipe). Standard Charpy impact V-notch specimens were prepared according to ASTM 370 [15]. Each of the samples was rough cut using a band saw and an abrasive saw with a coolant. Then the samples were machined to approximately \(0.25\) mm oversize and finally precision ground to obtain a \(10\) mm \(\times\) \(10\) mm cross section with approximately \(55\) mm length. All specimens were macro-etched to reveal the fusion boundary, which served as the location for the notch such that the fracture path would traverse the heat-affected zone.

To “magnify” the small heat-affected zone regions in order for large samples of similar microstructure to be tested, a Gleeble was used to simulate the multi-pass welding process [16]. The measured thermal cycle for each individual HAZ zone was reproduced in three smooth Charpy specimens. The simulated samples were then heat-treated at different temperatures from \(600\) to \(840\) °C with a temperature difference of \(40\) °C. Notches were machined in the middle of the test specimens. Two specimens for each tempering temperature were tested and both results were reported. The average energy values for these “pure” metals (of the simulated HAZ regions, fusion zone, and base metal) are listed in Table 2. This method of creating simulated samples by using the thermal cycle is different from most previous studies, because not only the first peak temperature but also the subsequent temperature peaks by multi-passes were applied to achieve similar properties of the as-welded samples.
2.6. Creep test

A series of conventional creep testing samples were extracted. From the optimized pipe welds, eight samples were rough cut out and then heat treated at either 840°C, 820°C, 760°C, or 600°C for either two or eight hours. From the thick plate section welds, 4 blocks were cut out and heat treated at either 800°C, 760°C, 740°C, and 650°C for 5 hours each. It should be noted that the thick section samples were heat treated for five hours each to correlate with one hour for each inch of thickness. Also, a single base metal sample was cut from both the pipe and the thick plate section in the as-received condition to produce a bench mark test. After heat treating, the samples were machined to precise dimensions, and finally hand sanded to remove machining marks that could produce stress risers and promote premature crack propagation.

The cross-weld specimens were extracted from the as-welded pipe along the longitudinal direction with effective uniform gauge length of 84 mm. The weld metal was kept in the middle of the specimen so that heterogeneous material properties will be represented in the gauge length.

The creep test specimens were standard plate specimen with shoulder ends as shown in Figure 5 with cross section 8.8 × 12.7 mm and 84 mm effective gauge length. The standard test procedures specified by ASTM E139-06 were followed. The load was applied via pins placed in through holes in the shoulder ends of the specimen. Eight heat treated and one as-received base metal specimens were creep tested at the applied test temperature of 650 °C and engineering stress of 70 MPa (10 ksi). The temperature inside the furnace was controlled so that the fluctuation was ±1.7 °C. Two strain gauges were placed in the front and rear sides of the specimen and average strain was reported as a function of time until rupture.

Finally, a set of stress relaxation coupons was extracted from the pipe sections. After rough cutting, each of the samples was exposed to a heat treatment of either 840°C, 820°C, 760°C, or 600°C for either 2 or 8 hours. Additionally, a single base metal sample was cut from the pipe to serve as a bench mark. Each of the samples were then machined into 10 mm by 10 mm square bars approximately 150 mm long. A reduced cross sectional area was machined at the center of the samples that was 5.3 mm by 5.3 mm and 50.8 mm in length. The Gleeble mechanical testing system was used to perform all stress relaxation tests.

3. Results

3.1. Ductility characterization

The bend test was selected to establish a distinction between brittle and ductile behavior in welds as affected by post-weld heat treating. This test is essentially a three point bending test in which the welded sample was placed with the weld metal at the center point of the bending fixture and an increasing deformation was applied to the fixture until the sample either fractured or was bent 180° into a U-shape. This test allowed for a large quantity of samples with a wide variety of heat treating parameters to be tested efficiently. The outcome is a range of possible temperatures and times for heat treating in which the weld metal exhibits ductile behavior at room temperature. Figure 6a shows tabulated ductility results obtained from each of the side-bend tests. From these results, a plot was created to estimate the range of temperatures and times that will produce ductile behavior. This plot is shown in Figure 6b.
It can be seen that as heat treating time increases, the range of temperatures in which samples show ductile behavior also expands. The industry standard for post weld heat treating of Grade 91 steel at 760°C for 2 hours remains encapsulated at the center of this matrix and appears to be the among the safest pairs of time and temperature for heat treating in order to ensure ductility. It can also be seen that if the time is decreased to one hour and held at 760°C for temperature, there is only a 40°C ductile range below it. This could be an issue in certain industry situations where a high level of temperature accuracy during heat treatment is not feasible.

All samples that failed completely did so in the center of the weld metal. However, it appears that the majority of samples that failed at temperatures below 600°C had a straight line brittle crack directly perpendicular to their length. In contrast, samples that fractured at or above 827°C had a jagged crack at approximately 45° angles to their length indicating failure in shear. Samples near the boundary between ductility and fracture also appear to exhibit failure in shear.

3.2. Microhardness

For the first set of eight pipe section welds, Vickers hardness values were taken from each of two samples from every pipe weld. One being in the as-welded condition and the other after exposure to a heat treatment of 760°C for 2 hours. Several data points were taken in a traverse starting in the weld metal, spanning across the HAZ, and ending in the base metal. It can be seen that after heat treatment, hardness variance between the base metal, HAZ, and weld metal is nearly eliminated.

Figures 7 and 8 compare for each welding experiment the hardness profiles of the as-welded and heat treated samples. For all the welding parameters, the as-welded welding fusion zone and majority of the HAZ has a hardness number higher than 400Hv, indicative of the martensitic microstructure. Heat treatment at 760°C is shown to temper all the weldments to a hardness level close to that of the as-received base metal (220 - 240 Hv). The effect of the welding parameters on the as-welded hardness levels can be seen from Table 3, in which the results are listed along with the test conditions.

3.3. Toughness of the heat-affected zone

The impact energy values for the heat-affected zones (HAZ) in the as-welded samples are shown in Figure 9. The average impact energy exceeds 180 J for the as-welded samples. The difference in impact energy values with different process parameters during welding is not significant, although a greater preheat temperature (250°C) seems to have produced wider scatter in impact energy of the HAZ. Lower preheat (150°C) seems to have produced a much narrower scatter band in impact energy of the HAZ. A few as-welded samples have impact energy values close to 30 J. An inspection of the fracture path reveals the propagation of fracture in these low toughness specimens that originates from the notch, passes through the weld metal, the HAZ, and the base metal (Figure 10). The weld metal in the as-welded condition has an impact energy of 7 J. Clearly, the measured impact energy is a sensitive function of the position of the notch for the heterogeneous weld joint. Similar observations have been made by other researchers, such as Moitra et al. [17] and Jang et al. [18], in a study of the effect of notch location on impact toughness of weld metal and heat-affected zone.
After a post-weld heat-treatment at 760 °C for 2 hours, the impact energy of all heat-affected zone specimens has increased consistently (Figure 11). The minimum energy level for joints made using different welding parameters is 220 J. The wide scatter of impact energy levels for the as-welded weld HAZ has been narrowed down. An inspection of fractography of tested specimens revealed the fracture paths to be consistently starting from the notch, traversing the HAZ and the base metal. None of the fracture paths in the heat-treated samples has deviated into the weld metal, which after the 760 °C for 2 hours heat-treatment, has the impact toughness increased from 7 J to 56 J.

The Gleeble-simulated microstructure is verified to be similar to that from the heat-affected zones of the welded samples. An example comparison of microstructures for the coarse-grained heat-affected zone (CGHAZ) is shown in Figure 7. The two microstructures not only share the same grain size, but also the martensitic substructure, the size and amount of carbide particles. Such simulated microstructure for the entire cross-section of the Charpy specimen enables an accurate evaluation of impact toughness of individual heat-affected zones.

