Development of Novel Functionally Graded Transition Joints for Improving the Creep Strength of Dissimilar Metal Welds in Nuclear Applications

Reactor Concepts Research Development and Demonstration (RCRD&D)

John DuPont
Lehigh University

Collaborators
University of Tennessee, Knoxville

Sue Lesica, Federal POC
Richard Wright, Technical POC
Development of Novel Functionally Graded Transition Joints for Improving the Creep Strength of Dissimilar Metal Welds in Nuclear Applications

Award No. DE-NE0008278

Prof. John N. DuPont
Principal Investigator
Lehigh University
Bethlehem, PA

Prof. Suresh Babu
University of Tennessee, Knoxville
Knoxville, TN

Dr. Zhili Feng
Oak Ridge National Laboratory
Oak Ridge, TN

June 29, 2018
Executive Summary

Steam generators of the small modular Very High Temperature Reactor (VHTR) will require use of Alloy 800 and 2.25Cr-1Mo steel in different regions depending on temperature and corrosion resistance requirements. As a result, Dissimilar Metal Welds (DMWs) between these two materials are critical for design, development, and manufacturing of the VHTR. DMWs are also used in many other applications in the power generation and petrochemical industries. Experience has demonstrated that failures of such DMWs can occur prematurely well below the expected creep life of either base metal, and these failures have significant consequences in terms of plant reliability, safety, and economic impact. These DMWs failures are caused by the abrupt change in composition that occurs at the weld interface between the two alloys.

In this research, the concept of developing and optimizing Graded Transition Joints (GTJ) was developed through an integrated modeling and experimental approach. With GTJs, the composition is gradually graded between the two alloys, thus eliminating the sharp change in composition that causes failure. Microstructural modeling was first conducted to identify the optimum grade length required for improved resistance to carbon diffusion that is largely responsible for premature failure. Next, GTJs were fabricated and characterized in detail to understand how the microstructure evolves during fabrication and long term aging. Finally, the creep behavior of the GTJs was investigated and compared to that of conventional DMWs to demonstrate the superior creep strength of the GTJs.

The results of this research provide significant insight into design, fabrication, microstructural evolution, and resultant performance of GTJs that can be used for reducing or eliminating DMW failures in a number of high temperature applications. The results also demonstrate that the GTJs can be readily fabricated with either conventional or additive manufacturing processes, and detailed guidelines are provided on the length of grades needed for optimal performance. The results of this research benefits the general public by improving the reliability of power generation and petrochemical plants that rely on DMWs for safe and reliable operation to deliver power and other products.
Comparison Expected and Actual and Project Deliverables:

1. A detailed methodology for producing GTJs that avoid premature creep failure of joints made between 2.25Cr-1Mo steel and alloy 800. The methodology will include details on the optimized concentration gradient required for the GTJs along with a demonstrated set of process conditions that can be used to fabricate the GTJs.

   *This objective was fully realized and is explained in detail in Sections 1 and 2 of this final report.*

2. Refined methods for measuring and modeling the localized creep strains that occur under the microstructural gradients within DMWs and GTJs.

   *A new method for measuring the localized creep strains was fully realized and is explained in detail in Section 3. There was a slight delay in developing this technique due to the complexity of the experimental set up. As a result, the modeling work is still in progress and will continue to be developed and described in peer reviewed publications.*

3. A database on the creep performance of GTJs that will pave the way for eventual ASME code acceptance.

   *This objective was realized and is explained in detail in Section 3 of this final report.*

4. Initiation of discussions with ASME for adoption of GTJs into dissimilar joints in the VHTR application.

   *This objective has begun and, as expected, will require additional time and testing.*

5. Preparation of progress reports and a final report and publications that summarize these advances.

   *All progress reports have been provided. Two manuscripts (based on Sections 1 and 2) have been submitted for publication in Metallurgical and Materials Transactions A. At least one more peer reviewed publication is expected from Section 3.*

**Summary of Project Activities**

All the project activities are described in detail in Sections 1 through 3.
Products Developed

Peer Review Manuscripts Submitted for Publication


Peer Review Manuscripts in Preparation

- Mohan Subramanian, Suresh Sudarsanam, Jonathan P. Galler, and John N. DuPont, Study of heterogeneous creep deformation in Dissimilar Metal Welds and Graded Transition Joints.

Conference proceedings and Presentations

- J. Galler and J. DuPont, Development of Novel Functionally Graded Transition Joints for Improving the Creep Strength of Dissimilar Metal Welds in Nuclear Applications, AWS Annual Conference, Chicago, IL, November 9-12, 2015, American Welding Society, Miami, FL.
- Mohan Subramanian, Suresh Sudarsanam, Jonathan P. Galler, and John N. DuPont, Study of high temperature deformation behavior of Graded Transition Joints (GTJs), TMS annual conference 2017, San Diego, CA
- Mohan Subramanian, Suresh Sudarsanam, Jonathan P. Galler, and John N. DuPont, Study of high temperature deformation behavior of Dissimilar Metal Welds (DMWs)- Baseline work to evaluate the performance of Graded Transition Joints (GTJs), AWS- FABTECH annual conference 2017, Chicago, IL
- Mohan Subramanian, Suresh Sudarsanam, Jonathan P. Galler, and John N. DuPont, Rationalization of heterogeneous creep deformation behavior of Dissimilar Metal Welds (DMWs), TMS annual conference 2018, Phoenix, AZ

Techniques

- A novel method for conducting Digital Image Correlation (DIC) during creep testing to identify strain localization.
1. Design of Graded Transition Joints through Thermodynamic and Kinetic Modeling

Abstract

Dissimilar metal welds between ferritic and austenitic alloys are common in high temperature applications in the energy industry. Under service conditions, these welds experience carbon diffusion and concomitant detrimental microstructural changes that can render them susceptible to premature creep failure. Therefore, it is helpful to identify a filler metal and joint design to reduce the driving force of carbon diffusion and prolong the expected service life. In the current investigation, the use of thermodynamic modeling was used to identify candidate alloys that reduce the chemical potential gradient, which is the driving force for carbon diffusion. Additionally, diffusion modeling was used to determine an optimal grade length for a graded transition joint to further reduce the extent of carbon diffusion. A graded transition joint was fabricated and aged to understand the microstructural evolution, and the results were used for direct comparison with the observed trends from the model simulations. It was determined that increasing the grade length to the millimeter length scale reduced the extent of carbon diffusion by an order of magnitude when compared to a conventional dissimilar metal weld with a concentration gradient on the length scale of ~10 – 100 microns. Additionally, a step function composition gradient was implemented into the diffusion calculations to simulate the more realistic gradients produced with additive manufacturing processes. In these stepped gradients, enhanced carbon diffusion occurred at the layer interfaces, indicating important regions that undergo microstructural evolution. The experimentally observed carbide distribution in the characterized graded transition joints is in good agreement with the calculated carbide distribution. Finally, the formation of a nickel-rich martensitic constituent was observed in the graded region after aging. This constituent was originally austenite at the aging temperature, and transformed to martensite upon cooling with no change in substitutional alloying element concentrations. The formation of this constituent was also accurately captured in the model results.
Introduction

Dissimilar metal welds (DMWs) are commonly used in energy applications where varying material behavior is necessary due to the high temperature operating conditions. DMWs are typically produced by joining a ferritic [body centered cubic matrix (bcc)] steel such as 2.25Cr1Mo (Grade 22) to alloy 800H with an austenitic [face centered cubic (FCC)] matrix using a nickel-base filler metal. The ferritic steel is used in lower temperature, less corrosive environments, whereas the austenitic alloy is used in higher temperature regions where enhanced creep strength and corrosion resistance are required. In this application, the alloys are usually joined using standard fusion welding processes. This produces very abrupt and uncontrolled gradients in composition, microstructure, and resultant thermal and mechanical properties.\(^1\,^2\) These sharp gradients lead to premature failure in the ferritic steel near the weld interface, which is a primary concern of the power generation industry. These failures can result in forced plant outages that may cost a power company up to $850,000 per day in lost revenue and repair costs.\(^3\)

It is important to have a thorough understanding of the failure mechanism of these DMWs to design an alternative solution to prevent failure. A large body of work has been conducted to study the mechanisms that lead to premature failure\(^1\,^4\)–\(^8\), and the failure mechanism has recently been reviewed in detail.\(^2\) The failures can be attributed to three major factors. The first is the difference in carbon concentration between the two materials, and the microstructural evolution due to carbon diffusion during aging. This promotes sharp changes in microstructure and concomitant mechanical properties along the fusion line. The second factor is the formation of carbides along the fusion line in the heat affected zone (HAZ) of the ferritic material due to carbon migration during service. A typical DMW failure is initiated from creep voids that form at the carbide-matrix interface.\(^6\,^9\) Therefore, the presence of these carbides creates a microstructure that is susceptible to creep failure. Finally, the coefficient of thermal expansion (CTE) mismatch between the ferritic and austenitic materials can cause strain localization along the weld interface. This, combined with the previously mentioned issues, exacerbates the susceptibility to failure.

It is well known that concentration gradients defines the diffusion path in a binary alloy system, as an element will typically diffuse down the gradient from high to low concentrations.\(^10\,^11\) However, tertiary and higher order additions of alloying elements in real, multi-component materials will create a chemical potential gradient that is relieved by the diffusion of elements in the system. Studies have demonstrated the presence of additional elements can create a chemical potential gradient that may be opposite to the concentration gradients, which results in “uphill” diffusion.\(^5\,^10\,^12\)–\(^14\) In a DMW, the chemical potential of carbon (CPC) gradient in a system level generally causes carbon to diffuse from the ferritic material to the austenitic material. This results in a carbon depleted region in the ferritic material, and a carbon enriched region in the partially mixed zone (PMZ). The differences in the chemical potential in ferrite matrix region is usually reflected in the amount and type of carbides that are in equilibrium with the ferrite. This microstructural evolution (along with CTE mismatch) leads to strain localization near the interface, and contributes to premature failure. Thus, the primary driving force for carbon diffusion in a multicomponent system is the chemical potential difference and resulting CPC gradient between two materials. The diffusion of carbon over time will eventually cease when the chemical potential difference is relieved in the system.\(^11\)
It has been reported that the chromium (Cr) content has the largest effect on the CPC. Brentrup and DuPont varied the Cr content in a Grade 22-800H DMW system to determine the effect on CPC gradient and the concomitant driving force for carbon diffusion. The larger Cr content in the austenitic material resulted in a steeper CPC gradient, and therefore, a stronger driving force for carbon diffusion. Thus, a reduction in Cr content in the filler metal can reduce the driving force for carbon diffusion. However, the need to further reduce the inevitability of carbon diffusion is necessary, and an alternate method is presented below.

In the current study, the design and fabrication of a graded transition joint (GTJ) was investigated, as it poses a new method to mitigate the carbon diffusion problem. A GTJ exhibits a gradual change in composition between the two materials over larger distances than a DMW. This reduces the sharp changes in microstructure and corresponding thermal and mechanical properties, and reduces the mismatch of CTE that is known to cause strain localization.

Brentrup and DuPont modeled the carbon diffusion of a DMW and GTJ between Grade 22 and Alloy 800H. As expected, a longer grade length significantly reduced the concentration gradient and associated CPC gradient and thus, the extent of carbon diffusion. Transition joints were also investigated by Sridharan et. al. using a powder blown additive manufacturing process to join Grade 22 to 316L using SS410 filler. This study attempted to reduce solidification cracking susceptibility of the common filler metal Inconel 82 during fabrication by grading 316L with an austenitic filler (SS410). While they successfully fabricated and characterized the GTJ that did not contain solidification cracks, there is still a need to understand the microstructural evolution and concomitant carbon migration that occurs during aging at high temperatures.

The primary objective of the current investigation is to understand the extent of carbon diffusion expected among different austenitic filler metals and Grade 22 steel, and analyze the microstructural evolution expected to occur during service conditions. The results from this study form a basis for optimizing GTJs to reduce the extent of carbon diffusion and the detrimental microstructures susceptible to premature failure. Based on the detailed modeling study, optimal grade lengths to minimize carbon diffusion are identified and the details of carbon diffusion behavior is discussed. Additionally, the model can be used as a predictive tool to calculate the extent of carbon diffusion for a given alloy system.

**Modeling and Experimental Procedure**

The allowable composition ranges of the base metal and filler metals investigated in this work are given in Table 1. A wide range of commercially available alloys were initially chosen for this study, as these alloys have been approved by pertinent codes and previously used for energy applications. The alloys selected have been welded directly to ferritic materials in many power generation applications, and are known to have good high temperature creep properties.

Initial thermodynamic calculations were conducted using ThermoCalc® software to isolate the effect individual elements had on the CPC of the system. The TCFE7 and NI-DATA-V7 databases were used for high iron (Fe) materials and nickel (Ni) base alloys, respectively. Individual elements were varied while simultaneously changing the Ni content (to account for the change), and the remaining composition stayed constant to
identify the individual effect an element had on the CPC. The chemical potential in the ferrite region is calculated at a system level stipulated by ferrite that is in equilibrium with carbide for a given local composition in a given location within the transition joint. Concentrations were selected to induce the largest CPC gradients that produce the largest extent of carbon diffusion. A revised set of compositions that were used for the simulations is shown in Table 2.

In both DMWs and GTJs, the CPC will vary within the PMZ that represents the region where partial mixing occurs between the base metal and filler metal. The incremental change in composition within this PMZ can be characterized by the dilution. The CPC gradient as a function of dilution was calculated at 400°C, 500°C, and 600°C to fully understand the extent of the driving force for carbon diffusion over a typical operating temperature range (400 and 500°C) and to also model an accelerated aging treatment (at 600°C) that was conducted experimentally. The dilution between the two materials was varied by 10% increments that resulted in 11 compositions (including Grade 22).

Aging simulations were performed using DICTRA® in conjunction with the two databases mentioned above and the MOB2 mobility database. The following phases were allowed to form under local equilibrium conditions based on relevant literature for each material: Austenite, ferrite, M\textsubscript{23}C\textsubscript{6}, M\textsubscript{6}C, and FCC2 (MC carbide phase). The length of the graded region was set as 50 μm for DMW calculations. This is a typical distance of the PMZ in a DWM. Grade lengths were varied for the GTJ calculations to develop the optimal grade design to minimize carbon diffusion. A linear composition gradient was assumed and included a source of each material as end members. Carbon diffusion and phase fraction profiles were extracted from the kinetic simulations for the DMWs and GTJs to compare to actual GTJs that were fabricated, aged at 600°C, and characterized.

Optimal grade lengths of 10 mm and 20 mm were chosen for the fabrication of the GTJs. These joints were fabricated using a dual wire gas tungsten arc welder, as described in prior work. Wire feed rates were varied for each layer within the GTJ to match a desired dilution for a given layer until the graded region was built to 100% filler metal. Samples were then placed in Fisher Scientific furnaces for an accelerated aging study at 600°C up to 8,000 hours to understand the microstructural evolution. Metallographic samples were cross sectioned and prepared using standard metallographic techniques to a final polished state of 0.05 μm colloidal silica. 2% Nital solution was used as an etchant to reveal grain boundaries and carbides in the ferritic material.

Compositional line traces with a 100 μm step size were conducted on the as-welded GTJs in the polished condition using a Leo Gemini 1550 VP scanning electron microscope equipped with an 80mm\textsuperscript{2} Oxford X-MAX silicon drift detector capable of count rates up to 100,000/sec. An operating voltage of 15 keV and 60 μm aperture allowed for 30% dead time. Secondary electron imaging and x-ray energy dispersive spectrometry (EDS) were performed in a Hitachi 4300SE/N Schottky field emission scanning electron microscope at an operating voltage of 15 keV. Compositions measured from this analysis are standardless and therefore, semi-quantitative. The accuracy of this technique was evaluated using custom “standards” analyzed via optical emission spectroscopy (OES). Table 3 shows the results from EDS alongside the OES results. The EDS measurements show reasonably good agreement with the OES measurements. Therefore, this measurement technique was sufficient enough to identify regions of local enrichment. Monte Carlo simulations using
the CASINO program demonstrated that the x-ray generation volume was approximately 1 μm³ under the operating conditions. Thus, results of sub-micron size particles are likely affected by the surrounding matrix material, but were still useful for identifying the formation of expected phases.

Results and Discussion

Chemical Potential of Carbon Calculations

The CPC was calculated for a wide range of alloys and compared to the CPC of Grade 22. Since the CPC gradient is the driving force for carbon diffusion, the most promising alloys will have a CPC similar to Grade 22. This will reduce the CPC gradient and corresponding driving force and thus, minimize carbon diffusion. Figure 1 shows the CPC of each material as a function of temperature with Grade 22 shown on the left (in red) for each temperature.

The justification of the selected candidate alloys are as follows: 347H, Inconel 182 and Inconel 82 are the three filler metals with the closest CPC to that of Grade 22 at each temperature. IN800 was included as a reference, as it represents the end member for the application (but is not available as filler metal). Work conducted by Brentrup et al. reported that the direct joining of Grade 22 to IN800 resulted in solidification cracks. Thus, IN800 would not be a viable filler metal for direct grading to Grade 22 steel. Inconel 617, 625, and 718 exhibit a relatively large difference in CPC compared to Grade 22 for all temperatures. In addition, recent results have shown undesirable brittle intermetallic phases would be stable when these filler metals are joined to a ferritic steel. Therefore, these three alloys do not appear to be good candidates and thus, were not considered. 347 was not considered since 347H has a higher carbon content (closer to Grade 22), and has a closer CPC to that of Grade 22.

Only considering the difference in CPC at each temperature, 347H, Inconel 182, and Inconel 82 were initially chosen as candidate alloys. However, as mentioned previously, the CTE mismatch at elevated temperatures between Grade 22 and the candidate alloys was also considered. In addition to having a reasonable CPC match, P87 has a closer CTE match to Grade 22 at elevated temperatures than the other alloys and was therefore considered. Thus, the candidate alloys chosen for this study were Inconel 82, 347H, and P87.

Within a GTJ, the Grade 22 steel will become diluted with the composition from the filler metal until the end of the graded region, which consists of “100% dilution” of filler metal (i.e. “pure” filler metal). The CPC gradient was determined by taking 10% increments of the candidate alloy’s dilution with Grade 22 (using Grade 22 and the filler metal compositions shown in Table 2) and calculating the CPC for each composition. Figure 2a-c shows the CPC as a function of dilution for the three candidate alloys. The results for Inconel 82 (Figure 2a) and 347H (Figure 2c) show a steep CPC gradient at the start of the dilution (after x=0). The gradient decreases towards the end of the dilution, and under certain conditions turns positive. Constitution boundary lines are superimposed onto the figure from the well-known Schaeffler diagram, which shows the major phases that form in ferritic and austenitic welds at a given composition. Note the Schaeffler diagram does not account for secondary phases such as carbides, thus only martensite and austenite are included. In each GTJ, martensite is expected to form at the beginning of the grade, encompassing Grade 22, followed by a region of mixed martensite and austenite. The
composition will reach a point where only austenite is expected to form. The beginning of the grade is where the CPC gradient is the steepest and the most carbon diffusion is therefore anticipated. Thus, as described in more detail in a later section, the beginning of the grade can be locally elongated to minimize the CPC gradient and extent of carbon diffusion.

