Advances in Materials Technology for Fossil Power Plants
Proceedings from the Seventh International Conference
October 22–25, 2013, Waikoloa, Hawaii, USA
https://doi.org/10.31399/asm.cp.am-epri-2013p1016

D. Gandy, J. Shingledecker, editors

EFFECT OF NON-STANDARD HEAT TREATMENTS ON CREEP PERFORMANCE OF CREEP-STRENGTH ENHANCED FERRITIC (CSEF) STEEL WELDMENTS

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ABSTRACT (074)

This paper summarizes recent efforts to improve creep performance in Grade 91 (Mod. 9Cr-1Mo, ASTM A387) steel weldments via non-standard heat treatments prior to welding. Such heat treatments offer a potential solution for minimizing Type IV failures in creep strength enhanced ferritic (CSEF) steels. A lower temperature tempering (LTT, 650°C) of the 9Cr steels prior to gas tungsten arc welding (GTAW) resulted in improved creep-rupture life at 650°C compared to the samples tempered at a standard condition (HTT, 760°C) before welding. From detailed characterization of precipitation kinetics in the heat affected zone, it was hypothesized that $M_{23}C_6$ carbides in the fine-grain heat-affected zone (FGHAZ) in the LTT sample were fully dissolved, resulting in re-precipitation of strengthening carbides during post weld heat treatment (PWHT). This was not the case in the HTT sample since $M_{23}C_6$ in the FGHAZ was only partially dissolved prior to welding, which caused coarsening of existing $M_{23}C_6$ after PWHT and premature creep failure in the FGHAZ. However, it was also found that the LTT raised the ductile-brittle transition temperature above room temperature (RT). Two different thermo-mechanical treatments (TMTs); two-step tempering and aus-forging/aus-aging, of the modified 9Cr-1Mo steels were attempted, in order to control the balance between creep properties and RT ductility, through control of precipitation kinetics of the $M_{23}C_6$ carbides and/or MX carbo-nitrides. The hardness map of the TMT samples after GTAW and PWHT were evaluated.

INTRODUCTION

Creep-strength enhanced ferritic steels containing 9-12Cr are now extensively used in coal-fired boilers, heat-recovery steam generators, and piping systems in new high-efficiency fossil power plants because of their excellent creep properties at elevated temperatures matched with reasonable material costs [1,2]. The performance of CSEF steel components, however, does not always meet expectations in many industrially relevant circumstances, and there have been reports of numerous failures in the heat affected zone (HAZ) of CSEF steel weldments after only a few years in service [3-6]. These failure locations are often traced back to the fine-grained heat

This manuscript has been authored by UT-Battelle, LLC, under Contract No. DE-AC05-00OR22725 with the U.S. Department of Energy.

affected zone (FGHAZ) of a weld, which experiences a weld thermal cycle with a peak temperature just above the A_{c3} temperature. It is a well-established fact that non-equilibrium microstructures and their gradients in FGHAZ regions will significantly reduce creep strength [7-10]. Moreover, the grain size and orientation of the FGHAZ can lead to high tri-axial stress states that may contribute to accelerated creep damage [11,12]. This combination of circumstances leading to premature failure of welded components is discussed in the literature as "Type-IV failures in CSEF steels." Published literature also shows that the reduced creep strength of such joints can be on the order of 40%, compared to the base metal for a given service life [13]. Therefore, minimizing Type IV failure is essential for improving the CSEF steel components used in many industrial applications, especially at service temperatures above 550°C [14].

Previously published work [15] indicated that modification of plate tempering and post weld heat treatment (PWHT) procedures had the potential of minimizing behaviors contributing to Type IV failures. The observations were based on testing a gas-tungsten arc weld (GTAW) made in Grade 91 (modified 9Cr-1Mo with V, Nb, and N, ASTM A387) plate using Grade 9 (9Cr-1Mo) filler metal. The plates used for welding were normalized as usual but tempered at 621°C (1150°F) rather than above the ASME Code specified minimum of 732°C (1350°F). (For reference, contemporary practice for Grade 91 uses tempering temperatures of 760°C and above). Pieces of the weld made with these plates were then given a PWHT at temperatures of either 732°C or 760°C. Specimens of both PWHT pieces were then prepared for metallographic examination, hardness testing, and creep-rupture testing. In creep-rupture testing at 593°C, cross-weld specimens from the 621°C temper/760°C PWHT coupon failed in the base metal with cup-cone characteristics, indicating relatively high ductility. In contrast, cross weld specimens from the 760°C temper/732°C PWHT coupon failed in the HAZ region under the same temperature and stress conditions. Shorter creep-life was also observed in the 760°C temper/732°C PWHT coupons.