The Charpy impact results of the simulated HAZ samples heat treated at various temperatures for two hours are shown in Figure 13. Among the three HAZ regions, the inter-critical heat-affected zone (ICHAZ) exhibits the highest toughness, while the CGHAZ has the lowest toughness and fine-grained heat-affected zone (FGHAZ) has an intermediate toughness. The CGHAZ exhibits the lowest impact energy following a 600 °C - 2 hour heat treatment. This low toughness remains until the PWHT temperature is at 720 °C. The impact energy of CGHAZ then increases significantly when the PWHT temperature is 760 °C. A post-weld heat treatment at 800 °C results in the peak toughness for the CGHAZ. The impact toughness then decreases when the PWHT temperature is 840 °C. The ICHAZ toughness remains at 220 J for PWHT temperatures below 760 °C, and reaches the peak value following an 800 °C heat treatment. The ICHAZ toughness also decreases when the PWHT temperature is 840 °C. The FGHAZ toughness increases with a higher PWHT temperature between 600 and 720 °C. After the 720 °C PWHT, the FGHAZ toughness has increased to the same level as that of the ICHAZ. Further increases in the PWHT temperature from 720 °C result in the exact same toughness for both the FGHAZ and ICHAZ. A notable trend is that all HAZ regions reach the peak toughness following an 800 °C PWHT; and all HAZ regions lose toughness following a 840 °C PWHT.

The measured impact toughness reported in Figure 11 represent the total energy for the fracture to traverse the entire specimen. The fracture path may have traversed the weld metal, various HAZ zones, and the base metal. As a first approximation, we can consider the total impact energy (\( E_{\text{Calc}} \)) to be consisted of a linear summation of contributions by various zones:

\[
E_{\text{Calc}} = E_{\text{CGHAZ}} + E_{\text{FGHAZ}} + E_{\text{ICHAZ}} + E_{\text{WM}} + E_{\text{BM}}
\]

where \( E_{\text{CGHAZ}} \) is the energy contribution of the coarse-grained heat-affected zone, \( E_{\text{FGHAZ}} \) is the energy contribution of the fine-grained heat-affected zone, \( E_{\text{ICHAZ}} \) is the energy contribution of the inter-critical heat-affected zone, \( E_{\text{WM}} \) is the energy contribution of the weld metal, and \( E_{\text{BM}} \) is the contribution of the base metal.

Because the Charpy specimen has a uniform width, the contribution of individual zones to the total Charpy impact energy can be calculated using the measured fracture length in each zone. For example, for the contribution of base metal (\( E_{\text{BM}} \)) to the total impact energy, the following
A formula can be used:

\[ E_{BM} = \left( \frac{L_{BM}}{L_{Total}} \right) E_{0}^{BM} \]  

(2)

where \( L_{BM} \) is the length of the fracture path that falls in the base metal, \( L_{Total} \) is the total length of the fracture path of the Charpy specimen, and \( E_{0}^{BM} \) is the measured impact energy of the “pure” base metal. Similar definitions can be made for \( E_{CGHAZ} \) for the CGHAZ, \( E_{FGHAZ} \) for the FGHAZ, and \( E_{ICHAZ} \) for the ICHAZ, respectively. Experimentally measured impact toughness data from simulated HAZ, the base metal, and pure weld metal are listed in Table 2 for the as-welded and 760°C-2H heat-treated conditions.

Tested Charpy toughness specimens have been polished and etched to reveal the microstructure along the fracture path as shown in Figure 10. Measurements of the length of fracture path that falls in each microstructure zone have been taken. Sample calculations to predict the total Charpy impact energy are shown in Table 3. Impact energy data shown in Table 2 and measured fracture length fractions are used as input parameters for the calculations. Compared with the experimental impact energy value \( (E_{Exp}) \), the calculated total energy \( (E_{Calc}) \) is within \( \pm 5\% \) difference. Using this linear summation method, a cross-weld HAZ toughness test result can be understood if the test specimen is measured metallographically for the fracture length and the toughness of individual HAZ zones are determined.

3.4. Thermal measurements

Recording thermal history is a crucial part of examining the effects of welding and post weld heat treating. In addition, recorded temperature data was used to reproduce samples on the Gleeble with the same microstructure as selected regions of the HAZ. The temperature profile in Figure 14 is representative of a typical pipe welding procedure. With thermocouples fixed to the inner diameter of the pipe during welding, it can be seen that each successive weld pass produces a significantly lower peak temperature at the inside surface of the pipe.

Temperature measurements of multipass welding can provide insight to the welding metallurgy, and provide valuable validation data for numerical and Gleeble physical simulations. The recorded thermal cycles near the root of the weld support the HAZ width measurements by the microstructure (Figure 17). 2.5 mm away from the FCAW fusion line gives the average HAZ width. The peak temperature clearly indicates that at 2.5 mm, most of the welding conditions have produced a temperature high enough to reach \( A_1 \). The HAZ width near the GTAW root pass is near 4.5 mm. The thermal cycle recording for 4.5 mm location clearly shows the peak temperature to be close to \( A_1 \). The general effect of welding parameters on the thermal cycles has been correlated with the microstructural observation of the HAZ width.

The width of the HAZ is influenced in a complicated way by the welding parameters. Shown in Figure 16, it can be seen that if other conditions are the same, the HAZ width in the pipe welds increases with a higher heat input. It also increases with a higher inter-pass temperature. And further, the HAZ width increases with the preheat temperature. Among these three welding parameters, the heat input is the most effective in increasing the HAZ size.

The post-weld heat treating procedure that was performed on the thick plate section welds was monitored for the 760°C case. Figure 15 shows the heating and cooling curve for the same
block after the measured temperature data was filtered using a simple Matlab numerical pro-
gram. While the surface of the block reaches the target temperature in approximately 5000
seconds, further modeling by FEM determined the time at which the center of the block reaches
this temperature would require another 2400 seconds. Therefore it would take 7400 seconds
(approximately 2 hours) for the center of a 5 inch block to reach the the set temperature of 760
°C. The post-weld heat treating hold time of 5 hours in this study is therefore justified.

3.5. Creep results

Figure 18 shows the creep curves of Grade 91 pipe cross-weld specimens tested at 650 °C and
stress level of 70 MPa. The as-received pipe base metal as a comparison has a low creep strain,
and the test was terminated after all weld specimens have ruptured. At the time of termination,
the base metal was still in the secondary creep regime. All the welded and heat-treated specimens
exhibit typical three-stage creep curves.

The results from pipe welds creep tests are summarized in Table 4. All the welded specimens
with different PWHT conditions ruptured below 1000 hours of test time. To compare the effect
of PWHT on the creep properties, minimum creep rate and average creep rate are computed. The
minimum creep rate is the minimum point on a curve in Figure 18b. The average creep rate ($\dot{\varepsilon}_{\text{avg}}$)
is calculated as $\dot{\varepsilon}_{\text{avg}} = \varepsilon_t / t_R$, where $\varepsilon_t$ is the total creep strain at rupture and $t_R$ is the rupture time.
The specimen with PWHT at 840 °C for 8 hours has ruptured with the shortest time of 205 hours.
The rupture occurred at the weld fusion zone. The specimen with PWHT temperature 840 °C
for 2 hours ruptured at 388 hours and the observed rupture location was also at the weld fusion
zone.

The creep strain and creep strain rate for the base metal is shown in Figure 18a. The base material
has the minimum creep rate of 0.0003% h⁻¹. The creep test results for base metal for longer times
can be found in literature [19].

Creep test results for the 5-inch block welds are shown in Table 5 and Figure 19. The block
welds have significantly increased creep properties compared with those of the pipe welds. The
rupture time for the 800 °C - 5 hour treated specimen is 1127 hours. This rupture time is longer
than the best performing pipe welds (925 hours of the 600 °C - 2 hour treated specimen). Testing
of other specimens were terminated at 1600 hours. The minimum creep strain rate and total
creep strain data indicate that a lower post-weld heat treatment temperature gives better creep
properties.