**Aging Simulations**

A DMW composition gradient across the PMZ is typically on the order of 10 – 100 microns in length.\(^1\textsuperscript{,}5\textsuperscript{,}14\textsuperscript{,}29\) Therefore, the simulation for a baseline case for conventional DMWs was selected as 50 \(\mu\)m in length using a linear change in composition. DICTRA\textsuperscript{®} was used to model the carbon concentration as a function of distance for various aging times at an elevated temperature. Figure 3a shows the carbon concentration as a function of distance for a DMW between Grade 22 and Inconel 82 with a 50 \(\mu\)m PMZ aged at 500°C up to 8,000 hours. On either side of the 50 \(\mu\)m transition region is a source of the two end members (Grade 22 and Inconel 82). It is known that DMWs between ferritic and austenitic materials result in a carbon depleted region prior to the fusion line in the HAZ of the ferritic material, and a carbon enriched region in the PMZ.\(^15\textsuperscript{,}35\textsuperscript{,}36\) Thus, the model results are consistent with findings in literature.

The carbon concentration in the as-welded condition at the weld interface (x=10 mm) initially starts at 0.15 weight percent, and is reduced to 0.10 weight percent after simulated aging for 8,000 hours. Furthermore, carbon enrichment in the PMZ increased to 0.70 weight percent after aging 8,000 hours, which can lead to extensive carbide formation. Figure 3b shows the same simulation with an output of phase fraction as a function of distance after simulated aging for 8,000 hours at 500°C. Body centered cubic (BCC) is the dominant phase before the interface since Grade 22 is a ferritic material, and face centered cubic (FCC) becomes the dominant phase through the PMZ as the dilution increases towards Inconel 82. Studies have shown that DMWs fabricated with Grade 22 form \(\text{M}_{23}\text{C}_6\) and \(\text{M}_6\text{C}\) carbides in the HAZ of the ferritic steel.\(^1\textsuperscript{,}4\textsuperscript{,}14\textsuperscript{,}17\textsuperscript{,}27\) The model shows the formation of three types of carbides; \(\text{M}_{23}\text{C}_6\) chromium carbide which reaches a maximum of 0.08 phase fraction, \(\text{M}_6\text{C}\) molybdenum carbide at a max of 0.005 and MC (niobium) carbide a max of 0.02 phase fraction. Again, the model results are consistent with findings in literature.

The composition gradient controls the chemical potential gradient and concomitant carbon diffusion. With GTJs, the concentration gradient is decreased by simply increasing the distance over which the composition varies from Grade 22 base metal to the austenitic filler metal. The decrease in the composition gradient will decrease the chemical potential gradient and the extent of carbon diffusion in the system. Figure 4 compares the effect of temperature and grade length on carbon diffusion after simulated aging for 8,000 hours for a DMW and GTJ between Grade 22 and Inconel 82. Figure 4a-c has a transition region of 50 \(\mu\)m (typical of a DMW). As expected, an increase in aging temperature results in greater carbon depletion prior to the fusion line, and a significant increase in carbon enrichment in the PMZ. Figure 4d-f shows results of a GTJ with a transition region of 10 mm. The results have the same trend as the DMW results, but the extent of carbon diffusion is significantly reduced when compared to a typical DMW. (Note the differences in the scales of the y-axes between the DMW and GTJ). Carbon enrichment in the PMZ of the DMW is as high as 1.7 weight percent when aged for 8,000 hours at 600°C, while the GTJ exhibits carbon
enrichment of only 0.2 weight percent under the same conditions. This illustrates that an increase in grade length significantly reduces the driving force and the extent of carbon diffusion.

A summary of the modeling results for all DMWs and GTJs aged at 500°C is shown in Figure 5. The percent carbon loss at the weld interface is plotted as a function of simulated aging time up to 20 years. The percent carbon loss at the interface refers to the difference in carbon content after aging compared to the nominal Grade 22 concentration (0.15 weight percent). The three candidate alloys are plotted using grade lengths of 50 μm (labeled “DMW” in Figure 5), 10 mm, and 20 mm to resemble a conventional DMW and GTJs, respectively. It is interesting to note that, after an initial loss in carbon after 2,000 hours, the P87 and Inconel 82 DMWs then exhibit a decrease in carbon loss with additional aging time. The DMW made with 347H shows a continuous increase in carbon loss with time, also demonstrated by the GTJs. Results from this summary are discussed below.

The CPC and concentrations of Fe, Ni, Cr, and C as a function of distance are shown in Figure 6a, 6c, and 6e for the DMW 347H, and Figure 6b, 6d, and 6f for Inconel 82. The starting condition of the CPC in the as-welded condition for 347H (Figure 6a) shows a significant decline from Grade 22 towards 347H until it remains constant when x > 10.050 mm. Note that the slope of the CPC is always negative within the graded region. In contrast, Inconel 82 exhibits a steep decline in CPC until the CPC gradient turns positive around x = 10.025 mm (Figure 6b). This gradient change can be explained by the composition profiles for each system, combined with the CPC trends shown in Figure 7, that reveal the effect individual elements have on the CPC. The CPC difference compared to Grade 22 is plotted against the concentration for Cr, Nb, C, and Ni (Figure 7a). Individual elements in the Inconel 82 system were varied with a simultaneous change with Ni to show how each element effects the CPC. As shown, a decrease in Cr will increase the CPC, an effect that is more prevalent for higher Cr contents (Cr > 10 wt %), resulting in a value closer to Grade 22 (designated at the top of the graph). Conversely, the effect of Ni (replaced with Fe) is almost negligible until lower Ni concentrations, where a decrease will decrease the CPC, thus increasing the mismatch of Grade 22. Within the relatively small composition range of interest, C and Nb have little effect on Inconel 82’s ability to minimize the mismatch of Grade 22’s CPC. While the effects are small, a decrease in Nb and an increase in C will increase the CPC, thus decreasing the CPC mismatch of Grade 22. The trends were also conducted on Grade 22 as a baseline while simultaneously varying Fe, with the trends shown in Figure 7b. The trends between the two materials are the same, however the slopes (and corresponding relevance) is different. For example, an increase in Nb in Grade 22 decreases the CPC almost 50 kJ/mol when compared to the change of Nb in Inconel 82. Additionally, the increase in Cr has a larger affect for low Cr values. Although these results are insightful, the CPC values are for the end members (base and filler metals), and does not account for variations in the PMZ. For example, decreasing the Cr content will decrease the mismatch of the CPC, but worsen the effects of the CPC “well” in the PMZ.

To fully understand the cause for the “well” seen in the PMZ between Grade 22 and Inconel 82, individual elements were varied and the initial CPC was calculated throughout the PMZ. It was determined the Nb concentration had the largest effect on the “well”, as shown in Figure 7c. The CPC as a function of distance for two Nb concentrations in Inconel 82 is shown, along with the phase fraction of the Cr and Nb carbides. A higher
Nb concentration causes the Cr carbide (M\textsubscript{23}C\textsubscript{6}) to dissolve at the expense of the Nb carbide (NbC), which significantly affects the CPC of the system. This is the condition the Grade 22-Inconel 82 DMW experiences. As expected, an increase in the Nb concentration results in an increase in stability of the NbC phase, which decreases the CPC in the PMZ. However, a decrease in Nb in Inconel 82 results in an increase in CPC in the PMZ, minimizing the formation of the “well”. Further evidence of the Nb effect is shown in Figure 7d for the DMW between Grade 22 and 347H. Similarly, two Nb concentrations (0.15 wt%, representative of 347H, and 3 wt%) were investigated to determine the effect on the 347H system that contains similar Cr contents, but significantly less Ni than Inconel 82. The increase in Nb concentration causes the M\textsubscript{23}C\textsubscript{6} to dissolve, as seen in the previous condition. This results in a steep reduction in the CPC. Increasing the Ni concentration in combination with matching the other elements to that of Inconel 82 will have an effect of increasing the CPC in the end member, thus creating a “well” in the PMZ.

Looking back at Figure 6e, there is a localized carbon depletion at the Grade 22-PMZ interface (x=10 mm), and the carbon concentration decreases with increasing aging time when x<10.025 mm. The carbon concentration increases with aging time when x>10.025 mm. The local decrease in carbon at the interface is attributed to the abrupt discontinuity of the CPC observed in the as-welded condition at the start of the PMZ in Figure 6a. To alleviate this discontinuity, carbon decreases in the Grade 22 during aging, since a decrease in carbon decreases the CPC (Figure 7a). The decrease in carbon concentration with aging when x<10.025 mm, and increase in carbon beyond this point, is attributed to the curvature of the CPC gradient shown in Figure 6a. The CPC profile has a negative curvature (concave down) for x<10.025 mm and, as a result, the carbon concentration values decrease with time (in agreement with Fick’s second law of diffusion). In contrast, for x>10.025 mm, the CPC profile has a positive curvature (concave up) so the same values increase. This also accounts for the localized enrichment shown by the carbon peaks in the PMZ.

The principles discussed above can be applied to the trends shown in the DMW between Grade 22 and Inconel 82. However, the difference between 347H and Inconel 82 filler metals lies in the behavior of the CPC curve and corresponding carbon profile at the Grade 22/PMZ interface. After the initial decrease in carbon at and near the interface after 2,000 hours (associated with alleviating the CPC discontinuity) the carbon content increases with aging time in the DMW between Grade 22 and Inconel 82. Although minor, this increase in carbon (not seen in the GTJs or 347H DMW) after aging beyond 2,000 hours is attributed to the behavior of the CPC curve and the presence of the CPC “well” (located in the center of the PMZ in Figure 6b) that reduces the driving force for carbon diffusion. Since carbon cannot diffuse up the CPC gradient, a pile-up of carbon will occur that causes an overall increase in carbon content that continues into the Grade 22. Additionally, due to the shape of the “well”, there will be carbon diffusion from both the Grade 22 and Inconel 82. This is evident in Figure 6f at x=10.05 mm. Carbon will diffuse down the right side of the CPC well resulting in a carbon depleted region at the start of the “pure” Inconel 82, and a localized carbon enriched region at the end of the PMZ. This explains why Ni-base filler metals reduce the extent of carbon diffusion, which has already been reported in literature.\textsuperscript{1,14,37} However, the fundamental reason for this reduction has not been recognized, as the CPC “well” has not been reported. This phenomenon explains the underlying reason for the increase in carbon observed before the interface that is not
present in DMWs made with Fe-base (as opposed to Ni-base) filler metals. The DMW using P87 (a Ni-base filler metal) follows almost identical trends as Inconel 82. Therefore only Inconel 82 was discussed.

The carbon content was calculated for each phase throughout the simulated GTJs aged at 500°C for 8,000 hours. The results for the GTJ between Grade 22 and Inconel 82 are shown in Figure 8a, where the carbon concentration in each phase as a function of distance is shown for the five stable phases. The carbon resides almost completely in the carbides, as the BCC and FCC phases have negligible carbon contents. This result can assist in understanding the microstructures of actual aged DMWs with regards to the extent of carbon, as a region enriched in carbides suggests a large extent of carbon. Thus, the observed carbides are a good indication of the extent of carbon. Additionally, the observed carbides can be compared with the calculated carbon content shown in Figure 6e-f. Figure 8b shows the carbide distribution in the unaffected Grade 22 base metal (i.e. away from the interface). The DMW using 347H shows a carbide depleted region in the Grade 22 prior to the weld interface (shown far left in Figure 8c), with carbides (represented by white particles) in the matrix. This can be directly compared to the DMW using Inconel 82 (Figure 8d) that shows a larger extent of carbides in the Grade 22 HAZ matrix at the interface (left of the image). This observation of a larger carbide depleted region in the DMW using 347H is consistent with the calculated carbon in Figure 6e-f, and provides further evidence that using a Ni-base filler metal reduces carbon diffusion. Furthermore, another major microstructural difference between the two DMWs is the formation of a larger martensitic layer in the PMZ of the DMW using 347H. A thicker martensite layer is expected in welds produced with Fe-base austenitic filler metals, as shown from DuPont and Kusko. This can be attributed to the smaller concentration gradient within the PMZ, when compared a Ni-base filler metal, due to the lower Ni concentration. A larger Ni concentration will stabilize austenite at a shorter location in the PMZ, resulting in a thinner martensitic layer. This is consistent with the calculated phase fractions for the DMWs using Inconel 82 and 347H aged for 8,000 hours at 600°C, shown in Figure 9. After 10 μm, there is no BCC phase in the Inconel 82 DMW, and the FCC phase consists of 90% of the system. Conversely, it takes a further distance, more than half of the PMZ, for the FCC phase in 347H to become the dominant phase.

The simulated GTJs for the same filler metals exhibit significantly less carbon depletion at the interface. This is illustrated in Figure 10a-b and Figure 10c-d that plots both the carbon and CPC profiles for 347H and Inconel 82, respectively. In both GTJs, carbon decreases prior to the interface with increased aging time, but the reduction is minimal relative to a DMW. Since the CPC gradient is significantly lower than that in the DMWs, the driving force is significantly lower for carbon diffusion to occur. Although a CPC “well” still occurs in Inconel 82 GTJ (Figure 10b), the aging time would need to be drastically extended for the carbon content at the interface to increase. For comparison, a 2 mm graded region was modeled under the same conditions as the DMWs and GTJs (500°C), and the aging time necessary for carbon to increase after the initial depletion prior to the interface is 1,000 years. These results demonstrate that grade lengths of 10 mm and 20 mm for each material result in less than 20% carbon loss at the weld interface when aged up to 20 years. Based on the findings, GTJs with graded regions of 10 mm and 20 mm in length were fabricated for this study.
**Reduced Composition Gradient**

As shown in Figure 2, the CPC gradient produced from a linear change in dilution (i.e. linear composition gradient) is generally the largest at the beginning of the grade, and appreciably decreases (or becomes positive) towards the “pure” filler metal side. Thus, carbon diffusion can be further minimized by simply elongating the grade locally within this region in order to reduce the concentration gradient, the corresponding CPC gradient, and the concomitant carbon diffusion. For example, the CPC gradient at 500°C for Inconel 82 (Figure 2a) illustrates that a composition at 60% dilution Inconel 82 represents the location where the gradient becomes positive. This composition was chosen to become the new end member of the graded region. Maintaining a linear concentration gradient, the new graded region has a composition from Grade 22 to an end member of 60% dilution Inconel 82 expanded over the same grade length. Figure 11 shows the carbon concentration as a function of distance for a GTJ 10 mm in length, aged at 500°C up to 20 years. Figure 11a shows the original carbon concentration curve that contained the initial CPC gradient, while Figure 11b shows the carbon profile with the revised composition and reduced CPC gradient. Note in Figure 11b, the final carbon content is 0.12 weight percent C in the Inconel 82, which is consistent with 60% dilution Inconel 82. The original end member contained 0.10 weight percent C. These results clearly show a decrease in the CPC gradient results in less carbon depletion prior to the fusion line in the Grade 22 HAZ and less carbon enrichment into the graded region. These results are insightful for future endeavors as they demonstrate how the graded region can be fabricated for the most effective results.

**Simulation Results for Fabricated GTJs**

The modeling results discussed thus far are useful to understand the effects of temperature on carbon diffusion and provide guidance on the optimal grade length required to reduce carbon diffusion. However, the results to date have assumed a linear composition gradient. In a graded material made with welding and additive manufacturing processes, it is not often recognized that the composition is uniform within each layer, and rather steep composition gradients exist between the layers. Figure 12 shows the EDS results for the major alloying elements Fe, Ni, and Cr across the GTJ between Grade 22 (left) and Inconel 82 (right). There is a graded region between the two end members with each layer representing a different composition. This result shows the composition gradient is not linear, and is actually a step function. Thus, the experimentally measured compositions of each layer were averaged to create a revised profile for modeling that consisted of a constant composition for each layer with a 50 μm partially mixed region at each layer interface.

Due to the difficulty of accurately measuring low amounts of carbon, the carbon concentration gradient was calculated through knowledge of the measured dilutions of the major alloying elements. The calculated carbon profile was used for the model, along with the other measured elements, to simulate a GTJ aged up to 8,000 hours at 600°C (matching experimental conditions). Figure 13a shows the carbon concentration as a function of distance for the GTJ between Grade 22 and Inconel 82 simulated at 600°C for 8,000 hours. The individual layers are marked between the vertical red lines, and designated as L1 for layer one, L2 for layer two, etc. The as-welded condition (time=0) shows the initial carbon profile as a function of distance throughout the GTJ. The results show that carbon diffusion is locally enhanced between the individual layers at the
interfaces, particularly near the Grade 22 side of the GTJ. The carbon diffusion is minimal when \( x > 20 \) mm. This can be attributed to the FCC phase dominating this region, as carbon is known to diffuse slower through an FCC matrix than in a BCC matrix. This is consistent with the enhanced carbon diffusion in the beginning of the grade where the BCC is the dominate phase. These results indicate that reducing the composition gradient at the beginning of the graded region could further decrease the extent of carbon diffusion expected under the current conditions.

This result can be directly compared to the conventional DMW from Figure 4c for identical simulation conditions of 8,000 hours at 600°C. Note that the extent of carbon diffusion is still significantly higher in the conventional DMW when compared to the GTJ with the step function composition. Carbon enriched regions reach up to 1.2 weight percent C in the DMW, but only reach about 0.2 weight percent in the GTJ. Thus, even though carbon diffusion is enhanced between the layers within the GTJ, the extent of the depletion and enrichment is still an order of magnitude lower than the DMW.

Simulations were also conducted to calculate the stable phases throughout the GTJ with the stepped composition profile. Figure 13b shows the phase fraction as a function of distance between Grade 22 and Inconel 82 aged for 2,000 hours at 600°C. BCC is the dominant phase in the beginning of the grade, and FCC dominates as the composition transitions to Inconel 82. Layers L2-L4 contain both BCC and FCC at 600°C. This transition is not as dramatic as a DMW transition seen in Figure 3b, thus the steep changes in phases and corresponding microstructures are reduced. Figure 13b also shows the amount of carbides in the system. There is a depletion of \( \text{M}_{23}\text{C}_6 \) carbides prior to every interface, with an enrichment into the subsequent layer. Furthermore, Nb carbides become the dominant carbide in the graded region towards Inconel 82.