Current activities related to microstructure control in CSEF steels included reexamining previous work, and additional creep testing of the modified 9Cr-1Mo steel weldments, with standard and non-standard pre-weld tempering [16]. It was found that non-standard pre-weld tempering conditions resulted in improved creep properties compared to the standard heat treated samples. Detailed microstructure analysis through an in-situ X-ray diffraction study of the simulated HAZ on modified 9Cr-1Mo samples revealed the potential improvement mechanism of the creep properties using the non-standard material heat treatment [17,18]. This paper summarizes our recent progress on characterization of the microstructure of the modified 9Cr-1Mo weldments; consideration of the ductile-brittle transition temperature of the base metals imparted by the non-standard heat treatment; and trial control of precipitation kinetics through two different types of thermo-mechanical treatments in order to control the balance between creep properties and room temperature ductility.

EXPERIMENTAL

A 25 mm thick plate of Grade 91 (modified 9Cr-1Mo) steel, with the chemistry shown in Table 1, was used in the present study. Several small pieces (50-100 mm x 150-250mm x 25mm) were sectioned from the hot-rolled plate sample [15]. The pieces were normalized at 1050°C for 1h under Ar cover gas, and then air cooled. Some pieces were tempered at several different temperatures (600, 650, 700 {=non-standard}, 760, and 800°C {=standard}) for 1.5-2h, and then air cooled. After applying single-V or double-V shape GTAW with a matching 9Cr-1Mo filler metal (the chemistry is also listed in Table 1), PWHT was conducted at 760 or 800°C for 2-4h.

Micro-Vickers hardness measurements were conducted on the cross-section of the welded 9Cr-1Mo steels after PWHT, using a 300g load and automated stage motion measuring hardness in 50-100µm steps on a traverse across the base metal, weld and heat affected zone. Sub-sized and full-sized cross-weld tensile specimens (ASTM E8) with a cylindrical gage section were machined and creep-rupture tested at 600-650°C and 50-120MPa in laboratory air. V-notch Charpy impact testing (ASTM E23) on samples machined from the mod. 9Cr-1Mo steel tempered at 650°C and 760°C was conducted in a temperature range from -100°C to 200°C. Microstructural characterization was conducted using an optical light microscope (OM) and a scanning electron microscope (SEM).

Aus-forging/ aus-aging was also applied to the mod. 9Cr-1Mo steel plates for potential enhancement of stable MX carbo-nitride precipitation prior to the martensitic transformation [19,20]. The samples were heated to 1050°C and held for 1h, followed by air-cooling to an intermediate temperature in the range between 700°C and 900°C (just below or above A_{c1}/A_{c3} at ~820°C, and higher than M_S at ~400°C). Once the intermediate temperature was achieved a 5-10% thickness reduction was applied by forging. The plates were subsequently placed in a furnace for 1 h at the same temperature that forging was performed, and then air-cooled to room temperature (RT). The samples were then tempered at 760°C for 2h. GTAW and PWHT at 760°C for 2h were performed and the HAZ hardness was evaluated.

Table 1: Chemical composition of the base and weld filler metal (Grade 91).

(wt%)	Fe	C	Mn	Si	Cr	Mo	Ni	V	Nb	N	B
Base	Bal.	0.08	0.27	0.11	8.61	0.89	0.09	0.21	0.07	0.06	<0.001
Filler	Bal.	0.08	0.41	0.31	8.62	0.92	0.15	0.24	0.08	0.04	<0.001

RESULTS AND DISCUSSION

Low Temperature Pre-weld Tempering

Significant improvement of the creep properties were observed in the mod. 9Cr-1Mo steel weldments when lower temperature pre-weld tempering (non-standard heat treatment) was applied. **Figure 1** represents the comparison of the creep-rupture test results on the modified 9Cr-1Mo steel weldments, with various pre-weld tempering conditions, by plotting the Larson-Millar Parameter (LMP) as a function of stress. The circle and diamond symbols correspond to the samples subjected to the lower temperature tempering (LTT, below 650°C) and higher temperature tempering (HTT, above 700°C), respectively, prior to welding. The LTT samples tend to show longer creep-rupture lives than the HTT samples, in agreement with previous reports [15] (the open symbols) and in the present tests (the filled symbols), especially at lower stress levels. The lower temperature tempering also resulted in an improvement of the weld strength reduction factor when compared with the creep properties of the base modified 9Cr-1Mo steel (the broken line).