The Type IV failure occurs when creep tested at 650 °C and stress level of 70 MPa for different
PWHT conditions below the $A_1$ temperature. Type I failure occurs when the PWHT is performed
at 840 °C. From the results presented above, the specimens post-weld heat treated below $A_{c1}$
temperature ruptures at ICHAZ. The specimen with PWHT temperature close to $A_{c1}$ i.e. 820
°C for 2 hours ruptures at ICHAZ while for 8 hours the rupture shifts to the weld deposits. The
rupture location is along the ICHAZ region and necking has also been observed as the weld
slips down in a perpendicular manner to the loading direction. Similar rupture locations and
times have also been observed in reference [20] (material condition - steel plate 140 mm thick,
creep condition - 625 °C and 80 MPa) while failures in the weld deposits have been observed
in reference [12] (material condition - steel plate 25 mm thick, creep condition - 650 °C and 80
MPa). However the PWHT temperature in both studies were 760 °C for 20 hours and 743 °C for
8.5 hours and the material was Grade 91 plate.
There is a small difference in minimum and average creep rates between PWHT 600 and 760 °C PWHT. However, the minimum and average creep rates increase as the PWHT temperature is increased. When the PWHT temperature is above 820 °C temperature, there is a sharp rise in minimum and average creep rate. Among all the PWHT conditions, 600 °C exhibits the lowest minimum creep rate while 840 °C 8 hours exhibits the highest creep rate. This high creep rate predicts an early rupture of specimen heat treated at 840 °C 8 hours. The as-received base metal creep test results show that it has a lower creep rate than any of the welded and PWHT-ed specimens.

4. Discussion

4.1. Thermal models and verification

A 3D finite element (FE) model was built using ANSYS to simulate the welding process, and to find the temperature history for the inner regions of the heavy section weld, where experimental measurements are not available. The actual bead geometry obtained by experimental measurement was used as the prototype of the FE model (Figure 20). The simulation of Grade 91 welding process focused on the heating and cooling of the beads from the melting temperature. This approach has the advantage of not having to solve the fluid dynamics of arc-material interactions, although the effect of fluid dynamics can still be considered. The 3-D 10-node tetrahedral thermal-solid element and the element re-activation technology were employed to perform this thermal analysis. The finite elements in the molten pool that were being deposited were activated as the heating source moved to position. As the heating source moved to the next position, a new molten pool volume was activated. The heat dissipation was difficult to estimate and strongly dependent on the surrounding conditions (e.g., radiation effect and the absorbability) of the base material and deposited materials. Therefore, the arc heating power was selected as the fitting parameter. It was adjusted until the predicted temperature by the FE model agreed well with the experimental temperature data. In addition to the heat conduction in solid-state beads and base material, heat transfer on the exposed surfaces was also included in the simulation as boundary conditions.

For characterization of the thermal history of weldments during heat treatment, a numerical model was also developed. Radiation heat transfer of Grade 91 inside the furnace was modeled in ANSYS using SURF 152 element and SOLID70 thermal element. A test coupon initially at room temperature was supported in such a manner that it gained heat by radiation from all its surfaces exposed to the environment at a temperature of 760°C. Transient analysis was conducted in order to obtain a temperature profile of the coupon during the heat treating period. The model predicted temperature change in the coupon was verified by the experimental measurements.

A realistic geometry representing the multibead thick section welds was incorporated in the FEM model (Figure 21). Results from the simulation were calibrated by the experimental data. Predictions of temperature fields for locations within the weldment were made. These locations are not accessible by temperature measurement equipment. One useful verification is that 2 mm away from the root GTAW pass, the temperature is 1433°C. The actual microstructure shows a coarse grained region, verified by the temperature range. The model predictions have been used in selecting the Gleeble simulation conditions to produce required HAZ microstructures.
Similar to the welding model, a FEM model for the heat treatment was developed. Predictions were made for the middle-thickness location, in which the temperature changes with time in furnace (Figure 15b). For an 1 inch sample, it would take more than 2400 seconds for the middle-thickness to reach the furnace set temperature. This model has been useful for analysis of heavy section sample heating and cooling following furnace heat-treatment.

4.2. Microstructure evolution during welding and PWHT

Critical transformation temperatures – When heated to the austenitic temperature range, steels transform from room temperature ferritic microstructure to austenite. Subsequent cooling from austenite produces various phase transformations according to steel chemical composition and cooling rate. The equilibrium transformation in Grade 91 martensitic steel to form austenite starts at $A_\text{l}$, the lower transformation temperature, and finishes at $A_\text{m}$, the upper transformation temperature. During welding and heat treating, these transformation temperatures change because of the non-equilibrium heating and cooling. Conventionally the on-heating transformation temperatures are designated as the $A_\text{c1}$, the austenite transformation starting temperature, and the $A_\text{c3}$, the austenite transformation finish temperature. The equilibrium $A_\text{l}$ temperature can be calculated according to [3]

$$A_1 (^\circ C) = 845.5 - 43.9(Mn + Ni) - 9(Mn + Ni)^2$$ (3)

where $Mn$ and $Ni$ are the manganese and nickel contents (wt.%) in the alloy. The $A_1$ of the alloys under investigation has been calculated as 824 $^\circ$C for the pipe material, 811 $^\circ$C for the block material, 801 $^\circ$C for the root pass GTAW wire, and 771 $^\circ$C for the flux-cored filler wire. The transformation temperatures identified by dilatometry for slow heating (10 $^\circ$C/hour) and air cooling are $A_\text{c1} = 818 $^\circ$C, $A_\text{c3} = 925 $^\circ$C, and $M_\text{s} = 394 $^\circ$C for the as-received pipe base material. The critical temperatures of the different metals are estimated and arrows on the plots indicate the location of the departure from linear behavior. This departure from linearity indicates a change in microstructure and is therefore used to determine the critical temperatures (Figure 23). It can be seen that the calculated $A_1$ is very close to the slow-heating $A_\text{c1}$ identified by dilatometry. Table 6 summarizes the calculated and measured transformation temperatures of the test materials. If the PWHT temperature is above the $A_\text{c1}$ temperature, austenite will start to form, which on-cooling in air will transform to fresh martensite.

The as-received material of Grade 91 has undergone a normalization at 1060 $^\circ$C and tempering at the temperature of 786 $^\circ$C. The as-received microstructure consists of fully tempered martensite and ferrite along with finely dispersed M$_{23}$C$_6$-type carbide. Figures 24a and 24b show the XRD analysis of the as-received material, indicating the ferritic (bcc) crystal system for the tempered martensite, and the existence of M$_{23}$C$_6$-type carbide.

As-welded microstructure – Macro-etch clearly shows the GTAW root pass with multiple FCAW filling beads (Figure 25) in the as-welded condition. The fusion zone beads are partly tempered by subsequent beads. A small amount of martensite exists along the inter-dendritic boundaries in the un-tempered portion of the weld metal (Figure 26). The HAZ along the fusion line is uniform in width, except the HAZ near the root pass is twice as wide as that of the filling beads. The microstructure of the as-welded heat affected zone consists of several regions, as shown in Figure 27. The original coarse grain zone adjacent to the fusion line has been refined
by the subsequent weld bead such that it does not have the largest grain size. The region outside the original coarse grain zone is shown to have the largest grain size. Further outside the coarse grain zone is the fine grain zone. Even further outside, adjacent to the base metal, is the intercritical zone, in which a mixture of fine grains and original base metal grains can be seen together.

**Microstructure following PWHT** – Weld fusion zone microstructure changes during PWHT. Figure 28 shows the effect of heat treatment temperature on the microstructure and its hardness. The as-welded microstructure is a mixture of tempered martensite and some untempered martensite, with a 475Hv hardness. PWHT at 600 °C starts to temper the microstructure, and the hardness decreases slightly. PWHT at 760 °C to 820 °C significantly tempers the microstructure, with decomposition of tempered martensite to ferrite. The hardness decreases even more to 390Hv. PWHT at 840 °C, however, increases the hardness, because the heating temperature is above the \( A_1 \). Some fresh martensite has newly formed. The evidence is shown in Figure 29, in which finer fresh martensite is seen to disperse in the fusion zone of the 840 °C treated joint.

In the post-weld heat treated condition, the HAZ is more difficult to etch due to microstructural change and chemical homogenization. The HAZ microstructure shows that the coarse grain zone has seen martensite tempering, refinement of coarse grains through recrystallization, and the fine grain zone has seen grain growth (Figure 30). The microstructure changes during PWHT are better understood by looking at the associated hardness changes as a function of heat treating temperature (Figure 31). Because the microstructure is heterogeneous, a 1000 g load has been used during the Vickers hardness measurements to maximize the indentation size, so that an “average” hardness is obtained. In the as-welded HAZ, the hardest region is the CGHAZ, where the microstructure is fresh and tempered martensite. The second hardest is the FGHAZ, where the microstructure is fine-grained tempered martensite. The ICHAZ is the softest, where the microstructure is a mixture of ferrite from the base metal and tempered martensite.