These results are consistent with experimental observations and Figure 14 shows how the carbide content varies throughout the graded region. The unaffected base metal is shown in Figure 14a for reference. The HAZ adjacent to L1, beginning and end of L1, and beginning of L2 are shown in Figure 14b–e for the GTJ using Inconel 82 aged at 600°C for 4,000 hours. The phase fraction calculates \( \text{M}_{23}\text{C}_6 \) carbide depletion and enrichment at the layer interfaces, which matches well with the microstructures in each region. The HAZ (Figure 14b) depicts a region of a coarse-grained, tempered martensite that is depleted of carbides. This can be attributed to the prolonged aging treatment that caused tempering and carbon diffusion from this region. The beginning of L1 (Figure 14c) shows a relatively large population of carbides in a fine lath-like structure. EDS conducted on the white particles in the matrix confirmed an enrichment in the Cr content, suggesting the presence of \( \text{M}_{23}\text{C}_6 \) chromium carbides. The calculated carbide profile shows a decrease across L1, and a peak into L2. Again, this is consistent with the observed microstructure features. The end of L1 (Figure 14d) shows a coarse structure depleted in carbides (compared to the beginning of the layer), and a significant change in microstructure into L2 (Figure 14e). There is a secondary phase that forms at the initiation of L2 that will be discussed in a later section, however smaller carbides are observed between the matrix and secondary phase that are in good agreement with the simulation.

The discussion thus far has been in reference to the GTJ made between Grade 22 and Inconel 82. As stated earlier, three candidate alloys were chosen for this study. Therefore, simulations conducted on the Grade 22-Inconel 82 system were also performed on alloys P87 and 347H. Diffusion simulations were conducted for GTJs involving each
candidate filler metal in order to compare differences in diffusion behavior expected from the various filler metals. The summary of simulated carbon diffusion can be seen in Figure 15a and 15b, which shows the percent of carbon depletion and enrichment for each layer, respectively. The results indicate P87 to be the most promising candidate alloy, since the extent of carbon depletion and enrichment near the interfaces are generally the lowest for P87.

**Major Phase Analysis**

Figure 16 shows the microstructures of the first four layers in the GTJ between Grade 22 and Inconel 82. L1 contains a high population of carbides (Figure 16a). A secondary constituent forms and increases in volume fraction between L2 and L3, and L4 consists almost entirely of this constituent (Figure 16d). The formation of this constituent can be explained through the calculations of phase fraction seen in Figure 17. At 600°C (the aging temperature), L1 is ferrite (BCC) with carbides. Austenite (FCC) does not exist in L1 at 600°C, but is increasingly stable at 600°C in L2-L4, shown in Figure 17b-17d, respectively. The FCC phase at 600°C will transform to martensite with no change in composition when cooled to room temperature. This was confirmed through SEM imaging of this constituent combined with electron back scatter diffraction, which did not detect austenite in the beginning layers where this constituent was present. Figure 18a-b show the calculated and measured compositions for the major alloying elements in the FCC and BCC phases, respectively. As shown, the calculated composition of Ni enriched FCC at 600°C is in excellent agreement with the room temperature EDS measurements on the secondary phase. The as-welded microstructure did not contain this Ni-rich martensitic constituent. Thus, aging the GTJ at 600°C will result in portions of the sample transforming to austenite that, upon cooling, transform to Ni-rich martensitic.

**Actual Compositions Modeled**

The DMW simulations discussed thus far have used composition gradients that induce the largest CPC mismatch and therefore, the largest extent of carbon diffusion. This included carbon values of 0.15 weight percent in Grade 22, and the lowest allowable carbon value for the filler metals. Additionally, the carbon in the GTJs was chosen to match the Grade 22 starting condition for comparison (0.15 weight percent). However, the carbon in Grade 22 was chemically analyzed and measured to be 0.07 weight percent. Furthermore, the carbon in the filler metals was higher than the values used for the simulations. Therefore, the simulations were conducted with the exact compositions for Grade 22 and all filler metals for the DMWs, and a corrected carbon content for the GTJs for a direct comparison to the experimental results. The actual compositions can be seen in Table 4.

The results are shown in Figure 19 that represent the carbon profiles for both the DMW and GTJ aged at 600°C using Inconel 82 (Figure 19a-b), P87 (Figure 19c-d), and 347H (Figure 19e-f). The trends are similar for the DMW and GTJ using Inconel 82, only a lower extent of carbon diffusion due to the decrease in the original carbon content (0.15 to 0.07 weight percent). However, the DMW and GTJ using P87 have a notable change, since the carbon in Grade 22 is less than the carbon in P87, resulting in a positive concentration gradient. Due to this change in gradient, a phenomenon known as “up-hill diffusion” occurs, where the carbon will still deplete in Grade 22 and diffuse to the P87 weld metal (Figure 19c). This is attributed to the CPC mismatch that drives carbon to
diffuse down the CPC gradient, leaving Grade 22 and migrate to the P87 weld metal. Further evidence is shown in Figure 19d for the GTJ using P87, where the carbon increases from 0.07 to 0.11 weight percent, yet carbon diffusion occurs at the layer interfaces due to the CPC mismatch. This is insightful because, regardless of the carbon concentration gradient, carbon will still follow the CPC gradient and diffuse to regions to reduce the mismatch in CPC. Finally, the DMW using 347H shows similar trends to that in Figure 6e. However, a temperature of 600°C was used for the experimental aging treatment, and it is expected a larger degree of carbon diffusion to occur at higher temperatures. This is consistent between the two figures, as the carbon depleted region in Grade 22 at 600°C is essentially at zero under equilibrium conditions (Figure 19e). The 347H GTJ using the revised carbon content still exhibits carbon diffusion at the layer interfaces. However, similar to the other GJTs, the extent is not as significant as when carbon was 0.15 weight percent.

Conclusions
Thermodynamic and diffusion simulations were conducted to understand phase stability and carbon diffusion behavior in DMWs and GTJs. The following conclusions can be drawn from this work.

1. The chemical potential gradient and associated extent of carbon diffusion associated with joining dissimilar alloys can be reduced by simply extending the grade length within the region of the grade where the CPC gradient is negative.
2. For GTJs made with additive manufacturing processes that involve multiple layers of constant compositions (i.e. stepped compositions), enhanced carbon diffusion is expected at the layer interfaces due to locally high CPC gradients. However, the extent of carbon diffusion near the layer interfaces within such GTJs is still expected to be significantly less than that of conventional DMWs.
3. The reduction of carbon diffusion (in the HAZ of Grade 22) in DMWs using Ni-base filler metals is attributed to the CPC “well” located in the PMZ. This “well” causes a pile-up effect that restricts carbon diffusion. The “well” is not present in the DMW using 347H, therefore the carbon content decreases in the HAZ with aging.
4. A portion of the GTJs will transform to FCC austenite during aging at 600°C. This FCC phase then transforms to martensite with no change in composition during cooling to room temperature.
References

22. Thermo-Calc Software Ni-Data-v7 Ni-Alloys Database.
25. Thermo-Calc Software MOB2 TCS Alloy Mobility Database.
Figures

Figure 1. Chemical potential of carbon for a number of candidate alloys at various temperatures.
Figure 2. Chemical potential of carbon curves as a function of dilution for the three candidate alloys a) Inconel 82, b) P87, and c) 347H.
Figure 3. Typical a DMW between Grade 22 and Inconel 82 aged at 500°C showing a) carbon concentration profile with a 50 μm partially mixed zone and b) phase fractions plots aged at 500°C after 8,000 hours.
Figure 4. Carbon concentration profiles for a DMWs between Grade 22 and Inconel 82 aged at a) 400°C, b) 500°C and c) 600°C, and for GTJs aged at d) 400°C, e) 500°C and f) 600°C.
Figure 5. Summary for the three candidate alloys aged at 500°C showing the percent Carbon loss at interface up to 20 years.
Figure 6. CPC, Composition, and carbon concentration as a function of distance for 347H (a, c, e) and Inconel 82 (b, d, f), respectively.
Figure 7. Chemical potential of carbon difference as a function of alloy concentration Cr, Ni, Nb, and C using a) Inconel 82 as a baseline and b) grade 22 as a baseline. The effect of Nb on the CPC and corresponding carbide phases is shown in the DMW between Grade 22 and c) Inconel 82 and d) 347H.
Figure 8. a) Calculation of the amount of carbon in each phase for DMW between Grade 22 and Inconel 82 after simulated age at 500°C for 8,000 hours, and photomicrographs showing the extent of carbon diffusion after aging at 600°C for 8,000 hours for b) Grade 22 base metal, c) the interface of the DMW between Grade 22 and 347H and d) interface between Grade 22 and Inconel 82.
Figure 9. Phase fraction of BCC and FCC between Grade 22 and two filler metals Inconel 82 and 347H showing the difference in FCC stability in the PMZ after 8,000 hours at 600°C.

Figure 10. Carbon concentration and CPC as a function of distance for a 10 mm GTJ aged at 500°C 347H (a-b) and Inconel 82 (c-d).
Figure 11. Carbon concentration as a function of distance for a 10 mm GTJ aged at 500°C showing a) the original composition gradient and b) the revised composition that reduced the CPC gradient.
Figure 12. EDS trace of a 10 layer GTJ showing the main alloying elements iron, nickel, and chromium in the as-welded condition.
Figure 13. Carbon concentration as a function of distance for the GTJ made between Grade 22 and Inconel 82 simulated aged at 600°C up to 8,000 hours and b) phase fraction as a function of distance aged for 8,000 hours at 600°C showing all phases and a magnified view of carbide phases.
Figure 14. SEM photomicrograph of the GTJ made between Grade 22 and Inconel 82 aged for 4,000 hours at 600°C showing a) base metal, b) HAZ prior to interface, c) beginning of L1, d) end of L1, and e) beginning of L2.
Figure 15. Carbon diffusion results for the three filler metals showing a) percent carbon loss before each interface and b) percent carbon gain after each interface.
Figure 16. SEM photomicrographs of the extent of the secondary constituent in the GTJ using Inconel 82 aged for 2,000 hours at 600°C showing a) layer 1, b) layer 2, c) layer 3, and d) layer 4.
Figure 17. Calculated phase fraction as a function of temperatures for a) layer 1, b) layer 2, c) layer 3, and d) layer 4 for the GTJ using Inconel 82 for a simulated age at 600°C.
Figure 18. The measured and calculated compositions as a function of layers for the a) FCC phase and b) BCC phase in the GTJ using Inconel 82.
Figure 19. Carbon diffusion simulations with supplier provided/chemical analysis tested compositions with the revised carbon profiles for the DMW and GTJs for Inconel 82 (a-b), P87 (c-d), and 347H (e-f).
### Tables

**Table 1. Composition range for alloys used in this study.**

<table>
<thead>
<tr>
<th>Material</th>
<th>Fe</th>
<th>Al</th>
<th>C</th>
<th>Cr</th>
<th>Cu</th>
<th>Mn</th>
<th>Mo</th>
<th>Nb</th>
<th>Ni</th>
<th>Si</th>
<th>Ti</th>
<th>Co</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grade 22</td>
<td>Bal</td>
<td>-0.04-0.15</td>
<td>1.00-2.62</td>
<td>0.25 max</td>
<td>0.60 max</td>
<td>80.0-1.15</td>
<td>0.50 max</td>
<td>1.00 max</td>
<td>0.05 max</td>
<td>0.05 max</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>P87</td>
<td>38.0-42.0</td>
<td>0.10-0.20</td>
<td>0.80-0.14</td>
<td>8.50-9.50</td>
<td>0.25 max</td>
<td>1.20-1.80</td>
<td>1.80-2.20</td>
<td>0.90-1.40</td>
<td>54.0 max</td>
<td>0.05</td>
<td>0.05 max</td>
<td>-</td>
</tr>
<tr>
<td>347H</td>
<td>38.0-42.0</td>
<td>0.04-0.10</td>
<td>17.0-20.0</td>
<td>-</td>
<td>2.00 max</td>
<td>0.32-1.00</td>
<td>9.00-13.0</td>
<td>1.00 max</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>IN82 (ER NiCr-3)</td>
<td>3.00 max</td>
<td>0.10 max</td>
<td>18.0-22.0</td>
<td>0.50 max</td>
<td>2.50-3.50</td>
<td>2.00-3.00</td>
<td>67.0 min</td>
<td>0.50 max</td>
<td>0.75 max</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>Alloy 800</td>
<td>Bal</td>
<td>0.15-0.60</td>
<td>0.05-0.10</td>
<td>19.0-23.0</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>30-35</td>
<td>-</td>
<td>0.15-0.60</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>347</td>
<td>Bal</td>
<td>0.04-0.08</td>
<td>17.0-19.0</td>
<td>-</td>
<td>2.00 max</td>
<td>-</td>
<td>-</td>
<td>9.00-13.0</td>
<td>0.75 max</td>
<td>-</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>IN82 (ER NiCrFe-3)</td>
<td>10.0 max</td>
<td>0.10 max</td>
<td>13.0-17.0</td>
<td>0.50 max</td>
<td>5.00-9.50</td>
<td>1.00 max</td>
<td>59.0 min</td>
<td>1.00 max</td>
<td>1.00 max</td>
<td>-</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>IN617 (ER NiCrCo-Mo-1)</td>
<td>3.00 max</td>
<td>0.80-1.50</td>
<td>0.05-0.15</td>
<td>20.0-24.0</td>
<td>0.50 max</td>
<td>1.00 max</td>
<td>8.00-10.0</td>
<td>-</td>
<td>Bal</td>
<td>1.00 max</td>
<td>0.60 max</td>
<td>10.0-15.0</td>
</tr>
<tr>
<td>IN625 (ER NiCrMo-3)</td>
<td>5.00 max</td>
<td>0.04 max</td>
<td>0.10 max</td>
<td>20.0-23.0</td>
<td>-</td>
<td>0.50 max</td>
<td>8.00-10.0</td>
<td>3.15-4.15</td>
<td>Bal</td>
<td>0.50 max</td>
<td>0.40 max</td>
<td>1.00 max</td>
</tr>
<tr>
<td>IN718</td>
<td>Bal</td>
<td>0.20-0.80</td>
<td>0.08 max</td>
<td>17.0-21.0</td>
<td>0.30 max</td>
<td>0.35 max</td>
<td>2.80-3.30</td>
<td>4.75-5.50</td>
<td>50.0-55.0</td>
<td>0.35 max</td>
<td>0.65-1.15</td>
<td>1.00 max</td>
</tr>
</tbody>
</table>

**Table 2. Actual compositions used for modeling.**

<table>
<thead>
<tr>
<th>Material</th>
<th>Fe</th>
<th>Al</th>
<th>C</th>
<th>Cr</th>
<th>Cu</th>
<th>Mn</th>
<th>Mo</th>
<th>Nb</th>
<th>Ni</th>
<th>Si</th>
<th>Ti</th>
<th>Co</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grade 22</td>
<td>95.8</td>
<td>0.01</td>
<td>0.15</td>
<td>1.90</td>
<td>0.14</td>
<td>0.59</td>
<td>0.87</td>
<td>-</td>
<td>0.05</td>
<td>0.47</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>P87</td>
<td>30.0</td>
<td>0.20</td>
<td>0.08</td>
<td>9.50</td>
<td>0.25</td>
<td>1.80</td>
<td>2.20</td>
<td>1.40</td>
<td>54.0</td>
<td>0.50</td>
<td>0.05</td>
<td>-</td>
</tr>
<tr>
<td>347H</td>
<td>64.9</td>
<td>-</td>
<td>0.04</td>
<td>20.0</td>
<td>-</td>
<td>1.03</td>
<td>0.15</td>
<td>13.0</td>
<td>0.75</td>
<td>-</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Inconel 82</td>
<td>3.00</td>
<td>-</td>
<td>0.10</td>
<td>22.0</td>
<td>0.50</td>
<td>3.50</td>
<td>-</td>
<td>3.00</td>
<td>66.7</td>
<td>0.50</td>
<td>0.75</td>
<td>-</td>
</tr>
<tr>
<td>Alloy 800</td>
<td>41.3</td>
<td>0.42</td>
<td>0.09</td>
<td>20.9</td>
<td>0.27</td>
<td>1.03</td>
<td>0.16</td>
<td>34.7</td>
<td>0.45</td>
<td>0.56</td>
<td>-</td>
<td></td>
</tr>
<tr>
<td>347</td>
<td>68.3</td>
<td>-</td>
<td>0.05</td>
<td>19.4</td>
<td>0.13</td>
<td>1.68</td>
<td>0.63</td>
<td>9.13</td>
<td>0.46</td>
<td>-</td>
<td></td>
<td></td>
</tr>
<tr>
<td>IN82</td>
<td>10.0</td>
<td>-</td>
<td>0.10</td>
<td>15.0</td>
<td>-</td>
<td>7.25</td>
<td>1.75</td>
<td>63.9</td>
<td>1.00</td>
<td>-</td>
<td></td>
<td></td>
</tr>
<tr>
<td>IN617</td>
<td>3.00</td>
<td>1.15</td>
<td>0.10</td>
<td>22.0</td>
<td>0.50</td>
<td>1.00</td>
<td>9.00</td>
<td>49.2</td>
<td>1.00</td>
<td>0.60</td>
<td>12.5</td>
<td></td>
</tr>
<tr>
<td>IN625</td>
<td>5.00</td>
<td>0.40</td>
<td>0.10</td>
<td>21.5</td>
<td>-</td>
<td>0.50</td>
<td>9.00</td>
<td>3.75</td>
<td>58.9</td>
<td>0.50</td>
<td>0.40</td>
<td>-</td>
</tr>
<tr>
<td>IN718</td>
<td>16.4</td>
<td>0.50</td>
<td>0.08</td>
<td>19.0</td>
<td>0.30</td>
<td>0.35</td>
<td>3.10</td>
<td>5.00</td>
<td>53.0</td>
<td>0.35</td>
<td>0.90</td>
<td>1.00</td>
</tr>
</tbody>
</table>