Figure 2 shows the creep-rupture specimens tested at 650°C and 70MPa in the present study. These samples were tempered at various temperatures from 600 to 800°C, and then welded and post weld heat treated at 800°C. They showed a clear transition of creep-rupture life from relatively shorter to longer, between the pre-weld tempering temperatures above 700°C and below 650°C. However, all the specimens in the present study tended to rupture in HAZ. Cross-sectional observation of the creep-rupture specimens revealed that the fracture took place at

FGHAZ, as shown in the OM picture. These results indicate that the improvement of creep-properties is somehow associated with the microstructural changes in the FGHAZ through the non-standard heat treatment.

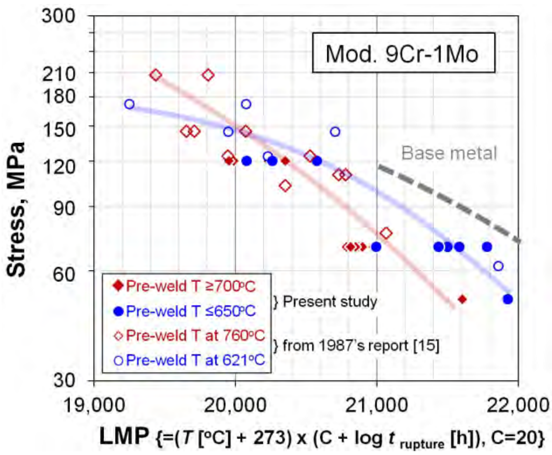

Figure 1. Larson-Miller parameters (LMP) of cross-weld 9Cr-1Mo steels with standard or non-standard pre-weld heat treatment, followed by GTAW and PWHT, plotted as a function of creep stress. Creep tests were conducted in a range of 593-650°C and 62-172MPa [15]. The LMP curve of the base metal is also drawn for comparison purpose.

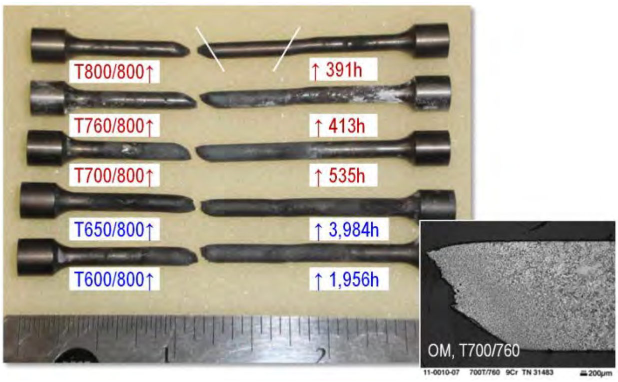

Figure 2. Creep-ruptured specimens of cross-welded 9Cr-1Mo steels with higher temperature tempering (T800, T760, and T700), or lower temperature tempering (T650 and T600), followed by GTAW and PWHT at 800°C. The creep test was conducted at 650°C and 70MPa. The cross-sectional OM micrograph of a creep-ruptured specimen is also attached.

It was found that the non-standard heat treatment drastically changed the hardness distribution in the HAZ. The hardness color maps of both standard and non-standard pre-weld tempered samples, after GTAW and PWHT, are shown in **Figure 3**. The standard heat treated sample showed a distinct low hardness region in the FGHAZ (and adjacent base metal, possibly due to an over-tempering effect). The LTT non-standard sample showed a gradual hardness reduction from the fusion line to the base metal. Since the observed microstructure by OM or SEM did not reveal a microstructural feature that could explain the difference, speculation suggests that the hardness difference could be attributed to the nano-scale precipitate formation in the area. Although the hardness traverse was conducted at room temperature the test results may be relevant to the observed improvement in the high temperature creep properties of the materials.