PWHT at increasing temperatures, the hardness of all HAZ regions consistently decreases as martensite gets tempered and decomposed to ferrite. The HAZ reaches the softest state near the 800 °C heat treatment. However, further increase of temperature to 840 °C shows the significant increase in the measured hardness for all HAZ regions. Since 840 °C is above the \( A_{C1} \) temperature, this hardness increase is due to fresh martensite formation. Figure 24c provides the evidence of martensite formation in a specimen air-cooled from 840 °C. Fresh martensite and tempered martensite in Grade 91 share most of the XRD signatures, except the subtle broadening of the peaks near their bases [21]. Comparison of Figures 24a and 24c does show a broadening of peak bases due to fresh martensite. The arrows in Figure 24c indicate the traces of fresh martensite in the specimen.

Microstructural evidence for martensitic transformation for PWHT above the \( A_{C1} \) temperature is available after etching the specimens with the LePera regent, which reveals the fresh martensite in white, ferrite in tan, and tempered martensite and Bainite in a dark color [14]. In Figure 32, the microstructure of ICHAZ following an 840 °C, 2 hour PWHT is shown to contain approximately 15 vol. % of fresh martensite, 25 vol. % of ferrite, and 60 vol. % of tempered martensite.

The coarse grained HAZ has experienced a peak temperature between 1100 °C and the melting temperature of 1382 °C, during which there is a complete dissolution of carbides and significant grain growth of the high carbon and high alloy containing austenite. Fresh martensite that forms
upon cooling is strong and brittle. Although it has been tempered by subsequent thermal cycles in the multiple welds, the tempered martensite microstructure in the CGHAZ is still brittle (Figure 7). With a PWHT at 640 °C, more tempering of martensite has occurred, but the microstructure is virtually identical with that of the as-welded CGHAZ (Figure 33a). The toughness of CGHAZ therefore remains low (Figure 13), until the PWHT temperature is further increased to above 720 °C. The microstructure of 800 °C heat treated CGHAZ shows the tempering of martensite to ferrite, with associated carbide precipitation. Although the grain size remains the same as the as-welded condition (average 30 micrometer diameter), there are new finer ferrite subgrains and annealing twins formed (Figure 33b). The CGHAZ in this microstructure has the highest impact toughness. PWHT at 840 °C refines the grain size, and coarsens the carbide particles (Figure 33c), but produces the brittle fresh martensite, as explained earlier. The toughness therefore decreases from the 800 °C PWHT value.

The fine grained HAZ has experienced a peak temperature above $A_{C3}$ (925 °C) but below the temperature for austenitic grain growth. The austenitized FGHAZ has an average grain size of 8 micrometers that transforms to martensite on-cooling. The as-welded microstructure following multi-bead welding is tempered martensite (Figure 34a). A PWHT at 640 °C produces tempered martensite and some ferrite (Figure 34b) with dispersed carbide particles. Toughness is therefore recovered to above 100 J following the PWHT at 640 °C. The FGHAZ also shows the maximum toughness and minimum hardness following a PWHT at 800 °C, due to a microstructure of fine grained ferrite with fine dispersed carbide particles (Figure 34c). PWHT at 840 °C increases the grain size, and coarsens the carbide particles (Figure 34d), but produces the brittle fresh martensite. The toughness therefore decreases from the 800 °C PWHT value.

The inter-critical HAZ has experienced a peak temperature between the $A_{C1}$ and $A_{C3}$, therefore is partially austenitized on-heating. The multi-bead as-welded microstructure is a mixture of base metal’s ferritic constituent, newly formed martensite, and tempered martensite (Figure 35a). The 180 J toughness of ICHAZ is contributed mostly by the base metal, which has a toughness of 230 J. A PWHT at 640 °C further tempers the martensite (Figure 35b), but since the toughness is governed by the dominating base metal, no significant changes in the toughness is observed. The ICHAZ also shows the maximum toughness and minimum hardness following a PWHT at 800 °C, due to a microstructure of fine grained ferrite with fine dispersed carbide particles (Figure 35c). PWHT at 840 °C increases the grain size, and coarsens the carbide particles (Figure 35d), but produces the brittle fresh martensite. The toughness therefore decreases from the 800 °C PWHT value.

4.3. Creep damaged microstructure

Microstructure after Creep – The Type IV failure in creep tested weld joints is characterized by a creep damaged zone (CDZ) in which most of creep deformation is concentrated. In Figure 36a, the rupture is seen to have occurred along a narrow region that is parallel to the weld fusion line. This narrow creep damaged zone is where most of slip and necking deformation is concentrated. Between the two sides of the weld fusion zone, damage occurred simultaneously, but the weaker side was the first to rupture. For a v-groove weld joint, the weld as a whole slips against the base metal during creep that ends in Type IV rupture. A cross-sectional view of the creep damaged zone reveals that it is sandwiched between the base metal and the fine grained heat-affected zone (Figure 36c). In the CDZ, shear deformation and associated cavitation creep damage are apparent.
The ruptured interface shows the separation has been through the coalescence of cavities (Figure 37). The creep damaged zone on the other side of the weld shows cavities and micro cracks due to coalescence of cavities (Figure 38). At higher magnification, the cavities are seen to have formed along the ferrite grain boundaries in the inter-critical heat-affected zone in the 760 °C treated weld (Figure 39). The hardness traverses (Figure 40) of creep tested specimens further identify the Type IV rupture location as the softest (average 250Hv, indicative of ferrite microstructure). For the specimens failed Type I rupture, the softest region tend to have shifted to the fusion zone. The locations of failure can be found in Tables 4 and 5.

**Particle analysis** – Comparing the microstructure before and after creep testing, we noticed that the grain sizes remain mostly unchanged during creep, however, there is a significant increase in carbide particle counts as tempered martensite decomposes into ferrite during creep. Therefore the carbide particles are analyzed from creep ruptured specimens to prove the observation.

The particles from the as-received base metal are also measured as a benchmark. The size ranges from 75 to 270 nm in the base metal (Figure 41). EDS and XRD analysis proved that these particles are Cr-rich $M_{23}C_6$-type carbides. The images for analysis are taken from the as-polished creep tested specimens. The location at which the image was taken is within a 2 mm distance from the rupture interface for all the specimens. The particle sizes are counted using Image J® software package. The resolution of the metallurgical microscope is calculated using Rayleigh criterion to be 500 nm (0.5 µm): $d = \lambda/2NA$, where $d$ is particle size, $\lambda$ is wavelength of visible white light, $NA$ is the numerical aperture of the objective lens. By using optical images in the analysis, particles smaller than 0.5 µm are not counted. It is believed that the neglecting of smaller particles will not change the results or conclusion significantly.

The frequency distributions of the particle size as a function of heat treating temperature are shown in Figure 42a for specimens treated for 2 hours, and Figure 42b for specimens treated for 8 hours. The particles tend to be more in number and larger in size for specimens that have been PWHT-ed at a higher temperature. This trend is more pronounced in the specimens that have been treated at temperature for 8 hours. Because the coarsening of carbides will deplete the hardening carbon and chromium, the remaining ferrite matrix will be weaker for creep resistance.

4.4. Stress relaxation

To further understand creep behavior, and to provide model input data, a series of stress relaxation samples were tested. The major advantage of the stress relaxation test is that it is much faster when compared to conventional creep testing. However, the correlation between creep data obtained in stress relaxation tests and that of conventional creep tests is in question because stress relaxation tests do not insure the same amount of plastic deformation as conventional creep tests. Also, the stress relaxation test is based on a constant displacement while measuring the decrease in stress and conventional creep tests are performed under a constant stress condition while strain is measured [22]. It has not as yet been definitively verified whether the results of these two tests are equivalent.

Figure 43 shows the results for the samples subjected to stress relaxation tests. Similar to the trend seen with conventional creep testing, it can be seen that for a constant stress level, higher heat treating temperatures produce increased strain rates. In Table 7, the minimum creep rates
identified by conventional creep testing and stress relaxation testing of the same set of cross-welds are compared. The stress relaxation tests are conducted at 650 °C with 1.5 % total strain. The creep tests are conducted at 650 °C with 70 MPa stress. For the base material, both tests produced almost identical minimum creep rate. For the cross-weld specimens, the stress relaxation tests tend to produce smaller creep rates, with relative differences of 6.4 to 79 % compared with creep tested results. It is encouraging to see the stress relaxation tests are able to produce minimum creep rates with an error of not more than a 79 %. More tests should be conducted to understand and standardize the stress relaxation test procedure so that more consistent results can be obtained.