**Table 3. Average EDS measurements compared to OES results for the custom standards.**

<table>
<thead>
<tr>
<th>Standards</th>
<th>Si</th>
<th>Cr</th>
<th>Mn</th>
<th>Fe</th>
<th>Co</th>
<th>Ni</th>
<th>Cu</th>
<th>Nb</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy 800</td>
<td>Ave</td>
<td>0.33</td>
<td>22.4</td>
<td>0.95</td>
<td>44.6</td>
<td>0.21</td>
<td>31.9</td>
<td>0.15</td>
<td>0.42</td>
</tr>
<tr>
<td>OES</td>
<td>0.44</td>
<td>20.9</td>
<td>1.03</td>
<td>41.1</td>
<td>0.07</td>
<td>34.7</td>
<td>0.27</td>
<td>0.15</td>
<td>0.16</td>
</tr>
<tr>
<td>Ave</td>
<td>0.72</td>
<td>18.2</td>
<td>1.81</td>
<td>67.8</td>
<td>0.29</td>
<td>9.51</td>
<td>0.38</td>
<td>0.76</td>
<td>0.53</td>
</tr>
<tr>
<td>OES</td>
<td>0.55</td>
<td>17.4</td>
<td>1.67</td>
<td>69.9</td>
<td>N/A</td>
<td>9.60</td>
<td>0.47</td>
<td>N/A</td>
<td>0.34</td>
</tr>
<tr>
<td>Ave</td>
<td>0.49</td>
<td>2.37</td>
<td>0.57</td>
<td>94.8</td>
<td>0.21</td>
<td>0.07</td>
<td>0.03</td>
<td>N/A</td>
<td>1.41</td>
</tr>
<tr>
<td>OES</td>
<td>0.38</td>
<td>2.19</td>
<td>0.50</td>
<td>Bal</td>
<td>N/A</td>
<td>0.06</td>
<td>0.09</td>
<td>N/A</td>
<td>1.01</td>
</tr>
</tbody>
</table>

**Table 4. Certified compositions used for the final DMW and GTJs.**

<table>
<thead>
<tr>
<th>Material</th>
<th>Al</th>
<th>C</th>
<th>Cr</th>
<th>Cu</th>
<th>Mn</th>
<th>Mo</th>
<th>Nb</th>
<th>Ni</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Ti</th>
<th>Fe</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grade 22 Substrate</td>
<td>0.03</td>
<td>0.07</td>
<td>2.20</td>
<td>0.14</td>
<td>0.52</td>
<td>0.92</td>
<td>&lt;0.01</td>
<td>0.10</td>
<td>0.007</td>
<td>0.009</td>
<td>0.20</td>
<td>&lt;0.01</td>
<td>Bal.</td>
</tr>
<tr>
<td>Inconel 82</td>
<td>0.027</td>
<td>21.4</td>
<td>0.016</td>
<td>3.20</td>
<td>2.36</td>
<td>70.8</td>
<td>0.003</td>
<td>&lt;0.001</td>
<td>0.19</td>
<td>0.43</td>
<td>Bal.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>EPRI P87</td>
<td>0.18</td>
<td>0.11</td>
<td>8.99</td>
<td>-</td>
<td>1.53</td>
<td>1.93</td>
<td>1.08</td>
<td>Bal.</td>
<td>0.004</td>
<td>0.001</td>
<td>0.22</td>
<td>0.10</td>
<td>40.4</td>
</tr>
<tr>
<td>347H</td>
<td>-</td>
<td>0.05</td>
<td>19.4</td>
<td>0.15</td>
<td>1.75</td>
<td>0.29</td>
<td>0.68</td>
<td>9.07</td>
<td>0.02</td>
<td>0.01</td>
<td>0.44</td>
<td>-</td>
<td>Bal.</td>
</tr>
<tr>
<td>90S-83 (Grade 22 Wire)</td>
<td>0.01</td>
<td>0.07</td>
<td>2.45</td>
<td>0.16</td>
<td>0.60</td>
<td>0.93</td>
<td>&lt;0.01</td>
<td>0.13</td>
<td>&lt;0.005</td>
<td>0.005</td>
<td>0.58</td>
<td>-</td>
<td>Bal.</td>
</tr>
</tbody>
</table>
2. Microstructural Evolution of Graded Transition Joints

Abstract

Carbon diffusion and the associated microstructural changes in dissimilar metal welds (DMWs) at elevated temperatures leads to a microstructure that is susceptible to premature failure. Graded transition joints (GTJs) can potentially provide a viable replacement to prolong the service life of these components. The purpose of the current investigation is to fabricate, age and characterize GTJs using three candidate filler metals (Inconel 82, EPRI P87, and 347H) to understand the microstructural evolution at elevated temperatures. Microhardness measurements were performed on the GTJs in the as-welded and aged conditions to understand the initial strength gradients throughout the graded region and how they evolve with aging time. Additionally, energy dispersive spectrometry was performed to measure the compositional gradients, which were input into thermodynamic and kinetic calculations to understand the carbon diffusion behavior and phase stability. Enhanced carbon diffusion occurred at the layer interfaces in the graded region of the GTJ, which indicated important regions that undergo microstructural evolution. The hardness results also revealed hardness changes at the layer interfaces. The analyzed interfaces demonstrated that carbon diffusion and corresponding carbide redistribution occurred that accounted for the observed hardness gradients. Additionally, the transition from a martensitic to austenitic region was observed in each GTJ that contributed to the hardness variations in the graded region. Finally, the formation of a nickel-rich martensitic constituent was observed in the graded region of all filler metals after aging. This constituent was originally austenite at the aging temperature and transformed to martensite with no change in composition upon cooling. The morphologies of the constituent in the three filler metals are presented and discussed.
Introduction

Dissimilar metal welds (DMWs) are critical for design, development, and manufacturing of very high temperature reactors, specifically steam generators in the nuclear industry. Experience has demonstrated that failures of such DMWs can be caused by strain localization attributed to carbon diffusion and thermal stresses due to the chemical potential of carbon (CPC) gradient and the coefficient of thermal expansion (CTE) and mechanical property mismatch. Each of these factors is caused by the large variation in composition across the narrow interface between the two dissimilar materials. These failures can occur well before the expected service lifetime and can cost power plants up to $850,000 per day in lost revenue and repair costs. A potential solution to DMW failures is the design and implementation of graded transition joints (GTJs).

Functionally graded materials have been considered for use in components exposed to high temperature gradients. A typical functionally graded material consists of a ceramic-metal mixture that can be tailored to utilize the advantages of the individual material properties. The ceramic part has good thermal and corrosion resistance capabilities, and the metallic part provides superior fracture toughness and ductility. A GTJ is a type of a fully-metallic functionally graded material that consists of two regions of “pure” material on either side of a region where the two materials are mixed in continuously varying proportions over a known distance. This region has a graded composition that is significantly longer than what is reported in DMWs, by as much as three orders of magnitude. By continuously grading the composition, the sharp changes in both composition and material properties within DMWs are broadened over the length of the grade to mitigate many of the factors that cause premature failure. In the context of the power generation industry, useful GTJs can be fabricated by multipass welding or additive manufacturing processes.

While GTJs present a potentially viable solution to the problems associated with DMWs, the complex microstructural and property gradients introduce challenges to their effective design and implementation. Design of GTJs using finite element analysis (FEA) tools can potentially expedite the process, provided that the inputs to the model are of sufficient quality so as to produce meaningful outputs. Recent studies have investigated the modeling of functionally graded materials using predictive finite element modeling incorporating ‘effective’ material properties. These effective properties are typically estimated using numerical methods such as a simple rule of mixtures, shown in equation 1 where:

\[ P_{eff} = f P_A + (1 - f) P_B \]  

Here, \( P_{eff} \) is the effective property, \( f \) is the volume fraction of material A, and \( P_A \) and \( P_B \) are the properties of material A and B, respectively. Young’s modulus, CTE and Poisson’s ratio are examples of thermal and mechanical properties that are typically estimated in this manner. This technique requires obtaining only the properties for the two materials, rather than measuring properties at a given location in the graded region. FEA tools such as ANSYS® can be used for modeling external forces and thermal behaviors the materials are likely to experience, thus the effectiveness of the graded material in service can be determined.
Even though the properties of the graded region can potentially be estimated with such techniques, experimental information is still needed to validate these models. Furthermore, it would be very difficult to estimate how the properties change during aging. One of the important requirements of a GTJ is the stability of the microstructure and properties at an elevated temperature during the service life of the component. It is therefore essential to understand the microstructural evolution of GTJs during long term exposure.

Brentrup and DuPont\textsuperscript{10} modeled the carbon diffusion of a DMW and GTJ between Grade 22 and Alloy 800H. A longer grade length significantly reduced the concentration gradient and associated CPC gradient and thus, reduced the extent of carbon diffusion. Transition joints were also fabricated by Sridharan \textit{et al.}\textsuperscript{11} using a blown powder additive manufacturing process to join Grade 22 to 316L with SS410 filler. That study attempted to reduce solidification cracking susceptibility of Inconel 82 during fabrication by grading 316L with an intermediate filler metal (SS410). Although they successfully fabricated and characterized a GTJ in the as-welded condition, there is still a need to understand the carbon migration and concomitant microstructural evolution that occurs during aging at high temperatures.

While there is a large body of work on the characterization of DMWs, it is unknown how GTJs will respond to elevated temperatures, and whether it is a suitable replacement for DMWs. The present paper aims to understand the microstructural evolution and localized hardness changes that occur during fabrication and after various aging conditions.

**Experimental Procedure**

A conventional DMW was fabricated between Grade 22 and Alloy 800H using Inconel 82 filler metal. The gas tungsten arc welding (GTAW) process was used with a single V-Joint configuration. This provided a baseline comparison to the fabricated GTJs. The GTJ fabrication processes utilized a dual wire GTAW system equipped with two copper cooling plates to contain the liquid weld pool. A substrate of Grade 22 plate 3” x 12” x $\frac{1}{2}$” (0.076 x 0.305 x 0.013 m) was placed between two copper cooling plates so that the torch and wire feeder assembly deposited metal on the $\frac{1}{2}$” (0.013 m) surface for a maximum travel distance of 12” (0.305 m). The fabrication setup is shown in Figure 1a. The feed rates of each filler metal were varied with every deposited layer to meet the required dilution for each layer, and the desired dilution values were calculated from previous modeling studies.\textsuperscript{12} Ten layers were deposited using a current of 250A, voltage of 12.5V and travel speed of 1 mm/s. The welding direction was rotated every two passes to avoid weld metal build up at the end members. The ten layers corresponded to ~10 mm graded region, as it was previously determined each layer was ~1 mm under the current welding conditions. The wire feed rates combined for a maximum of 50 in/min (21.167 mm/s). An additional three layers of filler metal were deposited so the graded region avoided affects from joining of 800H extensions. The final product was a three inch substrate of Grade 22, ~10 mm graded region, and a three inch 800H extension joined using Inconel 82 filler metal, as shown in Figure 1b. The three filler metals used to fabricate the three GTJs were Inconel 82, P87, and 347H. These candidate filler metals were selected from a wide range of austenitic alloys based on a separate modeling study.\textsuperscript{13} The compositions of the materials used in this study can be seen in Table 1.
Samples from the as-welded conventional DMW and GTJs were sectioned and prepared using standard metallographic techniques to a final polished state using 0.05 μm colloidal silica for characterization. Compositional line traces using energy dispersive spectrometry (EDS) were conducted on the as-fabricated GTJ samples in the as-polished condition using the Leo Gemini® 1550 VP scanning electron microscope equipped with an 80mm² Oxford® X-MAX silicon drift detector operated at an operating voltage of 15 keV with a 60 μm objective aperture. The remaining weld sections were aged at 600°C up to 8,000 hours (approximately one year) for an accelerated aging treatment to simulate the microstructural evolution expected from service conditions. Samples were removed after 2,000, 4,000 and 8,000 hours and prepared for characterization similar to the as-welded samples.

Hardness traces were conducted on the as-welded and aged samples in the as-polished condition using a LECO® LM 248AT Vickers microhardness indenter with a 13 second dwell time with 5 and 10g loads for DMWs and GTJs, respectively. A two percent Nital solution was used to reveal grain boundaries and carbides in the ferritic material, and a ten percent oxalic acid solution was used to reveal grain boundaries and carbides in the austenitic structures by electrolytic etching at 2V for 5-15 seconds, depending on filler metal.

Secondary electron imaging and X-ray EDS were performed in a Hitachi® 4300SE/N Schottky field emission scanning electron microscope at an operating voltage of 15 keV. Compositions measured from this analysis are standardless and therefore, semi-quantitative. However, the accuracy of this technique was evaluated using commercial alloy “standards” of known chemistry measured by optical emission spectroscopy (OES). Table 2 shows the results from EDS alongside the OES results. The EDS measurements show reasonably good agreement with the OES measurements. Therefore, this measurement technique was sufficient enough to identify regions of local enrichment. Monte Carlo simulations using the CASINO® program demonstrated that the X-ray generation volume was approximately 1 μm³ under the operating conditions. Thus, results of sub-micron size particles were likely affected by the surrounding matrix material but were still useful for identifying the formation of expected phases. Electron back scattered diffraction (EBSD) measurements were performed at an accelerating voltage of 20 keV and a step size of 0.2 microns across the welded interfaces. Image quality and phase maps were recorded to aid in microstructure characterization.

Thermodynamic and kinetic modeling was conducted on the conventional DMW and the three GTJs. Simulated aging times and temperatures were selected to match the accelerated aging treatment at 600°C. Aging simulations were performed using the DICTRA® software package in conjunction with the MOB2 mobility database. The TCFE7 and NI-DATA-V7 databases were used for high iron materials and nickel based alloys, respectively. The following phases were allowed to form based on relevant literature for each material: Austenite, ferrite, M23C6, M6C, and FCC2 (MC carbide phase). The length of the graded region was set as 50 μm for DMW calculations. This is a typical distance of the PMZ in a DWM. Composition profiles for the GTJs were chosen from the EDS line trace results utilizing a step function concentration gradient, with individual layer compositions averaged from experimental measurements. Carbon diffusion and phase fraction profiles were extracted from the kinetic simulations for both the DMW and GTJ and were compared to the observations from characterized samples.
Additionally, non-equilibrium (Scheil) solidification calculations were performed for compositions representative of select regions of the GTJs using Thermo-Calc software and the TTNi7 thermodynamic database to assess the compositional gradients during weld metal solidification. EDS line scans were acquired across the cellular substructure in the as-welded condition to directly compare to the Scheil simulations.

**Results**

*Microhardness Trends*

**As-welded condition**

Microhardness gradients in the as-welded condition for the conventional DMW and fabricated GTJs using Inconel 82, P87 and 347H are shown in Figure 2a-d, respectively. The microhardness and composition are plotted as a function of distance across the PMZ in the DMW, and graded region in the GTJ. Due to the difficulties of accurately measuring carbon, the carbon concentration gradient was estimated from knowledge of the concentration of major elements (measured by EDS) in the manner previously described by Kusko and DuPont.\(^2^4\) Using the measured values of manganese (Mn), nickel (Ni), chromium (Cr), and molybdenum (Mo), along with estimated values of carbon (C), the variation in martensite start (\(M_s\)) temperature across the PMZ and graded regions was also determined and shown in Figure 2. This is useful to determine the region where the \(M_s\) temperature is below room temperature (\(T_{RM}\)), which indicated the material will be austenitic. The expression used for this calculation was provided by Andrews\(^2^5\) and shown by equation (2):

\[
M_s(°C) = 539 - 423C - 30.4Mn - 17.7Ni - 12.1Cr - 7.5Mo
\]

where the composition is in weight percent. The conventional DMW (Figure 2a) shows a steep concentration gradient for the three major alloying elements Fe, Ni, and Cr. As expected, the \(M_s\) temperature also exhibits a steep gradient across the narrow PMZ. There is a high hardness region in the heat affected zone (HAZ) of Grade 22 prior to the fusion line, with a significant reduction in hardness from 400 HV to 200 HV across the PMZ and into the Inconel 82 weld metal.

The spatial variation in composition, hardness, and \(M_s\) temperature are shown for Grade 22/Inconel 82 GTJ in Figure 2b. The difference in length scales between Figure 2a and 2b should be noted, as the PMZ of the DMW is on the order of microns, whereas the graded region of the GTJ is on the order of millimeters. The primary goal of GTJs is to eliminate the sharp gradients in composition and associated properties. Although the GTJs do exhibit changes in hardness, these changes occur over much greater distances when compared to the conventional DMW. The composition changes gradually throughout the graded region, and is in the form of a “step-function”, as each layer consists of a constant composition. The base metal hardness starts around 200 HV, and increases throughout the HAZ and into the graded region. The hardness increases throughout the graded region and then decreases slightly in Layer 4. Layer 4 exhibits a larger reduction in hardness from 400 HV to 200 HV. (Note: layers will be labeled “L” followed by the layer number, i.e. layer 1 is L1.) Beyond L4, the hardness remains essentially constant until the end of the graded region, and experiences a slight increase as it transitions to the Inconel 82 weld metal.
The P87 and 347H GTJs (Figure 2c-d) follows similar trends to that of Inconel 82. The region of mixed hardness in the P87 GTJ is L6 (opposed to L4 in Inconel 82) and in L10 for the 347H GTJ. Furthermore, the concentration gradient is less severe in the 347H GTJ than the other two filler metals because 347H is an iron-base filler metal, whereas Inconel 82 and P87 are nickel-base materials.

**Aged Condition**

The microhardness gradients in the aged conditions for the conventional DMW and GTJs are shown in Figure 3a-d. The conventional DMW in Figure 3a is relatively soft prior to the fusion line for the aged conditions, and the hardness peaks in the Inconel 82 weld metal. This can be directly compared to the microhardness gradients shown in Figure 3b for the Inconel 82 GTJ. The Inconel 82 GTJ also exhibits a reduction in hardness in the HAZ from the as-welded condition, but the extent of softening near the HAZ/L1 interface is significantly reduced. For all aged conditions, there is an overall decrease in hardness relative to the as-welded condition in the Grade 22 region, followed by an increase in hardness in the first several layers of the graded region. Layer 5 contains mixed hardness, followed by a reduction slightly below ~ 200 HV in the remaining graded region for the 2,000 and 4,000 hour aged conditions. After aging for 8,000 hours, the hardness is higher (in L6 through the Inconel 82 weld metal) than the 2,000 hour aged condition. Similar trends are shown in the P87 GTJ (Figure 3c), except the hardness does not increase after 8,000 hours aging, and the mixed hardness layer is L6 (opposed to L5 in the Inconel 82 GTJ). The 347H GTJ (Figure 3d) also exhibits similar hardness trends, only over a larger region, and does not exhibit the sharp decrease in hardness like Inconel 82 and P87 in L6 and L7, respectively. Instead, the reduction in hardness occurs beyond L10 (~x=25mm), and increases after aging.