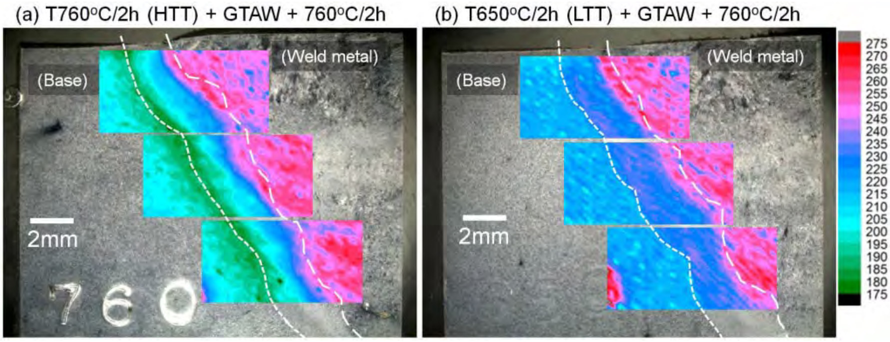

Figure 3. Cross sectional microstructure of the mod. 9Cr-1Mo steel after (a) standard (HTT) or (b) non-standard (LTT) pre-weld heat-treatment + GTAW + PWHT, together with super-imposed hardness color maps measured across the base metal, heat affected zone, and weld metal.

Detailed analysis of the precipitation kinetics was attempted through an in-situ X-ray diffraction study of the HAZ simulated mod. 9Cr-1Mo samples at SPring-8, Japan. The detail experimental procedure is described elsewhere [17,18]. The study revealed that the standard heat treated sample showed incomplete dissolution of $M_{23}C_6$ at the FGHAZ during welding, and it caused coarsening of the residual $M_{23}C_6$ after PWHT in the FGHAZ area, which would not contribute to the high temperature strength. On the other hand, the non-standard heat treated sample exhibited less $M_{23}C_6$ formation (not fully "tempered") which allowed complete dissolution of the $M_{23}C_6$ during welding, and then redistribution of fine $M_{23}C_6$ during PWHT. The fine distribution of the carbides may contribute to the higher temperature creep resistance. Based on the results, the potential microstructure evolution at the FGHAZ during welding and PWHT are hypothetically proposed via illustration in **Figure 4**. It should be emphasized that MX carbo-nitrides were also detected in the diffraction study, and they showed almost no change in volume fraction during the HAZ simulated heat treatment. This indicates that MX is very stable and it could be utilized for a source of high temperature creep resistance at the FGHAZ through a proper control.

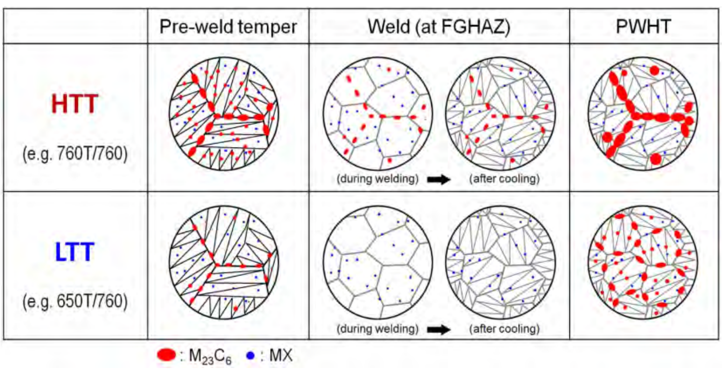

Figure 4. Schematic illustration showing microstructure evolutions during welding and PWHT of the mod. 9Cr-1Mo steel with HTT and LTT conditions [17,18].

Lower temperature tempering caused the ductile-brittle transition temperature (DBTT) of the base mod. 9Cr-1Mo steel to increase above RT. **Figure 5** shows the impact absorbed energy measured by Charpy impact testing of the mod. 9Cr-1Mo steel with standard and non-standard tempering (for 2h at 760 and 650°C, respectively), plotted as a function of test temperature. The HTT sample showed a DBTT of approximately -50°C, whereas the LTT sample was approximately 80°C. Longer time tempering (24h) at 650°C did not significantly improve the ductility. This indicates that the LTT base metal could be easily broken during installation at a factory or in the field if mishandled. The high DBTT could be due to high amount of residual C in solution in the martensitic matrix thereby decreasing the toughness of the material. On the other hand, a drastic improvement was observed when the second tempering (760°C for 2h) was applied to the LTT sample. This result indicates that there is a potential solution to control both creep properties and RT ductility; use LTT for refining $M_{23}C_6$ carbides, and add the second-step tempering to return the DBTT to below RT.