4.5. A model for cross-weld creep

Constitutive equation – The total creep deformation \( \delta l_{cw} \) in a cross-weld specimen is assumed to be a linear combination of contributions from all microstructural constituents:

\[
\delta l_{cw} = \delta l_{weld} + \delta l_{cghaz} + \delta l_{fghaz} + \delta l_{ichaz} + \delta l_{bm}
\]  

(4)

where

- \( \delta l_{weld} \) is deformation of the weld fusion zone,
- \( \delta l_{cghaz} \) is deformation of the CGHAZ,
- \( \delta l_{fghaz} \) is deformation of the FGHAZ,
- \( \delta l_{ichaz} \) is deformation of the ICHAZ,
- \( \delta l_{bm} \) is deformation of the base metal.

The creep strain in each constituent is calculated for the primary and secondary creep stages. For example the creep strain \( \varepsilon_{weld} \) at time \( t \) in the weld fusion zone can be obtained by integrating the creep strain rate \( \dot{\varepsilon}_{weld} \):

\[
\varepsilon_{weld} = \int_0^t \dot{\varepsilon}_{weld} = \int_0^{t_p} \dot{\varepsilon}_{weld_p} + \int_{t_p}^t \dot{\varepsilon}_{weld_s}
\]  

(5)

where \( t_p \) is time at the end of primary creep stage, \( \varepsilon_{weld_p} \) is the primary-stage creep strain rate of weld metal, and \( \varepsilon_{weld_s} \) is the creep strain rate of weld metal during the secondary creep stage, which is obtained from the stress relaxation test by fitting the data into the Norton equation

\[
\dot{\varepsilon}_{weld_s} = B \sigma^n,
\]

where \( \sigma \) is stress, \( B \) and \( n \) are material parameters.

Temperature-dependent material properties – Uniaxial tensile tests have been conducted on the Gleeble 1500D thermal-mechanical simulator to determine the temperature-dependent mechanical properties of Grade 91 steel weldments. Poisson’s ratio is assumed to be constant at 0.3. The young’s modulus tested at 650 °C for all the microstructural constituents are shown in Table 8. The primary creep equation has been obtained for the short term conventional creep tests while the secondary creep equation has been obtained from the stress relaxation tests. The result obtained from stress relaxation test for the CGHAZ, FGHAZ, ICHAZ, WM and BM are shown in Figure 44.

Creep model – Creep is an irreversible strain which is based on deviatoric behavior. The material is assumed to be incompressible under creep flow. Creep can be modeled using explicit (Euler forward) or implicit (backward integration). In explicit creep, the creep strain rate used at each
time step corresponds to the rate at the beginning of the time step and is assumed to be constant throughout that time step. Euler backward or implicit creep is suitable for most methods and is both more accurate and faster. This method is numerically unconditionally stable, which means it does not require a small time-step as does the explicit creep method.

The modified total strain tensor at time step, \( t \) is calculated using the creep strain tensor at the previous time step, \( (t-1) \), using the additive strain decomposition method [23]

\[
\varepsilon'_{\text{mod}} = \varepsilon_t - \varepsilon^{0} - \varepsilon^{h} - \varepsilon_{t-1} \quad (6)
\]

The equivalent modified total strain is then calculated using the modified total strain,

\[
\varepsilon_{\text{eq}}^{\text{mod,total}} = \frac{1}{\sqrt{2(1+\mu)}} \sqrt{\left(\varepsilon_{t,x}^{\text{mod}} - \varepsilon_{t,y}^{\text{mod}} - \varepsilon_{t,z}^{\text{mod}}\right)^2 + \left(\varepsilon_{t,y}^{\text{mod}} - \varepsilon_{t,z}^{\text{mod}}\right)^2 + \left(\varepsilon_{t,z}^{\text{mod}} - \varepsilon_{t,x}^{\text{mod}}\right)^2 + \frac{3}{2}(\varepsilon_{t,x,y}^{\text{mod}})^2 + \frac{3}{2}(\varepsilon_{t,x,z}^{\text{mod}})^2 + \frac{3}{2}(\varepsilon_{t,y,z}^{\text{mod}})^2} \quad (7)
\]

The equivalent stress is calculated as,

\[
\sigma_{\text{eq}} = E\varepsilon_{\text{eq}}^{\text{mod,total}} \quad (8)
\]

Then the equivalent creep strain increment is calculated using the appropriate creep law (Equation 5) and then converted to a full creep strain tensor as follows

\[
\begin{bmatrix}
\Delta \varepsilon_{t,x}^{c} \\
\Delta \varepsilon_{t,y}^{c} \\
\Delta \varepsilon_{t,z}^{c} \\
\Delta \varepsilon_{t,x,y}^{c} \\
\Delta \varepsilon_{t,x,z}^{c} \\
\Delta \varepsilon_{t,y,z}^{c}
\end{bmatrix} =
\frac{\Delta \varepsilon_{\text{eq}}^{\text{mod,total}}}{2(1+\mu)\varepsilon_{\text{eq}}^{\text{mod}}} \begin{bmatrix}
2 & -1 & -1 & 0 & 0 & 0 \\
-1 & 2 & -1 & 0 & 0 & 0 \\
-1 & -1 & 2 & 0 & 0 & 0 \\
0 & 0 & 0 & 3 & 0 & 0 \\
0 & 0 & 0 & 0 & 3 & 0 \\
0 & 0 & 0 & 0 & 0 & 3
\end{bmatrix} \quad (9)
\]

The elastic strain is then calculated using

\[
\varepsilon_{t,x}^{e} = \varepsilon_{t,x}^{\text{mod}} - \Delta \varepsilon_{t}^{c} \quad (10)
\]

And the creep strain is calculated using

\[
\varepsilon_{t,x}^{c} = \varepsilon_{t-1}^{c} + \Delta \varepsilon_{t}^{c} \quad (11)
\]

The geometry of the 3-dimensional finite element model is set up according to actual measurements of creep tested cross-weld specimen. Weld dimensions and thickness of heat-affected zone regions are determined metallographically. The SOLID185 element type in ANSYS is used. This element has 8 nodes with three degrees of freedom at each node. This element has creep, plasticity, stress stiffening, large deflection and large strain nonlinearity capabilities. It also has mixed formulation capability for simulating deformations of nearly incompressible elastoplastic and fully incompressible hyperelastic materials. The geometry was meshed with hexagonal mesh with 59,619 nodes and 53,120 elements (Figure 45). The finer mesh sizes were assigned to the heat-affected zones of the model.

**Model results** – Model predicted cross-weld creep strain is compared with the actual creep test results in Figure 46. Good agreement between the creep strains verifies the proposed primary
and secondary creep model. The model predicted creep strain distribution within the specimen is shown in Figure 47. The highest creep strain location is at the ICHAZ. Necking has also been predicted, and the wedge-shaped weld slips down parallel to the ICHAZ. These features of creep deformation for Type IV rupture have been observed in the specimens in this study. The developed model, along with the minimum creep strain rates identified by stress relaxation tests as the inputs, is capable of predicting cross-weld creep properties of a joint with unknown creep life. Because a stress relaxation test only requires a few days to finish, the proposed model has the potential to become an accelerated creep assessment method. Further validation of the model is therefore needed.

5. Conclusion

Through instrumented temperature acquisition and thermal modeling, a comprehensive understanding has been gained on how welding parameters affect the microstructure and properties. An FE model has been developed to model the thermal history during welding and heat treatment. Using the optimized welding procedure, a 5-inch thick Grade 91 test sample has been prepared. The heat-treated pipe and heavy section Grade 91 have been tested for creep rupture properties. Correlations between creep behavior and post-weld heat treatment of the joints have been established. A creep model for cross-weld has been developed that is able to predict Type IV rupture using short-time stress relaxation testing to identify input property data.