**Thermodynamic and Kinetic Modeling**

The calculated carbon concentration and phase fraction as a function of distance at the simulated aging temperature of 600°C for up to 8,000 hours is shown for the conventional DMW (Figure 4a-b) and the Inconel 82 GTJ (Figure 4c-d), respectively. (Note the large difference in both concentration and distance scales between the DMW and GTJ). The conventional DMW shows a carbon depleted region prior to the fusion line (x=10mm) and a carbon enriched region in the PMZ for all aging conditions. The phase fraction at 600°C in Figure 4b shows three stable carbides, a molybdenum rich carbide (M₆C), chromium rich carbide (M₂₃C₆) and niobium rich carbide (NbC). The NbC is stable in Inconel 82 due to the higher Nb content in the filler metal. There is a rather sharp transition from BCC to FCC, which is attributed to the steep concentration gradients between the two materials. These figures can be directly compared to the GTJ between Grade 22 and Inconel 82 showing the carbon concentration and phase fraction in Figure 4c-d, respectively. The initial carbon concentration profile (Figure 4c) exhibits a slight, local reduction at the interfaces associated with the change in composition of each layer. After 8,000 hours, the extent of carbon depletion and enrichment increases at the first four layer interfaces. This is insightful, as it demonstrates the location of significant microstructural evolution – in the beginning of the grade, and at the layer interfaces. While carbon diffusion occurs in the GTJ, the extent of the enriched regions are less than 0.17 weight percent C, as opposed to 0.70 weight percent C in the DMW PMZ. This is expected,
as an increase in grade length and corresponding decrease in the CPC gradient (discussed below) results in a lower driving force for carbon diffusion.

Localized variations in the BCC and FCC phase distribution occur through the first five layers of the graded region (Figure 4d). The \( \text{M}_{23}\text{C}_6 \) carbide fraction follows the trend of the carbon concentration. This is expected, as it was previously demonstrated that most of the carbon in the system resides primarily in the carbides.\(^{13}\) Similar to the DMW, the Nb carbide is stable throughout the FCC range until the end of the graded region and also follows the carbon concentration behavior once the \( \text{M}_{23}\text{C}_6 \) carbide is no longer stable.

The carbon concentration and phase fraction as a function of distance is shown for the P87 GTJ (Figure 5a-b) and the 347H GTJ (Figure 5c-d), respectively. Similar trends are observed for these GTJs. Carbon diffusion occurs at the layer interfaces, and the extent of diffusion increases with increased aging time. A major difference between these filler metals and Inconel 82 is that the carbon depletion and enrichment at the layer interfaces continues further into the graded region (Figure 5a and c). The phase fraction for P87 GTJ (Figure 5b) shows that all three carbides are stable throughout the graded region. The phase fraction for the 347H GTJ shows a gradual transition (in the form of a step function) from BCC to FCC, attributed to the lower concentration gradient as compared to the Ni-base filler metals. Additionally, the \( \text{M}_{23}\text{C}_6 \) phase fraction shows significant depletion and enrichment at the layer interfaces, with NbC stable throughout the entire 347H graded region.

The carbon concentration is lower in Grade 22 than in P87, resulting in a positive concentration gradient in the P87 graded region (Figure 5a). In a binary system, carbon will diffuse from high to low concentrations.\(^{26,27}\) However, in a multicomponent system, the CPC gradient is the driving force for carbon diffusion. The CPC gradient is shown in the as-welded and aged conditions for the conventional DMW, Inconel 82, P87, and 347H GTJs in Figure 6a-d, respectively. In the as-welded condition, the conventional DMW has a steep CPC gradient after the fusion line that drives the carbon to diffuse from Grade 22, down the CPC gradient, and into the PMZ. In the aged condition, the CPC discontinuity that existed at the fusion line in the as-welded condition is alleviated due to carbon diffusion. The three GTJs show a step function in the negative CPC gradient for all conditions. Even though the P87 GTJ has a positive carbon concentration gradient, it exhibits a negative CPC gradient across the graded region, as shown in Figure 6c. This results in carbon to diffuse up the concentration gradient, a phenomenon known as “uphill” diffusion.\(^{7,8,26,28,29}\)

**Microstructure Characterization**

Photomicrographs were acquired in the Inconel 82 GTJ aged for 4,000 hours at locations of significant microstructural evolution seen from the hardness and modeling results shown in Figure 7. The HAZ directly adjacent to the interface, and the beginning of L1 is shown in Figure 7a-b, respectively. The HAZ was originally martensitic in the as-welded condition. Upon aging, carbon diffusion resulted in a microstructure depleted of carbides, as shown in Figure 7a. The microstructure in L1 adjacent to the HAZ/L1 interfaces is comprised of fine lath martensite populated by carbides, which were determined to be Cr rich. Figure 7c shows a microstructure at the end of L1 that is somewhat similar to Figure 7, however the population of carbides is reduced and a coarser, lath martensite is observed. Finally, the microstructure in L2 (Figure 7d) shows in increase
in carbide content, and the formation of a secondary constituent. Identical microstructural trends were observed in GTJs made with P87 and 347H filler metal.

Figure 8a-b shows EBSD phase maps overlaid with image quality maps of the layer interfaces where the BCC/FCC transition occurs in GTJs using Inconel 82 and P87, respectively. (EBSD results shown here do not differentiate between BCC ferrite, BCC bainite, and BCT martensite. Thus, for simplicity, the phase map is shown for BCC ferrite and FCC austenite.) All samples were examined in the 2,000 hour aged condition. The phase maps are shown for the L4/L5 and L5/L6 interfaces for the Inconel 82 GTJ. Layer 4 consists of all BCC, and L5 shows the initial formation of austenite. Beyond the L5/L6 interface, only FCC is stable. Similar results are shown in Figure 8b for the P87 GTJ, except the interfaces between L6/L7 and L7/L8 were investigated. Layer 7 represents the region of mixed BCC/FCC, with L8 consisting of all FCC. Figure 8c represents four regions throughout the 347H GTJ where the martensite/austenite transition occurs. Layer 9 consists of all BCC, and L10 has a small amount of FCC in a BCC matrix. Layers 11 and 12 exhibit increasing amounts of the FCC phase. Beyond L12, Inconel 82 was joined to the 800H extension and exhibited all FCC (as confirmed with EBSD).

Layer 7 in the as-welded P87 GTJ was analyzed to understand the cause for the mixed BCC/FCC microstructure. Figure 9a shows a light optical image superimposed on an SEM photomicrograph of L7 with hardness indents used as markers for regions of interest. The dark and light regions, representing cell core and intercellular regions, correspond to the BCC and FCC austenite phases, respectively. Additionally, an EDS line scan (shown by the black arrow in Figure 9a) was conducted across the region to understand the elemental distribution that could be attributed to the stability of the FCC phase. The results of the EDS line scan are presented in Figure 9b showing Fe, Cr, Ni, C, and Mo (other elements were analyzed but not included). The analysis of carbon was calculated by considering a constant carbon concentration of L7 (0.091 weight percent). Next, the EDS trace was subjected to a simulated weld thermal cycle from DICTRA. Due to the alloying element segregation and corresponding CPC gradient, the carbon will diffuse to the cell core, as shown in Figure 9b. The trace initiated in an intercellular region that is depleted in Fe, and enriched in the remaining measured elements. Conversely, the cell core is enriched in Fe and depleted in the remaining elements. Figure 9c-d show results from the solidification simulation for this layer composition. For this composition profile shown in Figure 9d, the cell core is the initial solidification region (far left on figure), with the intercellular region solidifying last. The calculated distribution of the elements shown is in good agreement with the measured elements, as Fe is depleted in the intercellular region when the other elements become enriched.

**Evaluation of Ni-rich Constituent**

Briefly mentioned above (in Figure 7d) was the formation of a secondary constituent observed in all three GTJs. An example of this is shown in Figure 10a-d that represents L2 through L5 in the P87 GTJ aged for 8,000 hours, respectively. The constituent is in the form of elongated particles in L2 and L3 (Figure 10a-b) that increases in volume fraction with increasing layer. The calculated phase fraction as a function of temperature for the P87 GTJ is shown in Figure 11a-d for L2-L4, respectively. Paying specific attention to 600°C, the austenite that forms during aging increases in phase fraction with increasing layer, going from 12 percent FCC in L2 (Figure 11a) to 76 percent FCC in
L5 (Figure 11d). The austenite that exists at 600°C will also transform to martensite without a change in composition when cooled to $T_{RM}$, thus accounting for the presence of this martensite constituent within the GTJs after aging.30 Also labeled on the plots are the “Ac3” temperatures, which are designated as the location where BCC completely dissolves upon heating. As shown, increasing layers exhibit progressively lower Ac3 temperatures, ranging from 734°C in L2 (Figure 11a) to 625°C in L5 (Figure 11d). It must be recognized that these are under equilibrium conditions. The heating rates experienced during welding are significantly faster than equilibrium and therefore, it is expected for these temperatures to be higher.31 Additionally, the accuracy of ThermoCalc at lower temperatures is limited due to the insufficient low temperature data available from the databases used for the models. Thus, the trends are more important than the actual Ac3 values. This will be discussed further in the next section. Figure 12a shows the calculated composition of the FCC phase at 600°C directly compared to the measured composition of the martensite constituent. This constituent is Ni rich at $T_{RM}$ and is in very good agreement with the calculated composition of the austenite phase at 600°C. Similarly, Figure 12b shows very good agreement between the measured matrix and calculated BCC phase compositions.

The morphology of the Ni rich martensite was different between filler metals. This is shown in Figure 13 where the Ni-rich martensite is observed in the 347H GTJ. The Ni-rich martensite has a different morphology than that observed in the GTJs using Inconel 82 and P87 (see Figure 7d and Figure 10b for comparison). The martensite morphology in Inconel 82 and P87 GTJs is rod-like, whereas the martensite in the 347H GTJ exhibits a blocky morphology.

Discussion

As-Welded Microstructures

The analysis of the as-welded microstructures is summarized in Figure 2 with the combination of hardness, composition, and $M_s$ temperature. The conventional DMW (Figure 2a) shows a high hardness in the HAZ. This is attributed to martensite formation during fabrication from the high cooling rates associated with welding. Directly after the HAZ, the GTJs exhibit increased hardness with each layer (i.e., before the fully austenitic region is reached). This hardness variation is associated with several factors that are all related to the variation in composition, including changes in both the $M_s$ and Ac3 temperatures, along with solid solution strengthening. The increase in alloying elements along the grade suppresses the $M_s$ temperature (Figure 2b-d). If martensite forms at an elevated temperature (i.e., the $M_s$ temperature is relatively high), then significant auto-tempering can occur during cooling to $T_{RM}$. Therefore, the layers with higher $M_s$ temperatures will experience a larger degree of auto-tempering, thus lowering the hardness. This explains, in part, the lower hardness values observed near the beginning of the grade where the $M_s$ temperature is relatively high.

As demonstrated in Figure 11, the Ac3 temperature will also decrease with each additional layer within the graded region (due to the increased alloy content). The relatively high alloy content within the layers provides high hardenability that, combined with the fast cooling rates from welding, will produce martensite during cooling for any region heated above the Ac3 temperature. Since the Ac3 temperature decreases along the graded region, the amount of material exposed to peak temperatures above Ac3 increases with each additional layer, thus leading to more as-quenched martensite. This effect also partly
accounts for the increased hardness along the length of the grade. The increased hardness in the first several layers can also partly be attributed to solid solution strengthening, as it is known that Ni and Cr increase the strength of martensite.

After the initial increase in hardness within the first several layers of the graded regions in the GTJs, there is a significant hardness decrease (at locations of x=15, 19, and 24 mm for Inconel 82, P87 and 347H GTJs, respectively). Figure 8 shows that this hardness reduction is associated with austenite formation. The austenite phase stability can primarily be attributed to the increase in Ni concentration along the grade that eventually drives the $M_s$ temperature below $T_{RM}$, thus stabilizing austenite. Formation of the mixed martensite/austenite regions can be understood with reference to solidification simulations, shown in Figure 9. Austenite (FCC) and carbides form during solidification (Figure 9c), and the segregation of the alloying elements plays a major role in the microstructure that forms upon cooling to $T_{RM}$. Cr, Ni, and Mo segregate to the intercellular regions (light regions in Figure 9a). Equation (2) can be used to estimate the variation in $M_s$ temperature with position along the cellular substructure due to the microsegregation. The $M_s$ temperature varies above and below $T_{RM}$ (shown by the blue horizontal line) in the cell core and intercellular regions, respectively. This is consistent with the phases present from EBSD analysis in L7 of the P87 GTJ where austenite is stable in the intercellular regions, which is attributed to the $M_s$ temperature suppressed below $T_{RM}$.

**Simulated Results**

Some of the simulation results provide particular insight into microstructural evolution during aging (discussed below). Note that the $M_{23}C_6$ carbide stability follows the carbon concentration profiles (Figure 4c-d for Inconel 82, Figure 5a-b for P87, and Figure 5c-d for 347H). The carbon concentration depletes prior to every interface, and enriches into the subsequent layer. This is controlled by the CPC gradient (Figure 6), as carbon will diffuse down the gradient to alleviate the CPC discontinuity at the interface. Nearly all the carbon resides in carbides. Therefore, carbon must diffuse into the matrix in order to diffuse. Of the three carbides in the system ($M_{23}C_6$, $M_6C$, and NbC), the $M_{23}C_6$ carbide is the least stable. $M_{23}C_6$ is considered a low temperature carbide due to its complex structure and lower dissolution temperature, which is confirmed in the equilibrium calculations shown in Figure 11. For these reasons, this carbide will dissolve before the $M_6C$ and NbC carbides.

In the Inconel 82 GTJ, there is negligible diffusion at the layer interfaces after L4 (Figure 4c), which is coincident with the $M_{23}C_6$ carbide dissolution and NbC stability (Figure 4d). Additionally, austenite becomes the dominant phase in this region. Carbon can diffuse faster through BCC, thus the FCC matrix after L4 limits the diffusion of carbon in the matrix. Furthermore, the carbon resides in the Nb carbide (since the $M_{23}C_6$ carbide dissolved), which also shows negligible enrichment or depletion at the layer interfaces. Even though chromium is relatively high in Inconel 82, the increase in Nb concentration across the graded region stabilizes NbC over the $M_{23}C_6$ carbide at 600°C. It is interesting to note that even the sharp discontinuities in CPC at the interfaces are not alleviated after aging up to 8,000 hours. This effect may be associated with carbon being tied up in the highly stable NbC. Similar trends are shown in the other two filler metals.
**Aged Microstructures**

The modeling results described above can be combined with experimental observations to explain the hardness trends observed in Figure 3a-d. The conventional DMW exhibits a reduction in hardness in the HAZ that is attributed to tempering of the martensitic microstructure during aging. Additionally, there is a localized reduction in hardness directly adjacent to the fusion line that can be attributed to carbon diffusion, which results in a carbon depleted region in the HAZ of Grade 22, and a carbon enriched region in the PMZ. This is consistent with the carbon diffusion calculation (Figure 4a) that shows the same carbon depleted and enriched regions in the HAZ and PMZ, respectively. The carbon diffusion is attributed to the negative chemical potential gradient from the Grade 22 to the Inconel 82. Carbon will eventually exceed the solubility limit in the PMZ, resulting in $M_{23}C_6$ formation, as shown in the phase fraction of carbides in Figure 4b. A local increase in carbide content will increase the hardness, and this is consistent with the hardness trace shown in Figure 3a.

The GTJs experience the same trend as the DMW regarding the tempering of the martensitic microstructure in the HAZ, which contributes to the observed reduction in hardness. Additionally, there is an overall reduction in hardness in the martensitic layers of the graded region for all three filler metals, prior to the austenite formation. This is also attributed to the tempering of the martensitic microstructure during aging. Furthermore, enhanced carbon diffusion occurs in the GTJs at the layer interfaces, as shown in Figure 4c and Figure 5(a and c). This is consistent with the photomicrographs in Figure 7 that show the carbides in the regions of significant hardness changes in the beginning layers, specifically in the Inconel 82 GTJ. All three filler metals exhibit a hardness decrease in the HAZ adjacent to the HAZ/L1 interface, and an increase at the beginning of L1. The carbon (and corresponding carbide) calculation also exhibits depletion and enrichment in these regions, shown by the calculated carbon and $M_{23}C_6$ carbide in Figure 4c-d. The microstructural features in these images presented in Figure 7 are consistent with the $M_{23}C_6$ carbide trend, and account for the hardness decrease in the HAZ, and the increase into L1 (Figure 3b). Additionally, a decrease in hardness across L1 is shown for Inconel 82 and P87 in all aged conditions, and observed in 347H after 8,000 hours. This can also be attributed to variation in $M_{23}C_6$ content shown in the model, and validated by the observations in the photomicrographs represented by the Inconel 82 GTJ in Figure 7b-c. In addition, the lath martensite is relatively fine in the beginning of L1 and coarser at the end of L1, which can also contribute to the hardness decrease across this layer. The combination of reduced carbides and coarser lath martensite accounts for the observed decrease in hardness across the layer. As described previously, although carbon diffusion and the associated microstructural changes are somewhat accelerated near the interfaces of the GTJs, they are significantly reduced relative to the conventional DMW. The P87 GTJ exhibits similar trends. In this case, L6 exhibits decreased hardness (relative to L5) that is associated with a mixed martensite/austenite structure.

The increase in hardness observed in the Inconel 82 GTJ after 8,000 hours and the 347H GTJ after L10 after aging for 2,000 hours can likely be attributed to precipitation that is known to occur in these alloys. Alloy 347H forms Nb carbides during aging. Hajiannia et al. revealed that fine intercellular Nb carbide precipitates increased the hardness of the welded 347H after aging. This is consistent with the results of Minami et al. who used transmission electron microscopy to show the presence of fine nanometer...
scale Nb carbides in the same alloy. The presence of the fine Nb carbides also contribute to the increase in hardness in the Inconel 82 GTJ. Klueh and King\textsuperscript{42} investigated the thermal aging behavior of Inconel 82 after aging up to 10,000 hours at various temperatures. They also observed a precipitate that developed in the matrix that was associated with the cellular-dendritic substructure and the concomitant solute rejection from the solidification process. Since Inconel 82 has a Nb content of 2.36 weight percent, it is expected Nb carbides will form and provide an overall increase in strength. The presence of Nb carbides in both materials are consistent with the calculated carbide phase fraction from the simulations that show the stability of NbC in the graded region.

\textit{Ni-Rich Martensite}

The Ni-rich martensite does not form in the as-welded condition, but forms in all the GTJs after aging at 600°C due to austenite stability at this temperature. Austenite stability was confirmed in Figure 11 that shows thermodynamic equilibrium plots for a given layer. With increasing layer, the amount of FCC phase increases and, upon cooling, transforms to martensite. This is confirmed with the photomicrographs in Figure 10. Since the martensite transformation is diffusionless, the as-quenched martensite has the same composition of the parent austenite, most notably Ni.\textsuperscript{30} This is confirmed with EDS results showing that the martensite is rich in Ni for the FCC phase (Figure 12a) as compared to the Ni in the BCC phase (Figure 12b). This is also confirmed from the EBSD results that show BCC is the stable phase until L7 (P87 GTJ), L5 (Inconel 82 GTJ), and L10 (347H GTJ). The lack of Ni-rich martensite in the as-welded condition is associated with the time necessary for austenite formation at 600°C. Figure 14a provides simulation results for non-equilibrium solidification of L3 for the Inconel 82 GTJ that shows FCC and carbides are expected to form during solidification. Due to the high cooling rates from the weld thermal cycles (and the high hardenability of this layer), the austenite transforms to martensite during cooling. The subsequent thermal cycles do not allow enough time for the FCC phase to form at an elevated temperature. This is demonstrated in Figure 14b that shows the phase fraction as a function of time, aging from 400°C to 600°C. Since low temperature kinetic data is limited, the Inconel 82 GTJ L3 (as an example) was simulated at a temperature of 400°C and then heated to 600°C. It takes almost 20 hours for the BCC and FCC phases to stabilize to the 600°C equilibrium phase fraction values. The sluggish transformation kinetics can be attributed to the low diffusivity of Ni in austenite. The inability to form Ni-rich austenite during the short thermal cycles associated with welding accounts for the lack of Ni-rich martensite in the as-welded condition.

Although the hardness and amount of Ni-rich martensite each increase along the grade, the increase in hardness cannot be attributed to the presence of the Ni-rich martensite. The hardness of martensite is largely affected by carbon concentration. The Ni-rich martensite could potentially have relatively high carbon (due to the high solubility of carbon in austenite). However, under these conditions, nearly all the carbon is tied up in the carbides.\textsuperscript{13} This is confirmed from thermodynamic calculations, which demonstrate that the carbon in the FCC and BCC phases at 600°C was less than 1e\textsuperscript{-5} weight percent carbon, and the rest of the carbon was in the carbides. Additionally, nanohardness measurements conducted on each phase showed that the BCC matrix hardness was 4.84±0.77 GPa, while the Ni-rich martensite had a hardness of 5.06±0.43 GPa. Thus, there is no significant difference in hardness between these two phases. Finally, note that the hardness increases
along the grade in the as-welded condition when no Ni-rich martensite is present, which is also consistent with the above observation.

The formation of a similar Ni-rich martensite has been reported in other studies.\textsuperscript{43–46} The martensite morphology is largely effected by the initial microstructure prior to aging. When the sample is heated to 600°C, there will be regions where this temperature is above the Ac1 temperature (defined as the initiation of austenite transformation upon heating) and thus, austenite is stable. The initiation sites for the parent austenite formation are the martensite laths or prior austenite grain boundaries (PAGB).\textsuperscript{43,44,47,48} The austenite that forms at the PAGB and/or lath boundaries will transform to a Ni-rich martensite, and take the form of rod-like morphology or blocky morphology, also described in the literature as “islands”.\textsuperscript{49} The blocky Ni-rich martensite forms mostly at the PAGB, while the rod-like martensite forms on the martensite laths.\textsuperscript{47,48,50} A blocky morphology is clearly observed in Figure 13 for the 347H GTJ. Conversely, the elongated morphology of the Ni-rich martensite can be seen in Figure 7d and Figure 10b for Inconel 82 and P87, respectively.

Work is in progress to understand the tensile and creep properties of these GTJs relative to conventional DMWs and will be reported in separate articles.

**Conclusions**

Microstructural characterization was conducted on three GTJs in the as-welded and aged conditions. Thermodynamic and kinetic modeling of the joints was compared to the experimental results. The following conclusions can be drawn from this work.

1. The graded regions of the GTJs in the as-welded condition exhibit martensite in the first few layers of the grade, followed by a mixed martensite/austenite region, and then a fully austenitic region in the final layers.
2. The austenite stability in the final layers is attributed to the increase in Ni concentration that reduces the martensite start temperature below room temperature, thus stabilizing austenite. The preceding layers of mixed FCC/BCC are attributed to element segregation upon solidification that causes the martensite start temperature to vary above and below room temperature.
3. The increase in hardness within the martensitic region of the grade in the as-welded condition is associated with a decrease in the Ms temperature (less auto-tempering), a decrease in the Ac3 temperature (more as-quenched martensite), and solid solution strengthening. The subsequent decrease in hardness is associated with the austenite stability in the final layers.
4. Enhanced carbon diffusion occurs at the layer interfaces during aging, and is more significant in the beginning layers of the graded region. Although the carbon is tied up in carbides, the M\textsubscript{23}C\textsubscript{6} carbide will dissolve, allowing carbon to diffuse due to the chemical potential gradient that is steepest in the beginning of the grade.
5. Hardness gradients between the layers can be attributed to the distribution of carbides throughout the layers. Carbon/carbide depleted and enriched regions form at the end and beginning of a layer, respectively. This coincides with the hardness variation, as the overall hardness was observed to be higher at the beginning of each layer.
6. During aging at 600°C, both FCC and BCC phases are stable in the first several layers of the grade. The amount of Ni-rich FCC (austenite) increases as the Ni concentration increases throughout the grade. The Ni-rich austenite transforms to martensite with no change in composition during cooling.
References
16. Thermo-Calc Software MOB2 TCS Alloy Mobility Database.
18. Thermo-Calc Software Ni-Data-v7 Ni-Alloys Database.
44. Isheim D, Hunter AH, Zhang XJ, Seidman DN. Nanoscale Analyses of High-


Figures

Figure 1. a) Image of the GTJ fabrication set up and b) schematic representation of the final GTJ product.
Figure 2. Composition, hardness, and martensite start temperature as a function of distance for a) DMW and GTJs between Grade 22 and b) Inconel 82, c) P87, and d) 347H.
Figure 3. Hardness as a function of distance for a) DMW and GTJs between Grade 22 and b) Inconel 82, c) P87, and d) 347H for the as-welded and aged conditions.
Figure 4. Calculated carbon concentration and phase fraction as a function of distance at the simulated aging temperature of 600°C for 8,000 hours for (a-b) conventional DMW and (c-d) the Inconel 82 GTJ.
Figure 5. The carbon concentration and phase fraction as a function of distance at the simulated aging temperature of 600°C for 8,000 hours for (a-b) the P87 GTJ and (c-d) the 347H GTJ.
Figure 6. The chemical potential of carbon at 600°C in the as-welded and aged conditions for a) conventional DMW, b) Inconel 82 GTJ, c) P87 GTJ, and d) 347H GTJ.
Figure 7. SEM photomicrographs of the Inconel 82 GTJ aged for 4,000 hours showing a) Grade 22 substrate HAZ, b) beginning of L1, c) end of L1, and d) beginning of L2.
Figure 8. EBSD phase maps overlaid with image quality maps showing the transition to austenite for the GTJ using a) Inconel 82, b) P87, and c) 347H. (Note micron bar in L9 is for all four layers).
Figure 9. a) LOM image superimposed over the SEM image showing the location of the EDS scan in L7 of the P87 GTJ, b) the results from the EDS scan with the $M_s$ temperature, and c) Scheil solidification simulation of L7 showing the phases that form, and d) element distribution of L7 in FCC during solidification.
Figure 10. SEM photomicrographs of the P87 GTJ showing layers 2-5 aged for 8,000 hours showing the extent of the Ni-rich martensite formation.
Figure 11. Calculated phase fraction as a function of temperatures for in a) L2, b) L3, c) L4, and d) L5 for the P87 GTJ for a simulated age at 600°C.
Figure 12. The measured and calculated compositions as a function of layers for the austenite phase in the P87 GTJ.
Figure 13. SEM Photomicrograph of L6 in the 347H GTJ aged for 8,000 hours showing the Ni-rich martensite for a) low and b) high magnifications.
Figure 14. a) Scheil solidification model for the Inconel 82 GTJ L3 showing the phases that form upon solidification, and b) phase fraction and aging temperature as a function of time after reaching 600°C that shows how long it takes for the BCC and FCC phases to stabilize in L3 of the Inconel 82 GTJ.
### Tables

**Table 1.** Chemical compositions of the Grade 22 plate and four welding wires, and inputs to the thermodynamic and kinetic models.

<table>
<thead>
<tr>
<th>Material</th>
<th>Al</th>
<th>C</th>
<th>Cr</th>
<th>Cu</th>
<th>Mn</th>
<th>Mo</th>
<th>Nb</th>
<th>Ni</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Ti</th>
<th>Fe</th>
</tr>
</thead>
</table>
| Grade 22 Substrate     | 0.03| 0.07| 2.20| 0.14| 0.52| 0.92| <0.01| 0.10 | 0.007 | 0.009 | 0.20 | <0.01 | Bal.
| Inconel 82             |    | 0.027| 21.4| 0.016| 3.20|     | 2.36| 70.8| 0.003 | <0.001 | 0.19 | 0.43 | Bal.
| EPRI P87               | 0.18| 0.11| 8.99|     | 1.53| 1.93| 1.08| Bal. | 0.004 | 0.001 | 0.22 | 0.10 | 40.4 |
| 347H                   |    | 0.05| 19.4| 0.15| 1.75| 0.29| 0.68| 9.07| 0.02  | 0.01  | 0.44 |     |     |
| 90S-B3 (Grade 22 Wire) | 0.01| 0.07| 2.45| 0.16| 0.60| 0.93| <0.01| 0.13 | <0.005 | 0.005 | 0.58 |     |     |

**Table 2.** Average EDS measurements compared to OES results for the custom standards.

<table>
<thead>
<tr>
<th>Standards</th>
<th>Si</th>
<th>Cr</th>
<th>Mn</th>
<th>Fe</th>
<th>Co</th>
<th>Ni</th>
<th>Cu</th>
<th>Nb</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alloy 800</td>
<td>Ave</td>
<td>0.33</td>
<td>22.4</td>
<td>0.95</td>
<td>44.6</td>
<td>0.21</td>
<td>31.9</td>
<td>0.15</td>
<td>N/A</td>
</tr>
<tr>
<td></td>
<td>OES</td>
<td>0.44</td>
<td>20.9</td>
<td>1.03</td>
<td>41.1</td>
<td>0.07</td>
<td>34.7</td>
<td>0.27</td>
<td>0.15</td>
</tr>
<tr>
<td>347</td>
<td>Ave</td>
<td>0.72</td>
<td>18.2</td>
<td>1.81</td>
<td>67.8</td>
<td>0.29</td>
<td>9.51</td>
<td>0.38</td>
<td>0.76</td>
</tr>
<tr>
<td></td>
<td>OES</td>
<td>0.55</td>
<td>17.4</td>
<td>1.67</td>
<td>69.9</td>
<td>N/A</td>
<td>9.60</td>
<td>0.47</td>
<td>N/A</td>
</tr>
<tr>
<td>T22</td>
<td>Ave</td>
<td>0.49</td>
<td>2.37</td>
<td>0.57</td>
<td>94.8</td>
<td>0.21</td>
<td>0.07</td>
<td>0.03</td>
<td>N/A</td>
</tr>
<tr>
<td></td>
<td>OES</td>
<td>0.38</td>
<td>2.19</td>
<td>0.50</td>
<td>Bal</td>
<td>N/A</td>
<td>0.06</td>
<td>0.09</td>
<td>N/A</td>
</tr>
</tbody>
</table>
3. Study of heterogeneous creep deformation in Dissimilar Metal Welds (DMWs)

Abstract
Dissimilar Metal Welds (DMWs) made between ferritic low alloy steels and austenitic alloys are widely used in high temperature components of nuclear and fossil energy power plants. Ex-service data demonstrates that these bimetallic welds experience premature creep failure in the ferritic steel component close to the interface of the ferritic base material and austenitic weld deposit with lifetimes much less than the designed creep lives of either of the base metal components. Previous creep studies on these DMW configurations have shown that creep cavities are associated with a network of Type I interfacial carbides that develop on the ferritic steel side close to ferrite/austenite boundary during prolonged high temperature exposure. The main objective of the current investigation is to extract local creep constitutive properties in these DMWs and rationalize them with underlying microstructural evolution during prolonged high temperature exposure. In the present research study, a DMW coupon was fabricated between 2.25Cr-1Mo steel and Alloy 800H base material using a Ni base Inconel weld consumable. In addition, the as-welded DMW coupon was aged at 600°C for 2000h and 4000h to induce the presence of two different sizes and distributions of creep detrimental Type I interfacial carbides. Short term (~1 month) creep tests were integrated with Digital Image Correlation (DIC) technique to extract local creep deformation data along the entire gauge section of these aged DMW specimens. In both the 2000h and 4000h aged DMW specimens, creep strain concentration occurred in the ferritic steel portion. In the 2000h aged DMW specimen, the location of strain concentration was observed close (~5μm) to ferrite/austenite boundary. However, in the 4000h aged DMW specimen, it was observed away (~500μm) from the ferrite/austenite boundary. Detailed microstructural investigation on the crept specimens revealed: (i) in the 2000h aged DMW specimen, creep strain concentration occurred in the local region close (~5μm) to ferrite/austenite due to the depletion of alloying element: Mo in the ferritic matrix surrounding Type I interfacial carbides, which is an indication that this local region in transition is tempered faster than the 2.25Cr-1Mo base material (ii) in the 4000h aged DMW specimen, creep strain concentration occurred in the local region away (~500μm) from ferrite/austenite boundary due to the apparent dissolution of metastable carbides leading to the growth of Type I interfacial carbides close to ferrite/austenite boundary.
Introduction

Dissimilar Metal Welds (DMWs) made between ferritic chrome-molybdenum steels (BCC) and austenitic alloys (FCC) have been widely used in the steam generators of fossil and nuclear power plant applications. Relatively less expensive ferritic steels are used in the low temperature and less corrosive components and the high temperature oxidation and corrosion resistant austenitic alloys are placed in the high temperature components of a power plant. In an entire power plant, there could be around thousands of such DMW configurations. These DMW configurations are often welded using Nickel-base austenitic weld consumables, which provides comparable creep strength as that of the base materials and bridges the extensive coefficient of thermal expansion (CTE) mismatch between the base materials in high temperature service\(^1\). Data from ex-service welds\(^2,3\) have demonstrated that premature failure of these DMWs occur close to the weld fusion line between ferritic steel and Ni-base weld deposit, well below the expected creep life of either of the base materials. These premature failures can cost a power company up to $850000/day loss in revenue due to forced plant outages\(^4\).

Earlier creep failure studies on DMWs\(^5,6,7\) made between ferritic Cr-Mo steels and austenitic Ni base weld deposit have described the failure as low ductility intergranular creep fracture in ferritic steel Heat Affected Zone (HAZ) close to the fusion line of Nickel-base weld deposit. DuPont\(^8\) summarized all the research studies that tried to explain the creep mechanism behind premature creep failure in these DMWs and attributed the failure to two major factors. The first factor is the formation of a continuous network of carbides, termed as Type I interfacial carbides, along ferritic steel Heat Affected Zone (HAZ), at about 5-10μm close to the weld fusion line due to Carbon migration during high temperature service. Creep cavities have been found to nucleate at the interface of carbide-ferritic matrix interface\(^5,6\). Even though these research studies have described this network of carbides to be detrimental to the creep life of DMW configurations, there is a gap in understanding how these carbide/ferritic matrix interfaces tend to become the nucleation sites for the creep cavities. The second major factor associated with failure is coefficient of thermal expansion (CTE) mismatch between ferritic steel and austenitic Nickel-base weld consumable, which results in strain localization close to weld fusion line when these two materials are heated to service temperature. The extent of contribution to failure from both these factors has not also been discussed in any of these studies. These shortcomings emphasize the need for an appropriate creep strain measurement methodology that can capture underlying local creep strain behavior in these heterogeneous weld configurations. Such methodology will be an important tool in the material design and performance of heterogenous weld configurations during high temperature service.

In one of the earliest studies on local creep strain measurements, Parker and Stratford\(^9\) used local strain sensors planted across the weld interface of ferritic 2.25Cr-1Mo steel and austenitic Inconel-82 weld deposit. This arrangement was intended to measure local creep strains along a length of 6-8mm across the weld interface in these DMWs. Local creep strain measurements captured strain concentration along the length of 6-8mm that included almost equal lengths of both 2.25Cr-1Mo steel and Inconel 82 weld deposit materials. However, the spatial resolution of the strain measurement was insufficient to describe discrete local creep strain behavior in microstructurally distinct regions covered by the strain sensors viz., 2.25Cr-1Mo HAZ, 2.25Cr-1Mo base material (unaffected by weld thermal cycles), and Inconel 82 weld during high temperature exposure. Moreover, such
arrangement of strain sensors was not suitable to measure discrete local creep strains along the entire transition length of a heterogeneous weld configuration, where exact location of creep damage is not known beforehand.

Digital Image Correlation (DIC), an in-situ and non-contact surface deformation measurement technique, has the ability to measure discrete local strains in heterogeneous materials with spatial resolution less than a millimeter. In the last decade, DIC has been successfully used to determine residual stress distribution\textsuperscript{10-12} and high temperature tensile behavior\textsuperscript{13-15} of materials. One of the pioneering studies to measure local creep strain measurements using DIC was carried out by Yu. et. al\textsuperscript{16}. In this work, they showed the effectiveness of the DIC technique in determining the localized creep deformation that occurred in the coarse-grained Heat affected zone (CGHAZ) of Gr 91 steel cross-weld specimens. These creep tests were carried out in a Gleeble\textsuperscript{®} thermo-mechanical simulator in an inert gas protected atmosphere, which may not replicate the actual service conditions. In addition, electrical resistance heating in the Gleeble\textsuperscript{®} simulator will not be suitable for testing dissimilar materials, due to the problems in maintaining uniform test temperature associated with abrupt changes in electrical resistivity.

The aim of this study was to extend the DIC test methodology to extract local creep constitutive properties of DMWs made between ferritic Cr-Mo steels and austenitic alloys during conventional creep tests in open air atmosphere. Characterization studies have been carried out to rationalize the observed local creep properties in these DMWs with underlying microstructure evolution.

**Experimental procedure**

A. **Sample fabrication (process, weld metal and base metals)**

The DMW Configuration considered for this study is the bimetallic weld made between 2.25Cr-1Mo steel and Alloy 800H base materials using Inconel weld consumable. These bimetallic welds are generally used in connecting the lower and upper bundles of helical coil steam generators, used in high-temperature-gas-cooled (HTGR) reactor of nuclear power plants.

The DMW coupon was fabricated with $\frac{3}{4}”$ (25.4mm) thick base metal plates of 2.25Cr-1Mo steel and Alloy 800H materials using Nickel-base weld consumables. Chemical compositions of base materials and Nickel-base weld consumables are given in Table 1. Two base metal plates were machined at edges to form 60° included angle single ‘V’ groove with 2mm of root opening. Initial two root weld passes were made with Inconel-82 bare filler wire using Gas Tungsten Arc Welding (GTAW) process with Ar gas purging and the remainder of the plate thickness was filled with Inconel-82 electrode using Shielded Metal Arc Welding (SMAW) process.