1. Type IV rupture in the fine-grain and inter-critical HAZ has been reproduced from creep testing at 650 °C under 70 MPa (10 KSI) stress. In the range of 600 to 800 °C, 2 and 8 hour PWHT, longer creep life is resulted from Grade 91 welds heat treated with a lower temperature and shorter time (600 °C, 2 hours). However, the room temperature toughness of the HAZ only recovers after a 720 °C, 2 hour PWHT. Ultimately, the results of this study indicate that the post-weld heat treatment of any Grade 91 steel should be conducted by first calculating the A1 temperatures of all involved materials, including the filler metals and base metals. The upper bound temperature for PWHT should not be higher than the lowest A1 temperature of all materials. The lower bound temperature for PWHT should be sufficient for restoring toughness to prevent cold cracking. For the alloys tested in this study, the recommended PWHT parameters would be higher than 720°C and no more than 770°C for at least 2 hours.

2. Following a PWHT at temperatures above the 820 °C (A1 of test material), the creep strain rate and creep ductility significantly increase, the rupture life is shorter, and the rupture location switches to the weld fusion zone, resulting in Type I failure.

3. PWHT changes the resultant grain size and degree of tempering of martensite, or leads to formation of new martensite, if the heating temperature is above A1 critical temperature. The number and sizes of carbide precipitates are proportional to the PWHT temperature and time. The precipitation and growth of carbide particles and fine-grained ferrite are believed to be a microstructure with decreased creep resistance.

4. Based on the insignificant difference between strain rates of the conventional creep samples heat treated at 760°C for 2 and 8 hours, it appears that there may not be any significant negative effect realized from increasing heat treating time. This would suggest that for thick section post weld heat treatment, an increased time might be allowable to ensure that the material furthest from the heat source reaches at least 720°C for 2 hours without the
risk of damaging the material that is closest to the heat source. However, further testing would be necessary to conclude whether times beyond 8 hours could be detrimental to creep behavior.

5. The minimum creep rates obtained from stress relaxation testing agree with the minimum creep rates obtained from conventional stress rupture testing. The relaxation tests correctly rank the creep rates of various specimens, and confirm that lower heat treating temperatures could produce reduced creep strain rates when compared to heat treating at higher temperatures.

6. The proposed cross-weld creep model is capable of predicting primary and secondary creep strains based on short-time test data obtained from stress relaxation tests. The model is able to correctly predict the creep strain distribution within the specimen and Type IV rupture mode.

6. Project Deliverables

6.1. Reports and publications

The optimum welding procedure has been identified, and the effect of post-weld heat treatment on joint microstructure and creep performance has been investigated. In addition to this final report, an annual report and numerous quarterly reports were submitted:


Two journal papers that summarize the research findings are being prepared for submission to technical journals. The following technical papers have been presented or published:


6.2. Students and educational activities

This project helped enhance engineering education of graduate students in Prof. Li’s research lab through nuclear engineering research. Over a three-year period, there have been five graduate students who have been fully or partially supported by and working on this project:

1. Andrew Deceuster (PhD candidate in Biological Engineering)
2. Sam (Chunbo) Zhang (PhD candidate in Mechanical Engineering)
3. Benjamin Griffiths (MS graduate student in Mechanical Engineering)
4. Jacob Walker (MS graduate student in Mechanical Engineering)
5. Bishal Silwal (PhD candidate in Mechanical Engineering)

An undergraduate research assistant, Jess Housekeeper, has also been partially supported by the project, and has participated in research. These students worked together on the project, but Andrew, Benjamin, and Jacob mainly conducted the experimental studies; Sam and Bishal mainly conducted numerical studies. During the third year of the project, Sam, Andrew, and Benjamin had graduated and moved on to engineering positions. Bishal Silwal and Jacob Walker are finishing up their theses that are based on the project research.

7. Acknowledgment

This research is being performed using funding received from the DOE Office of Nuclear Energy’s Nuclear Energy University Programs. We would like to thank Dr. Richard Wright, the technical point of contact from the DOE, for his advising and discussions during the entire project period. We also would like to thank Euroweld for donating some of the filler materials, and the Welding Research Council (WRC) and its technical director, Dr. Martin Prager, for technical discussions on creep and creep testing of welds.
8. References


Table 1: Chemical composition (wt.%) of base metals and filler metals. The as-received pipe material was treated at 1050 °C × 8 minute normalizing, and followed by 785 °C × 45 minute tempering. The as-received block materials was forged and treated at 1050 °C × 3.5 hour normalizing, and followed by 780 °C × 3.5 hour tempering.

<table>
<thead>
<tr>
<th></th>
<th>ER90S-B9 GTAW</th>
<th>E91T1-B9 FCAW</th>
<th>Grade 91 Pipe</th>
<th>Grade 91 Block</th>
</tr>
</thead>
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<tr>
<td>C</td>
<td>0.097</td>
<td>0.10</td>
<td>0.11</td>
<td>0.086</td>
</tr>
<tr>
<td>Cr</td>
<td>8.83</td>
<td>9.10</td>
<td>8.47</td>
<td>8.89</td>
</tr>
<tr>
<td>Mo</td>
<td>0.928</td>
<td>0.88</td>
<td>0.94</td>
<td>0.87</td>
</tr>
<tr>
<td>Mn</td>
<td>0.56</td>
<td>0.79</td>
<td>0.37</td>
<td>0.39</td>
</tr>
<tr>
<td>Si</td>
<td>0.25</td>
<td>0.28</td>
<td>0.37</td>
<td>0.33</td>
</tr>
<tr>
<td>Ni</td>
<td>0.307</td>
<td>0.55</td>
<td>0.08</td>
<td>0.30</td>
</tr>
<tr>
<td>Al</td>
<td>0.002</td>
<td>0.001</td>
<td>0.002</td>
<td>0.003</td>
</tr>
<tr>
<td>V</td>
<td>0.197</td>
<td>0.20</td>
<td>0.19</td>
<td>0.22</td>
</tr>
<tr>
<td>Nb</td>
<td>0.064</td>
<td>0.03</td>
<td>0.071</td>
<td>0.096</td>
</tr>
<tr>
<td>S</td>
<td>0.004</td>
<td>0.008</td>
<td>0.002</td>
<td>0.001</td>
</tr>
<tr>
<td>P</td>
<td>0.006</td>
<td>0.02</td>
<td>0.016</td>
<td>0.020</td>
</tr>
<tr>
<td>N</td>
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<td>0.05</td>
<td>0.048</td>
<td>0.034</td>
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<td></td>
<td></td>
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<tr>
<td>Ti</td>
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<td></td>
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<tr>
<td>Cu</td>
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<td>0.04</td>
<td></td>
<td></td>
</tr>
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<td>As</td>
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<td>0.002</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sn</td>
<td>0.003</td>
<td>0.008</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sb</td>
<td>0.003</td>
<td>0.002</td>
<td></td>
<td></td>
</tr>
<tr>
<td>B</td>
<td>0.0007</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Zr</td>
<td>0.001</td>
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<td></td>
</tr>
<tr>
<td>Co</td>
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<td></td>
<td></td>
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<tr>
<td>Ca</td>
<td>0.003</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ta</td>
<td>0.001</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>W</td>
<td>0.005</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>H</td>
<td>3 ppm</td>
<td></td>
<td></td>
<td></td>
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Figure 1: Pipe welding setup, showing the fixture, back shielding, and thermocouples.
Figure 2: Welding setup for the forged heavy section Grade 91 (a), and a welded specimen (b).
<table>
<thead>
<tr>
<th>Weld Test</th>
<th>Preheat (°C)</th>
<th>Inter-pass (°C)</th>
<th>Heat input</th>
<th>HAZ Width (mm)</th>
<th>Charpy As-Welded (J)</th>
<th>Charpy Heat Treated (J)</th>
<th>FZ Boundary As-Welded (HV)</th>
<th>FZ Boundary Heat Treated (HV)</th>
<th>ICHAZ As-Welded (HV)</th>
<th>ICHAZ Heat Treated (HV)</th>
</tr>
</thead>
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<tr>
<td>1</td>
<td>250</td>
<td>300</td>
<td>0.9986kJ/mm (WFS 7.62mm/min @ 27V)</td>
<td>2.69</td>
<td>186</td>
<td>209</td>
<td>419</td>
<td>236</td>
<td>274</td>
<td>209</td>
</tr>
<tr>
<td>2</td>
<td>250</td>
<td>300</td>
<td>0.8655kJ/mm (WFS 6.36mm/min @ 20V)</td>
<td>2.43</td>
<td>208</td>
<td>267</td>
<td>460</td>
<td>240</td>
<td>301</td>
<td>208</td>
</tr>
<tr>
<td>3</td>
<td>250</td>
<td>200</td>
<td>0.9986kJ/mm (WFS 7.62mm/min @ 27V)</td>
<td>2.47</td>
<td>175</td>
<td>244</td>
<td>482</td>
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<td>316</td>
<td>211</td>
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<tr>
<td>4</td>
<td>250</td>
<td>200</td>
<td>0.8655kJ/mm (WFS 6.36mm/min @ 20V)</td>
<td>2.24</td>
<td>196</td>
<td>216</td>
<td>454</td>
<td>264</td>
<td>213</td>
<td>219</td>
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<tr>
<td>5</td>
<td>150</td>
<td>300</td>
<td>0.9986kJ/mm (WFS 7.62mm/min @ 27V)</td>
<td>2.53</td>
<td>224</td>
<td>241</td>
<td>435</td>
<td>232</td>
<td>223</td>
<td>222</td>
</tr>
<tr>
<td>6</td>
<td>150</td>
<td>300</td>
<td>0.8655kJ/mm (WFS 6.36mm/min @ 20V)</td>
<td>2.38</td>
<td>214</td>
<td>249</td>
<td>446</td>
<td>233</td>
<td>263</td>
<td>203</td>
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<tr>
<td>7</td>
<td>150</td>
<td>200</td>
<td>0.9986kJ/mm (WFS 7.62mm/min @ 27V)</td>
<td>2.4</td>
<td>182</td>
<td>264</td>
<td>450</td>
<td>239</td>
<td>232</td>
<td>205</td>
</tr>
<tr>
<td>8</td>
<td>150</td>
<td>200</td>
<td>0.8655kJ/mm (WFS 6.36mm/min @ 20V)</td>
<td>2.22</td>
<td>177</td>
<td>240</td>
<td>511</td>
<td>233</td>
<td>307</td>
<td>232</td>
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Figure 3: The welding test matrix, and results on HAZ width and mechanical properties.
Figure 4: Schematic of Charpy coupon extraction, and the location of the notch relative to the weld HAZ.

Figure 5: The cross-weld creep coupon. The weld is located in the middle section of the gage length.
Figure 6: Matrix of ductility (a) and plot of ductility range (b) for various heat treating temperatures and times.
Figure 7: Hardness profiles of the as-welded and heat treated samples for every welding condition.
Figure 8: (continued) Hardness profiles of the as-welded and heat treated samples for every welding condition.
Figure 9: Charpy impact test results from specimens in the as-welded condition for different welding parameters.
Figure 10: Fracture path of an example of Charpy impact-tested, as-welded specimen. The fracture originated from the notch (in the lower left corner), passed through the weld metal, heat-affected zone (HAZ), and base metal.
Figure 11: Charpy impact test results for joints heat-treated at 760 °C for 2 hours. The joints were made using different welding parameters.
Figure 12: The as-welded microstructure of the coarse-grained heat-affected zone (CGHAZ) from weld Test #5 (a), and the corresponding simulated multi-pass CGHAZ microstructure (b). Nital etching.
Figure 13: Charpy impact energy results of different HAZ zones after post-weld heat-treating at various temperatures for 2 hours.
Table 2: Charpy impact test results for various zones of the as-welded and heat-treated joints.

<table>
<thead>
<tr>
<th>Condition</th>
<th>As-welded (J)</th>
<th>PWHT: 760C-2H (J)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base Metal (BM)</td>
<td>229</td>
<td>242</td>
</tr>
<tr>
<td>Weld Metal (WM)</td>
<td>7</td>
<td>56</td>
</tr>
<tr>
<td>CGHAZ</td>
<td>10</td>
<td>184</td>
</tr>
<tr>
<td>FGHAZ</td>
<td>75</td>
<td>246</td>
</tr>
<tr>
<td>ICHAZ</td>
<td>180</td>
<td>239</td>
</tr>
</tbody>
</table>

Table 3: Sample calculation of contributions of base metal and HAZ to the total impact energy.

<table>
<thead>
<tr>
<th>As-welded (J)</th>
<th>PWHT: 760C-2H (J)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$L_{CGHAZ}$ (mm)</td>
<td>0.5</td>
</tr>
<tr>
<td>$L_{FGHAZ}$ (mm)</td>
<td>0.7</td>
</tr>
<tr>
<td>$L_{ICHAZ}$ (mm)</td>
<td>0.3</td>
</tr>
<tr>
<td>$L_{BM}$ (mm)</td>
<td>6.0</td>
</tr>
<tr>
<td>$E_{CGHAZ}$ (J)</td>
<td>0.6</td>
</tr>
<tr>
<td>$E_{FGHAZ}$ (J)</td>
<td>9</td>
</tr>
<tr>
<td>$E_{ICHAZ}$ (J)</td>
<td>6</td>
</tr>
<tr>
<td>$E_{BM}$ (J)</td>
<td>203</td>
</tr>
<tr>
<td>$E_{Calc}$ (J)</td>
<td>220</td>
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<tr>
<td>$E_{Exp}$ (J)</td>
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<tr>
<td>Difference (%)</td>
<td>-5</td>
</tr>
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</table>

Figure 14: Typical temperature curve obtained from pipe section welding.
Figure 15: Measured surface temperature data for 9” × 5” × 5” thick-plate section post-weld heat treating procedure (a), and FEM predicted temperature change in the center of the 5 inch section (b).
Figure 16: Width of the HAZ as affected by welding parameters.
Figure 17: Thermocouple recordings at various locations during the first FCAW pass on top of the GTAW root pass.
Figure 18: Cross-weld creep strain (a) and creep strain rate (b) as a function of time for Grade 91 pipe welds of various post-weld heat treating conditions.
Table 4: Creep test results for specimens extracted from welded 1” pipe.

<table>
<thead>
<tr>
<th>T (°C)</th>
<th>PWHT time (h)</th>
<th>Rup. time, t&lt;sub&gt;R&lt;/sub&gt; (h)</th>
<th>ε&lt;sub&gt;min&lt;/sub&gt; (h&lt;sup&gt;−&lt;/sup&gt;)</th>
<th>ε&lt;sub&gt;R&lt;/sub&gt; (%)</th>
<th>ε&lt;sub&gt;avg&lt;/sub&gt; (h&lt;sup&gt;-&lt;/sup&gt;)</th>
<th>Rup. location</th>
</tr>
</thead>
<tbody>
<tr>
<td>600</td>
<td>2</td>
<td>942</td>
<td>6.8 E-4</td>
<td>1.5</td>
<td>1.6 E-3</td>
<td>ICHAZ</td>
</tr>
<tr>
<td>600</td>
<td>8</td>
<td>925</td>
<td>6.8 E-4</td>
<td>1.0</td>
<td>1.1 E-3</td>
<td>ICHAZ</td>
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<tr>
<td>760</td>
<td>2</td>
<td>649</td>
<td>1.3 E-3</td>
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<td>1.8 E-3</td>
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<td>1.3 E-3</td>
<td>1.1</td>
<td>1.9 E-3</td>
<td>ICHAZ</td>
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<tr>
<td>820</td>
<td>2</td>
<td>760</td>
<td>3.0 E-3</td>
<td>3.4</td>
<td>4.5 E-3</td>
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<tr>
<td>820</td>
<td>8</td>
<td>617</td>
<td>5.0 E-3</td>
<td>7.0</td>
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<td>Fusion zone</td>
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<tr>
<td>840</td>
<td>2</td>
<td>388</td>
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<td>15.4</td>
<td>4.0 E-2</td>
<td>Fusion zone</td>
</tr>
<tr>
<td>840</td>
<td>8</td>
<td>205</td>
<td>2.6 E-2</td>
<td>14.3</td>
<td>7.0 E-2</td>
<td>Fusion zone</td>
</tr>
<tr>
<td>Pipe BM*</td>
<td>-</td>
<td>2402</td>
<td>3.0 E-4</td>
<td>1.0</td>
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</tr>
</tbody>
</table>