B. **Selection of accelerated creep test condition to replicate interfacial service failure**

Parker and Stratford\textsuperscript{6,9}, Nicholson\textsuperscript{5}, and Laha et. al\textsuperscript{17} have carried out accelerated creep tests of DMWs involving 2.25Cr-1Mo steel and an austenitic alloy base material welded with Inconel 82 weld consumable. These tests were carried out at temperatures higher than
the actual service temperature to accelerate low ductility interfacial failure as seen in ex-service DMWs.

Laha et al.\textsuperscript{17} carried out creep tests in the stress range: 90-250\textsuperscript{MPa} at 550\textdegree{}C, but the failures were not associated with the Type I interfacial carbides, typical of service failure. It was identified that the stresses were too high and the corresponding failure times were too short to reproduce creep failure mechanism as seen in service.\textsuperscript{8} Nicholson et al.\textsuperscript{5} carried out tests at temperatures of 570\textdegree{}C and 640\textdegree{}C and stresses of 62 \textsuperscript{MPa} and 100\textsuperscript{MPa}, but the failure time was insufficient for the formation of Type I interfacial carbides. Parker and Stratford\textsuperscript{6,9} understood the existence of a narrow window for the selection of stresses to simulate service failures and developed a matrix of creep tests at temperatures 590-625\textdegree{}C and reduced stresses of 30-80\textsuperscript{MPa}. Samples in the above mentioned test temperatures and stresses were tested in the ‘New’ condition, which was only post weld heat treated (PWHT) at 700\textdegree{}C for 3h, and the ‘Aged’ condition, which was given an aging heat treatment of 625\textdegree{}C for 3500h in addition to the PWHT at 700\textdegree{}C for 3h. This aging treatment was carried out to simulate Type I interfacial carbide morphologies which were perceived to be responsible for the nucleation of creep cavities in these DMW configurations. New samples exhibited low ductility interfacial failure, similar to service, at all stresses 30-80\textsuperscript{MPa} and the aged samples exhibited low ductility interfacial fracture at stresses 50\textsuperscript{MPa} and below and high ductility fracture in 2.25Cr-1Mo base metal at stresses above 50\textsuperscript{MPa}. In all the new and aged welds tested at stresses 50 MPa and below, failure occurred as a consequence of nucleation and interlinkage of creep cavities formed in association with Type I interfacial carbides. These results established that DMWs made between 2.25Cr-1Mo steel and an austenitic alloy with Inconel 82 weld consumable can be tested at stresses of 50\textsuperscript{MPa} and below to simulate the failure seen in service. Hence, the creep test condition of 625\textdegree{}C and 50\textsuperscript{MPa} has been selected for this work.

Moreover, similar to the efforts of Parker and Stratford\textsuperscript{9}, aging heat treatments were carried out on as-fabricated DMW blocks to induce nucleation and growth of Type I interfacial carbides before the start of any creep test. DMW samples were aged at 600\textdegree{}C for 2000 hours and 4000 hours to induce the presence of Type I interfacial carbides of varying sizes and distribution. It was initially anticipated that the presence of Type I interfacial carbides will accelerate creep strain accumulation and will lead to noticeable strain concentration in a short duration of test on the order of one month.

C. Microstructural characterization

Detailed microstructural analyses were performed on the aged DMW samples to examine microstructure features before and after creep tests. Characterization specifically covered 2.25Cr-1Mo HAZ region and the transition region between 2.25Cr-1Mo and Inconel 82 materials as shown in the schematic illustration in Fig. 1. Characterization was conducted using multiple techniques like optical light microscopy, scanning electron microscopy (SEM), X-ray energy dispersive spectroscopy (EDS) and transmission electron microscopy (TEM), as and when deemed necessary.

Metallographic samples for optical and electron microscopy characterization were prepared by grinding through 1200 grit SiC grit papers, followed by diamond polishing in 3\textmu{}m and 1\textmu{}m suspensions. The final polish was done in vibratory-polishing with 0.05\textmu{}m colloidal silica suspension for 3 hours. For the purposes of carbide characterization in ferritic steel, polished DMW metallography samples were swab-etched with Glyceria
etchant for 20 seconds. A Leica DM2500 metallograph was used for performing all light optical microscopy analyses. Scanning electron microscopy (SEM) analyses were carried out using a JEOL 6500 electron microscope equipped with both secondary electron (SE) and backscattered electron (BSE) detectors, with accelerated beam voltages in the range of 15-20kV. Characterization studies to analyze the size and distribution of (i) carbides in different regions along the entire length of 2.25Cr-1Mo HAZ, and (ii) Type I interfacial carbides close to the ferritic/austenitic boundary, as referenced as (1) and (2) in Fig. 1, were carried out in the SEM. SEM images of carbide particles were acquired at uniform intervals of distances and uniform magnifications to make an even comparison between the two aged conditions. Collected SEM greyscale images were imported to ImageJ® software to perform detailed carbide particle analysis. Particle analysis was performed by binary thresholding between carbide particles and the background 2.25Cr-1Mo ferritic matrix in the corresponding SEM greyscale images.

Chemical composition analyses were performed across a 60μm transition length, as referenced as (3) in Fig.1, covering almost equal amounts of ferritic and austenitic materials, in a Versa 3D scanning electron microscope (SEM) built with Oxford® X-ray EDS detectors. X-ray EDS area maps and line profiles were carried out at a beam accelerated voltage of 20kV with a step size of 0.06μm.

For a specific microstructural analysis, transmission electron microscopy (TEM) studies were done in FEI F2000X Talos scanning and transmission electron microscope (STEM). Sample for TEM characterization was extracted along a 30μm transition length, as referenced as (4) in Fig.1, covering almost equal amounts of ferritic and austenitic materials. Electron transparent sample for transmission electron microscopy (TEM) was prepared in Quanta 3D DualBeam microscope equipped with focused ion beam (FIB) machining capability.

D. Creep experiments procedure with Digital Image Correlation (DIC)

An ATS 2330 series ® lever arm tensile creep testing system was used for performing the creep tests. The furnace in the creep test frame was customized with a viewport opening of size 3” (L) x 1” (W) on the front side. This viewport enabled viewing of the test specimen from the outside while it was undergoing creep deformation inside the furnace. The creep test frame with the 3D-Digital Image Correlation (DIC) set up is shown in Fig.2. the 3D-DIC set up consists of two digital cameras mounted at a distance ~100mm apart on a vertical bar and inclined at 10-15º with respect to the vertical bar. This set up covers the full view of the gauge surface of the sample and tracks 3D displacements of every point on the gauge surface. Digital cameras (Point grey® cameras: 2.4MP) with Schneider lenses with a fixed focal length of 28mm were linked to a computer with VIC-snap® image acquisition software: for the programmed capturing of images. An external LED lamp, clamped in between the cameras, was used for illuminating the specimen gauge surface. Flat dog-bone specimens were used for all the creep tests. (redundant). The total gauge length for measuring local strain using DIC covered a length of 47mm with 14-14.2mm of 2.25Cr-1Mo material and the remainder of Inconel-82 and Alloy-800H materials. The gauge surfaces of these test specimens were sandblasted for good adherence to the paints that would be applied for developing a random speckle pattern as part of DIC test procedure. Random speckle patterns were created by an innovative 3-layer speckle pattern procedure with randomly distributed black speckles on a white background. Three
thermocouples were attached to the back surface of the specimen, one on each region of the test specimen, i.e., 2.25Cr-1Mo steel, middle of the Inconel-82 weld and Alloy 800H to monitor temperature gradients within +/- 1°C during creep tests. Speckle patterned test specimens were heated inside the furnace to the test temperature of 625°C at a heating rate of 150°C/hr. After 1 hour of soak time at 625°C, a test load corresponding to 50MPa stress was applied. As soon as the test load was applied, the VIC-Snap® software was programmed to capture images of the speckled gauge surface at every 5.25-minute interval throughout the entire duration of test. While the VIC-Snap® software was periodically collecting images of speckled sample surface during creep deformation, collected images were parallelly imported to VIC-3D® software to determine creep strain ($\varepsilon_{yy}$) distribution in the gauge surface along the loading direction. DIC image capturing was stopped after 268h and 712h in 4000h aged and 2000h aged DMW creep tests respectively, once the locally developed regions of strain concentration reached tertiary stage of creep in the respective test specimens. However, both the test specimens were unloaded after 712h of creep test, to make an even comparison of the extension of creep damage in both the crept samples.

Results

A. Pre-test microstructural characterization of aged DMW samples

Characterization of microstructures and carbide distribution in the Heat Affected Zone (HAZ) of 2.25Cr-1Mo steel during high temperature exposure has been of prime importance in understanding heterogeneous creep behavior of DMW samples, since majority of DMW creep failures have been associated with ferritic steel HAZ\textsuperscript{5,9}. In both the aged conditions, carbide sizes and distribution over the entire length of 2.25Cr-1Mo HAZ were determined. The width of the 2.25Cr-1Mo HAZ in the 2000h aged sample was measured to be approximately 1.6-1.8mm. The region adjacent to the boundary between ferritic/austenitic materials consisted of a coarse grained microstructure of tempered martensite as shown in Fig.3 and this region constituted almost 270-300μm of the total width of the HAZ. The region next to the coarse grained HAZ was fine grained microstructure of ferritic grains as shown in Fig.3, which constituted the remainder of the width of the HAZ. Since these DMW samples were aged for relatively long periods of time, the Inter Critical Zone (ICZ) could not be differentiated in the HAZ of these samples. The width of the HAZ and the observed microstructures in different regions of the HAZ in the 4000h aged sample were similar to that of the 2000h aged sample.

In both aged conditions, the size and distribution of Type I interfacial carbides (redundant) have been studied. Characterization was performed along the entire length of boundary between ferritic and austenitic materials. From the carbide particle analysis, it was observed that the majority of Type I interfacial carbide particles developed a lenticular shape, with the Major axis (M) lying parallel to the ferrite/austenite boundary (perpendicular to the stress direction) and minor axis lying perpendicular to the ferrite/austenite boundary (parallel to the stress direction). This observation was in agreement with the Type I interfacial carbide characterization study done by Parker and Startford\textsuperscript{19}. 

76
Typical representation of Type I interfacial carbide distribution observed in the 2000h and 4000h aged DMW samples are shown in Figs. 4a and 4b respectively. Average carbide sizes along the Major (M) and minor (m) axes and the count of carbides considered for the study are tabulated in Table 2. From the data shown in the Table 2, it is evident that there is a noticeable increase in the carbide sizes along the Major (M) and minor (m) axes in the 4000h aged DMW sample, in comparison to the 2000h aged DMW sample. These results are an obvious indication of carbide particle growth due to the increase in thermal exposure between the two samples. However, the count of particles considered for this evaluation along an approximately equal length of ferrite/austenite boundary increases considerably in the 4000h aged sample, in comparison to the 2000h aged sample. It has been hypothesized that, in addition to growth, the bigger carbide particles in the 4,000 hr sample also coarsen at the expense of relatively smaller particles due to Ostwald ripening as seen in the precipitation of homogeneous alloys. In addition, new carbide particles were also nucleating due to the flux of Carbon towards ferrite/austenite boundary. The area fraction of Type I interfacial carbides along an area of 14 (L) (average length of the interface in each image) x 2 (W) μm was determined for both the aged conditions. The area fraction of interfacial carbides was observed to be 0.142 in the 4000h aged sample compared to 0.074 in the 2000h aged sample.

Chemical concentration analyses were performed along a distance of ~60μm across ferrite/austenite boundary in the as-welded, 2000h aged and 4000h aged conditions. The relative amounts of Fe, Ni, Cr, Mo, Mn and Nb were determined by plotting characteristic X-ray normalized intensity counts of all elements as a function of transition distance in Figs. 5a-c. In comparison to the chemical concentration profiles observed in the as-welded condition, chemical concentration of major alloying elements in both 2000h aged and 4000h showed local depletions and increments (indicated by arrows in Figs. 4b-c) primarily in the ferritic portion. These local variations in chemical concentration should be arising from various metastable carbides that form in the ferritic Cr-Mo steels during isothermal aging treatments. In both 2000h and 4000h aged samples, Mo-rich carbides were seen away from ferrite/austenite boundary and Cr-rich Type I interfacial carbides close to the ferrite/austenite boundary on ferritic side. It may be worthwhile to note that a direct comparison of the gradient lengths in the partially mixed zones between the samples cannot be made because the size the of gradients is highly dependent on the fluid flow conditions during welding, which can be highly variable along the fusion line.

B. Creep response of the aged DMW samples
In the 2000h aged specimen, minor speckle paint degradation occurred on Alloy 800H surface during the process of heating the specimen to the test temperature of 625ºC. This limited the total length of gauge section than was considered in the Region of Interest (ROI) used for post-process creep strain (εyy) analysis. Despite this limitation, 32mm of gauge section including 13.2mm of 2.25Cr-1Mo material and 19mm of Inconel 82 + Alloy 800H materials were included in the ROI for creep strain (εyy) analysis. In the 4000h aged specimen, almost the entire gauge length covering 13mm of 2.25Cr-1Mo material and 31mm of Inconel 82 + Alloy 800H materials was included in the ROI.
A summary of creep strain ($e_{yy}$) results as a function of time (hr) for both the 2000h aged and 4000h aged specimens are shown in Figs. 6a and 6b, respectively. Creep strain distribution in these DMW samples, in both the aged conditions, reveal the heterogenous creep behavior of such DMW configurations. The global creep strain ($e_{yy,\text{global}}$) in these DMW samples is a result of creep strain behavior of three discrete local regions viz., 2.25Cr-1Mo base material, 2.25Cr-1Mo HAZ and Nickel-base alloys (Inconel-82 + Alloy 800H) within this heterogenous configuration. In both the aged samples, creep strain ($e_{yy}$) emanating from the local 2.25Cr-1Mo HAZ crept faster than the 2.25Cr-1Mo base material and was driving the creep-rupture in these DMW samples. One noticeable difference between the creep test results of 2000h aged and 4000h aged samples is the time of emergence of a weak region inside the 2.25-1Mo HAZ and highly straining local region in the DMW configuration. In the case of 2000h aged sample, the weak local region in the 2.25Cr-1Mo HAZ took more than 120 hours of test duration, while in the 4000h aged sample the weak local region in the 2.25Cr-1Mo HAZ emerged in just 30 hours of test duration.

The minimum creep strain rates of Inconel-82 weld deposit and Alloy 800H materials in the transition were negligible in these creep tests for both the aged conditions. The minimum creep strain rates exhibited by both these alloys were of the order of $10^{-7}$ hr$^{-1}$ or less and remained in the steady state of creep throughout the entire duration of tests. This is expected, since both Inconel-82 and Alloy 800H materials are considered to be highly creep resistant in the underlying creep test condition. Fig. 7 shows calculated creep strain rates ($de_{yy}/dt$) as function of time for 2.25Cr-1Mo base material and 2.25Cr-1Mo HAZ for both the aged conditions. In both 2000h and 4000h aged conditions, 2.25Cr-1Mo base materials showed steady state of creep for the entire test duration. Minimum creep strain rates exhibited by 2.25Cr-1Mo base materials in 2000h and 4000h aged conditions were $1.9 \times 10^{-5}$ hr$^{-1}$ and $3.3 \times 10^{-5}$ hr$^{-1}$ respectively. The increase in creep strain rates between the two aged conditions can be rationalized as loss in creep strength of 2.25Cr-1Mo steel due to precipitate coarsening kinetics during thermal exposure. The 2.25Cr-1Mo HAZ in both the aged conditions reached tertiary stage of creep where strain rate accelerated with time, after almost 70% of test time. The minimum creep rates of 2.25Cr-1Mo HAZ in 2000h aged and 4000h aged conditions were $6 \times 10^{-5}$ and $10^{-4}$ hr$^{-1}$, respectively.

C. Location of creep strain concentration inside 2.25Cr-1Mo Heat Affected Zone (HAZ)

In order to locate the location of creep strain ($e_{yy}$) concentration, the creep strain ($e_{yy}$) distribution along the entire gauge length (mm) is shown as a function of test time (hr) in Figs. 8a and 8b for the 2000h aged and 4000h aged specimens, respectively. In the 2000h aged sample, creep strain concentration occurred on 2.25Cr-1Mo side close to the ferrite/austenite boundary. However, due to the DIC limitation of spatial resolution, any creep strain localizing in a region less than 300μm cannot be ascertained. This needs to be validated with the microstructural characterization of creep damage in the samples close to the weld interface. In the 4000h aged sample, creep strain concentration occurred more than 500μm away from the ferrite/austenite boundary. This observation also needed to be validated with the microstructural characterization.
D. Microstructural validation of crept samples

Optical light microscopy and Scanning Electron Microscopy (SEM) were used to characterize the presence of creep cavities in these samples.

2000h aged crept sample: Optical light microscope’s resolution was not able to detect any creep cavities in the location of strain concentration in 2.25Cr-1Mo HAZ. Mounted 2000h aged crept sample was taken to JEOL 65000 Scanning Electron Microscope (SEM) for further characterization. Specimen was characterized along a width of 200μm across the ferrite/austenite boundary for the possible identification of creep cavities. SEM images as shown in Fig. 9 show the presence of creep cavities as big as ~1μm that were formed close (<5μm) to the apparent weld interface on 2.25Cr-1Mo steel side. Examination along the interface length of 2mm side revealed the presence of 10 cavities in the size range of 0.5-1μm

4000h aged crept sample: Optical light microscopy showed copious amount of creep cavity formation in the 2.25Cr-1Mo HAZ. Creep cavities were finely distributed along a region as wide as 700μm and 300μm away from the ferrite/austenite boundary. Further characterization in the scanning electron microscope (SEM) as shown in Fig. 10 showed the presence of creep cavities in HAZ, approximately 300μm away from ferrite/austenite boundary. The observed cavities were in the size range of 1.5-4μm and were much bigger than the size of cavities that were observed in the 2000h aged crept sample, even though both the crept samples were examined tested in the same test condition (625ºC, 50Mpa, 712 hours).