<sup>*Terminated</sup>

Table 5: Creep test results for specimens extracted from welded 5” block

<table>
<thead>
<tr>
<th>T (°C)</th>
<th>PWHT time (h)</th>
<th>Rup. time, t&lt;sub&gt;R&lt;/sub&gt; (h)</th>
<th>ε&lt;sub&gt;min&lt;/sub&gt; (h&lt;sup&gt;−&lt;/sup&gt;)</th>
<th>ε&lt;sub&gt;R&lt;/sub&gt; (%)</th>
<th>Rup. location</th>
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<tr>
<td>650°</td>
<td>5</td>
<td>1600</td>
<td>1.5 E-4</td>
<td>0.40</td>
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<tr>
<td>740°</td>
<td>5</td>
<td>1599</td>
<td>1.5 E-4</td>
<td>0.36</td>
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</tr>
<tr>
<td>760°</td>
<td>5</td>
<td>1601</td>
<td>3.8 E-4</td>
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</tr>
<tr>
<td>800</td>
<td>5</td>
<td>1127</td>
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<td>5.70</td>
<td>Fusion zone</td>
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<td>Block BM*</td>
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<td>2402</td>
<td>1.0 E-4</td>
<td>0.31</td>
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</table>

<sup>*Terminated</sup>

Table 6: Calculated and measured transformation temperatures (°C) of test materials.

<table>
<thead>
<tr>
<th>Material</th>
<th>Calculated A&lt;sub&gt;1&lt;/sub&gt;</th>
<th>Measured A&lt;sub&gt;C1&lt;/sub&gt;</th>
<th>Measured A&lt;sub&gt;C3&lt;/sub&gt;</th>
<th>Measured M&lt;sub&gt;s&lt;/sub&gt;</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pipe Grade 91</td>
<td>824</td>
<td>826</td>
<td>928</td>
<td>398</td>
</tr>
<tr>
<td>Block Grade 91</td>
<td>811</td>
<td>818</td>
<td>924</td>
<td>405</td>
</tr>
<tr>
<td>FCAW filler wire</td>
<td>771</td>
<td>728</td>
<td>924</td>
<td>389</td>
</tr>
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</table>
Figure 19: Cross-weld creep strain (a) and creep strain rate (b) as a function of time for Grade 91 five-inch block welds of various post-weld heat treating conditions.
Figure 20: Schematic showing how the weld bead geometry was translated into a realistic FEM model.
The simulation results match the experimental measurements on the surface. Simulation predicts thermal histories at internal points, serving to correlate microstructure with thermal history.

Figure 21: FEM model prediction of the thermal cycles for various HAZ locations.
Figure 22: FEM model prediction of the temperature field in an one-inch thick specimen heat-treated at 760 °C for 2160 seconds.
Figure 23: Plot of dilation as a function of temperature for the pipe base metal (a), thick section base metal (b), and weld metal (c).
Figure 24: XRD spectrum of the as-received base metal Grade 91 (a), and magnified lower 2θ angle portion showing an $M_23C_6$ carbide peak (b). XRD spectrum of a specimen air-cooled from 840 °C, showing broadening of peaks, indicated by the arrows, due to fresh martensite (c).
Figure 25: Macrostructure of the as-welded 1 inch-thick pipe joint (a) and 5 inch thick block joint (b).
Figure 26: The untempered martensite in the as-welded fusion zone etched as bright white by the LePera etchant. The tan colored region is ferrite, and mark-etching region is tempered martensite.
Figure 27: Microstructure of the HAZ in a typical as-welded pipe joint.
Increase the temperature:

Temper, Dissolution, Grain growth, and New martensite above $A_1 = 771{\degree}C$

Figure 28: Fusion zone microstructure change as a function of PWHT temperature.
Figure 29: The untempered martensite in the 840 °C - 8 hour treated fusion zone etched as bright white by the LePera etchant. The tan colored region is ferrite, and mark-etching region is tempered martensite.
Figure 30: Microstructure of the HAZ in a 760 °C 2 hour post-weld heat treated joint.
Figure 31: Average Vickers hardness values of different HAZ zones after post-weld heat-treating at various temperatures for 2 hours.

Figure 32: The microstructure of ICHAZ heat treated at 840 °C for 2 hours, and air cooled. Etched with Le Pera reagent, the microstructure constituents include white-etching martensite (M), tan-etching ferrite (F), and dark-etching tempered martensite (TM).
Figure 33: Microstructure of coarse-grained heat-affected zone (CGHAZ) after heat-treatment at 640 °C (a), 800 °C (b), and 840 °C (c) for 2 hours. Nital etching.
Figure 34: Microstructure of fine-grained heat-affected zone (FGHAZ) in the as-welded (a), after heat-treatment at 640 (b), 800 (c), and 840 °C (d) for 2 hours. Nital etching.
Figure 35: Microstructure of inter-critical heat-affected zone (ICHAZ) in the as-welded (a), after heat-treatment at 640 (a), 800 (b), and 840 °C (c) for 2 hours. Nital etching.
Figure 36: (a) A creep tested specimen that failed in the creep damage zone in the FGHAZ and ICHAZ, or Type IV failure; (b) another example of creep damage zone between the weld HAZ on the left and base metal on the right; (c) cross-sectional microstructure of the specimen showing the concentrated creep damaged zone (CDZ) sandwiched between the base metal (BM) and heat-affected zone (HAZ).
Figure 37: Cross-sectional view of the rupture shown in the right of Figure 36a.
Figure 38: Cross-sectional view of the creep damaged zone shown in the left of Figure 36a.
Figure 39: Microstructure of the creep damaged zone shown in Figure 38.
Figure 40: Hardness traverses of creep tested specimens that post-weld treated at different temperatures for 2 hours (a), and Hardness traverses of creep tested specimens that post-weld treated at different temperatures for 8 hours (b).
Figure 41: The carbide particles in the as-received base metal pipe material (a), and an example image from a creep tested specimen for carbide measurements (b).
Figure 42: Carbide particle size distribution in creep tested specimens as a function of PWHT temperature for 2 hours (a), and carbide particle size distribution in creep tested specimens as a function of PWHT temperature for 8 hours (b).
Figure 43: Plot of stress relaxation test results showing strain rate as a function of stress.

Table 7: Comparison of minimum creep rate data from conventional creep testing and stress relaxation testing at 650 °C. The cross-weld specimens have the same treatment histories.

<table>
<thead>
<tr>
<th>Material</th>
<th>Creep $\dot{\epsilon}_{\text{min}}$ (1/sec)</th>
<th>Relaxation $\dot{\epsilon}_{\text{min}}$ (1/sec)</th>
<th>Relative Difference (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base metal</td>
<td>8.33E-10</td>
<td>8.79E-10</td>
<td>-5.5</td>
</tr>
<tr>
<td>760C-2H</td>
<td>3.61E-09</td>
<td>1.50E-09</td>
<td>58.5</td>
</tr>
<tr>
<td>820C-2H</td>
<td>8.33E-09</td>
<td>5.10E-09</td>
<td>40.0</td>
</tr>
<tr>
<td>820C-8H</td>
<td>1.39E-08</td>
<td>1.30E-08</td>
<td>6.4</td>
</tr>
<tr>
<td>840C-2H</td>
<td>4.17E-08</td>
<td>5.50E-08</td>
<td>-32.0</td>
</tr>
<tr>
<td>840-8H</td>
<td>7.22E-08</td>
<td>1.50E-08</td>
<td>79.0</td>
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</table>
Figure 44: Stress vs. strain rate results from stress relaxation testing at 650 °C of base metal and various weld joint regions that have been heat-treated at 760 °C for 2 hours.
Table 8: Young’s modulus of various weld regions following a 760°C-2H PWHT. Values were measured from high-temperature tensile tests at 650 °C.

<table>
<thead>
<tr>
<th>Constituents</th>
<th>Young’s Modulus (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CGHAZ</td>
<td>26,615</td>
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<tr>
<td>FGHAZ</td>
<td>25,394</td>
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<tr>
<td>IGHAZ</td>
<td>9,491</td>
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<tr>
<td>Weld Metal</td>
<td>7,040</td>
</tr>
<tr>
<td>Base Metal</td>
<td>89,643</td>
</tr>
</tbody>
</table>
Figure 46: The result comparison

Figure 47: Creep Strain