Discussion

A. Limitations in measuring local creep strains using DIC technique

Local strain analysis in the DIC technique is based on tracking displacements in small pockets of regions (subsets) placed at finite distances (steps) in every deforming image of the specimen with reference to the undeformed image of the specimen. Invariably, spatial resolution of strain measurements using DIC technique depends on two factors: (i) subset size and step size selection, and (ii) magnification at which images were captured. Uniform subset size- 17 x 17 pixels and step size- 4 x 4 pixels were used for creep strain analyses in these experiments, which yielded a spatial resolution of 280-300μm. A better spatial resolution could not be achieved in these tests due to the combination of two factors: (i) lack of fineness in distribution of black and white speckle patterns associated with manually spraying these paints, and (ii) errors in displacement values due to existence of convectional heat waves existing inside the furnace, which become increasingly prominent at high magnification images. Strain measurement methods in DIC need to be refined for a spatial resolution better than the current methods. To minimize thermal turbulence due to heat waves, a customized air knife arrangement for a uniform flow of air can be used. The fineness of DIC speckle patterns can be improved by using a fine point airbrush to spray paints. However, these two recommendations need to be validated with ample amount of experimental studies, which is not covered in the scope of the present study.
B. Comparison of global creep strain rates with previous creep studies

The present study has characterized the local creep deformation behavior of 2.25Cr-1Mo HAZ that drives the premature failure in the Dissimilar Metal Weld (DMW) made between 2.25Cr-1Mo steel and an austenitic alloy using Nickel-base weld consumable. Since local creep deformation behavior was not studied in the earlier creep studies of DMW, minimum global creep strain data obtained in the current study has been compared with that of the previous studies. In the creep studies done by Parker and Startford on DMWs made between 2.25C-1Mo steel and an austenitic alloy with (the same ??) Nickel-base weld consumable, a global creep strain rate of $3.33 \times 10^{-6} \text{ hr}^{-1}$ was obtained for the same creep test condition used here (625°C, 50Mpa). Samples used for these tests were post-weld heat treated (PWHT) at 700°C for 3h after weld fabrication. To make an even comparison with this study, minimum global creep rates in the present study were obtained by maintaining the same proportions of different materials (46.25% of Inconel 82 weld deposit, 26.875% each of ferritic and austenitic base materials) in the gauge section to that of the previous study. Minimum global creep strain rates obtained in the 2000h aged and 4000h aged specimens were $1.7 \times 10^{-5} \text{ hr}^{-1}$ and $2.72 \times 10^{-5} \text{ hr}^{-1}$, which are approximately an order higher than that observed in the creep tests by Parker and Startford in the same test condition. Considerable increase in the observed global creep strain rates in the present study can be rationalized due to the presence of pre-existing array of Type I interfacial carbides, resulting from the aging treatments (600°C for 2000h and 600°C for 4000h) employed in this work. However, it also needs to be duly noted that the location of creep strain concentration in the 4000h aged DMW specimen is away from the ferrite/austenite boundary and the underlying creep mechanism in this aged condition would be different from that observed in the 2000h aged DMW aged counterpart.

C. Validation of creep damage with interfacial failure seen in ex-service welds

SEM characterization of the 2000h aged specimen revealed a sparse distribution of creep cavities. The creep cavities were in the size range of 0.5-1μm and were distributed at an average of 5 cavities/mm on 2.25Cr-1Mo steel side close to the ferrite/austenite boundary. This was in good agreement with interrupted creep cavity damage evaluation done by Parker and Stratford, on crept samples of DMWs made between 2.25Cr-1Mo steel and AISI 316 stainless steel as parent materials, welded with Inconel 82 weld consumable. Their studies showed that creep cavities observed were in the size range of 0.5-1μm and they were distributed at an average of less than 10 cavities/mm until 50% of life. No cavity coalescence or microcracks were observed till 80% of life was exhausted in these earlier studies. These agreements gave a clear indication that the creep damage observed in the crept 2000h aged DMW specimen should replicate low ductility intergranular creep fracture observed close to ferrite/austenite boundary in ex-service DMWs. Creep cavities observed in the 4000h aged DMW specimen was finely distributed in the HAZ, away from the ferrite/austenite boundary. Moreover, observed creep cavities were in the size range of 1.5-4μm. These observations indicate 4000h aged DMW specimen will not replicate low ductility intergranular creep fracture as seen in ex-service welds.
D. 2000h aged DMW: Depletion of alloying elements in the ferritic matrix surrounding Type I interfacial carbides

High resolution TEM image close to the boundary of ferritic (α) and austenitic (γ) materials in the crept 2000h aged DMW sample showing creep cavity is shown in Fig. 11. EDS mapping was carried out in the selected regions as shown as 1 and 2 of Fe matrix surrounding the Cr-rich Type I interfacial carbides in Fig. 11. X-ray spectra analyses of these selected regions show the presence of alloying elements like Fe, Cr and Ni in ferritic matrix. X-ray intensity peak of Mo-L was not observed. It needs to be duly noted that molybdenum is completely depleted of the ferritic matrix. Elements like Ni and Cr appear to have diffused from the austenitic side during thermal exposures of aging treatment: 600°C for 2000h and creep test: 600°C for 712h, depletion of Mo in ferritic matrix is a good indication of level of temepering that local regions has undergone in comparison to 2.25Cr-1Mo base material.

Bairde and Jamie\textsuperscript{22} explained an ‘interactive solid solution’ effect in imparting creep strength to Cr-Mo steels. Their studies in a Fe-Cr-Mo-C solid solution showed that carbide forming substitutional alloying elements (Mo,Cr) will have the tendency to form clusters with interstitial (C). These clusters will extend the solid solution strengthening effect of C to much higher temperatures (>700K), than is the case in pure Iron. In the creep studies on 2.25Cr-1Mo steel above 700K, Kleuh\textsuperscript{23} and Wendell et. al\textsuperscript{24} demonstrated the combined effect of Mo and C in imparting creep strength to 2.25Cr-1Mo steel. Their studies explained that 2.25Cr-1Mo extracts its creep strength from the longevity of two subsequent phases of strengthening mechanisms: i) interaction solid-solution strengthening of Mo-C clusters in Fe-Cr-Mo-C solid solution during initial stages of tempering followed by, ii) precipitate strengthening as a result of fine dispersion of Mo$_2$C carbides in the subsequent stages of tempering. The present study shows that Mo is depleted in Fe-Cr-Mo-C solid solution in the ferritic steel portion close to α/γ boundary as a result of formation of a network of Type I interfacial carbides.

E. 4000h aged DMW: Dissolution of carbides in HAZ away from ferrite/austenite boundary

LOM and SEM characterization of the 4000h aged crept DMW sample showed a fine distribution of creep cavities in the 2.25Cr-1Mo HAZ approximately 500μm away from the ferrite/austenite boundary. The entire length of the HAZ in both 2000h and 4000h aged DMW specimens was characterized for the local region-wise distribution of carbides to rationalize this change in the local creep strain concentration location. Fig. 13(a) & (b) show SEM images of the etched local region (500μm away from ferrite/austenite boundary) in 2000h and 4000h aged DMW specimens and Figs. (should be Fig 13)(c) & (d) show the respective images processed in ImajeJ® software to characterize carbide distribution. These images show an apparent depletion in the amount of carbides in the 4000h aged samples, in comparision to the 2000h aged DMW sample. In the 2000h aged DMW, carbides are distributed both along the grain boundaries and inside the grains (indicated by black arrows in Fig. 13c). However, in the 4000h aged DMW, carbides are majorly distributed along the grain boundaries and the majority of grain interiors were depleted of carbides (indicated by black arrows in Fig. 13d)
Fig. 14 shows statistical data of area fraction of carbides and area fraction of creep voids (for just the 4000h aged DMW sample) plotted against HAZ distance. It is evident that creep voids were formed along a distance of ~500μm away from the ferrite/austenite boundary in 4000h aged sample as a result of carbide depletion in that local region, when compared to the same local region in 2000h aged DMW sample. The apparent carbide depletion in this region can be inferred due to the diffusion of C from Cr-depleted ferritic steel side towards Cr-rich austenitic alloy on the other side of the weld interface. This rationale is supported by the enrichment of carbides, as Type I interfacial carbides in the diluted portion of 2.25Cr-1Mo steel (Fig. 12b) and a relative depletion in carbides moving away from the interface (Fig. 12b). Kleuh also observed deterioration in creep-rupture properties of normalized and tempered 2.25Cr-1Mo steel with decreasing carbon content in the alloy.

**Comparative Creep studies on Graded Transition Joints**

Creep tests were carried on the Graded Transitions Joints (GTJs) fabricated with filler metals (i) Inconel 82, (ii) P87 and (iii) 347H, in a test condition of 625°C. GTJ samples used for the tests were machined from blocks that were aging heat treated at 600°C for 2000h. This was done to maintain the same thermal histories of the conventional DMW coupons that were tested in the same creep test condition. The goal of these tests is to obtain local creep constitutive properties along the entire gauge length of GTJ constituting i) 2.25Cr-1Mo base material, (ii) graded transition region, and (iii) Alloy 800H material and compare the same with the baseline creep strain results of conventional DMW.

Experimental procedure for creep tests is similar to the creep test procedure for conventional DMW specimen as discussed in the previous sections. Flat dog-bone specimens were used for all the creep tests. The total gauge length for measuring local strain using DIC covered a length of 47mm with 12-14mm of 2.25Cr-1Mo material, 20mm of grade length and the remainder of Inconel-82 and Alloy-800H materials. Creep test on GTJ fabricated with 347H filler metal is not completed till time. Hence, creep test results of the GTJs fabricated with filler metals: Inconel 82 and P87 are discussed in this section.

**Creep response of the aged GTJ samples**

In the 2000h aged Inconel-82 GTJ specimen, minor speckle paint degradation occurred in the mixed phase and FCC regions of the transition during the process of heating the specimen to the test temperature of 625°C. This limited the total gauge section that was considered in the Region of Interest (ROI) used for creep strain ($e_{yy}$) analysis. However, it was noted during the experiment that creep strain concentration didn’t take place in speckle pattern degraded regions. A summary of creep strain ($e_{yy}$) results as function of test time (hr) for both the 2000h aged Inconel 82 and P87 GTJ specimens are shown in Figs. 15a and 15b respectively. Similar to the creep behavior of 2000h aged DMW specimen, heterogenous creep strain distribution was observed inside the gauge section of these GTJ specimens. Global creep strain ($e_{yy,global}$) in these samples is a result of creep strain response of discrete regions inside the gauge section viz., 2.25Cr-1Mo base material, 2.25Cr-1Mo HAZ, BCC portion of the grade length, mixed and FCC phase portions of the grade length. In both the GTJ samples, creep strain emanating from 2.25Cr-1Mo HAZ...
region crept faster than the other local regions in the transition and it was driving the creep failure in these specimens. Width of the HAZ were observed to be to be close to 5mm in both the GTJ specimens.

In order to locate the exact location of creep strain (eyy) concentration, creep strain distribution along the entire gauge length is plotted as function of test time (hr) for Inconel 82 and P87 GTJs in Figs. 16a and 16b respectively. In both the GTJ samples, creep strain concentration occurred in 2.25Cr-1Mo HAZ at a distance of 3-3.5mm away from ferrite/austenite boundary.

**Comparison of GTJ’s creep strain results**
Creep strain results of aged GTJs were compared with that of the aged DMWs to check suitability of GTJs to overcome premature creep failure problem in the traditional DMWs in service. Since creep strain concentration occurred in the Heat Affected Zone (HAZ) of 2.25Cr-1Mo in both DMW and GTJs, minimum creep strain rates (deyy/dt) of 2.25Cr-1Mo HAZ and base material were compared in Fig. 17. It can be seen from Fig. 17, minimum creep strain rates observed in 2.25Cr-1Mo HAZ of Inconel 82 and P87 GTJs were 8.6 x 10^{-5} \text{hr}^{-1} and 7.9 x 10^{-5} \text{hr}^{-1} respectively. These observed values are marginally higher than the minimum creep strain rate of 6 x 10^{-5} \text{hr}^{-1} as observed in 2.25Cr-1Mo HAZ of conventional DMW configuration. Microstructural investigation of these local creep concentration regions in the GTJs are currently underway to determine the rationale behind this unexpected behavior.

**Summary**

1. Local creep strain measurements made using DIC technique has helped in understanding the heterogenous creep behavior of dissimilar metal weld (DMW) configurations. Further refining to address limitations in current methods will make this technique an appropriate methodology to study creep behavior is dissimilar metal configurations.

2. 2000h aged DMW specimens used for the creep tests exhibited strain concentration close to ferrite/austenite boundary. Strain concentration occurred in this local region due to depletion of alloying elements (Mo, Cr) in Fe solid solution, in virtue of formation of network of Type I interfacial carbides.

3. 4000h aged DMW specimens used for the creep tests appear to have been overaged to replicate strain concentration close to ferrite/austenite boundary as seen in ex-service welds. Strain concentration occurred in this local region due to the relative depletion of carbides in this region due to enrichment of Type I interfacial carbides. Failures in local regions away from ferrite/austenite boundary can also be envisaged, when power plants do not operate at their full capacities and experience frequent shutdowns.

4. Local creep strain results of 2000h aged DMW can be used as a baseline to design weld joint configurations, that could potentially overcome premature failures in DMW configurations used power plant applications.
References


7. Kleuh RL. Relaxation Behavior of 2.25Cr-1Mo steel under multiple loading.


18. Babu SS, David SA, Vitek JM, Miller MK. Phase Stability and Atom Probe Field Ion Microscopy of Type 308 CRE Stainless Steel Weld Metal. 27.


Figures

**Figure 1:** Schematic illustration showing different characterization studies in the aged DMW specimens: (1), (2) Optical & SEM, (3) EDS, (4) TEM

**Figure 2:** 3D-DIC set-up in front of ATS 2330 creep test system
Figure 3: Microstructures observed in the different regions of 2.25Cr-1Mo Heat Affected Zone (HAZ) of 2000h aged DMW specimen

Figure 4: SEM micrographs showing an array of Type I interfacial carbides close to the boundary between ferritic/austenitic materials (Inconel 82- Left, 2.25Cr-1Mo- Right) in (a):2000h aged DMW sample, (b) 4000h aged DMW sample
Figure 5: Chemical concentration profiles of alloying elements: Fe, Cr, Ni, Mn, Mo, Nb across ferrite/austenite boundary in (a) as-welded, (b) 2000h aged, and (iii) 4000h aged conditions
Figure 6a: Creep strain ($e_{yy}$) evolution in different regions viz., 2.25Cr-1Mo HAZ (Red), 2.25Cr-1Mo base material (Blue) and Nickel-base alloys (Pink) of 2000h aged DMW specimen. Creep test condition: 625°C, 50MPa, duration: 0-712h

Figure 6b: Creep strain ($e_{yy}$) evolution in different regions viz., 2.25Cr-1Mo HAZ (Red), 2.25Cr-1Mo base material (Blue) and Nickel-base alloys (Pink) of 4000h aged DMW specimen. Creep test condition: 625°C, 50MPa, duration: 0-268h
Figure 7: Local creep strain rate \( (d\varepsilon_{yy}/dt) \) as function of test time (hr) for the regions (i) 2.25Cr-1Mo HAZ and (ii) 2.25Cr-1Mo base material in the creep test condition: 625°C, 50MPa.

Figure 8(a): Creep strain \( (\varepsilon_{yy}) \) evolution along the gauge length of 2000h aged DMW specimen. Creep test condition: 625°C, 50MPa, 0-712h. (b): Creep strain \( (\varepsilon_{yy}) \) evolution along the gauge length of 4000h aged DMW specimen. Creep test condition: 625°C, 50MPa, 0-268h.
Figure 9: SEM micrographs close to the ferrite/austenite boundary (Inconel 82- Right, 2.25Cr-1Mo- Left) showing the presence of creep cavities in 2.25Cr-1Mo side close to the interface in the crept 2000h aged DMW sample. Creep test condition: 625°C, 50MPa, After 712h

Figure 10: SEM micrographs showing the presence of creep cavities in HAZ (~300μm away from ferrite/austenite boundary) in the crept 4000h aged DMW sample. Creep test condition: 625°C, 50MPa, after 712h
Figure 11: High magnification TEM image across interface of crept 2000h aged DMW sample (Creep test condition: 625°C, 50MPa, 712h) showing Cr-rich interfacial carbides close to ferrite/austenite boundary. (Interface between ferritic and austenitic alloys are denoted by black arrows, ferritic side on the left and austenitic side on the right)

Figure 12: X-ray spectrum corresponding to locations: 1 and 2 of Fe matrix as indicated in Fig. 11
Fig. 13: SEM micrographs and the respective processed images of HAZ region located 500μm away from ferrite/austenite boundary in (a,c) 2000h and (b,d) 4000h aged DMW conditions respectively before any creep tests
Fig. 14: Statistical data showing the area fraction of carbides in the HAZ of both 2000h and 4000h aged DMW samples. Data also plotted along with area fraction of voids in the crept 2000h and 4000h aged DMW sample. Creep test condition: 625°C, 50MPa, after 712h.

Figure 15a: Creep strain ($\varepsilon_{yy}$) evolution in different of 2000h aged Inconel 82 GTJ specimen. Creep test condition: 625°C, 50MPa, duration: 0-712h.
Figure 15b: Creep strain ($e_{yy}$) evolution in different of 2000h aged P87 GTJ specimen. Creep test condition: 625°C, 50MPa, duration: 0-712h

Figure 16: Creep strain ($e_{yy}$) evolution along the gauge length of (a) 2000h aged Inconel 82 GTJ specimen and (b) 2000h aged P87 GTJ specimen. Creep test condition: 625°C, 50MPa, 0-712h
Figure 17: Local creep strain rate ($\frac{de_y}{dt}$) as function of test time (hr) for the regions (i) 2.25Cr-1Mo HAZ and (ii) 2.25Cr-1Mo base material in the creep test condition: 625°C, 50MPa. Creep test specimens: Inconel and P87 GTJ, conventional DMW

Tables

**Table 1:** Chemical composition of materials used in high temperature applications (Single values are maximum)

<table>
<thead>
<tr>
<th>Materials</th>
<th>Chemical composition (Wt%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Al</td>
</tr>
<tr>
<td>2.25Cr-1Mo steel</td>
<td>--</td>
</tr>
<tr>
<td>Alloy 800H</td>
<td>0.15-0.6</td>
</tr>
<tr>
<td>Inconel 82</td>
<td>--</td>
</tr>
<tr>
<td>Inconel 182</td>
<td>--</td>
</tr>
</tbody>
</table>

**Table 2:** Average carbide particle dimensions along the major ($M_c$) and minor ($m_c$) axes in both the aged conditions

<table>
<thead>
<tr>
<th>Aging condition</th>
<th>Average carbide dimension along minor axis, $m_c$ (μm)</th>
<th>Average carbide dimension along major axis, $M_c$ (μm)</th>
<th>Number of particles considered for evaluation</th>
</tr>
</thead>
<tbody>
<tr>
<td>2000h aged</td>
<td>0.21</td>
<td>0.36</td>
<td>380</td>
</tr>
<tr>
<td>4000h aged</td>
<td>0.27</td>
<td>0.43</td>
<td>501</td>
</tr>
</tbody>
</table>