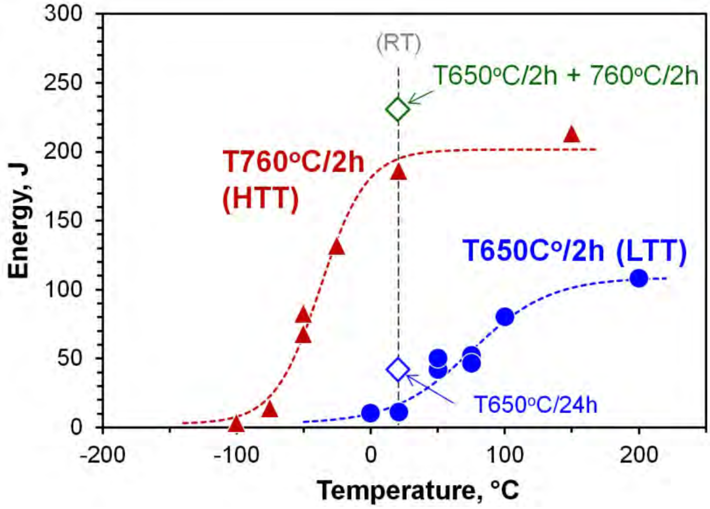

Figure 5. Charpy impact test results of the mod. 9Cr-1Mo steels applied standard (HTT) and Non-standard (LTT) tempering. The results of the longer-time tempered sample (650°C/24h) and two step tempered sample (650°C/2h + 760°C/2h) are also plotted.

Two-Step Tempering

Based on the discussion in the precious section, two-step tempering (650°C/2h + 760°C/2h) was evaluated in the mod. 9Cr-1Mo steel sample. **Figure 6** shows the cross-sectional OM micrograph and the hardness color map of the first trial two-step tempered sample after GTAW and PWHT. It exhibited a distinct weak zone at FGHAZ similar to the standard heat treated sample shown in **Fig. 3a**, indicating that the current two-step tempering condition did not achieve the desired control of the $M_{23}C_6$ size to allow the complete dissolution during welding. Preliminary SEM observation also confirmed that relatively coarse $M_{23}C_6$ was present in the base metal prior to welding. More experimental trials are required to further optimize the first and second tempering conditions to achieve both the carbide refinements and fully tempered conditions.

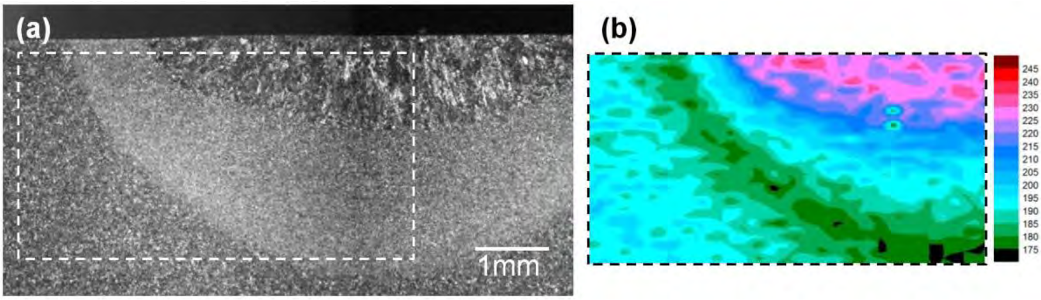

Figure 6. (a) OM micrograph showing a cross-sectional view of the mod. 9Cr-1Mo steel with two step tempering applied GTAW and PWHT, and (b) the hardness color map corresponding to the area with a broken line.

Aus-Forging/ Aus-Aging

Klueh et al. [19,20] reported a breakthrough concept for improving creep properties of 9-12Cr CSEF steels through a thermo-mechanical treatment (TMT) with aus-forging and aus-aging. This process enhances the formation of the stable MX carbo-nitrides at austenitizing temperatures by applying hot-work (e.g. rolling or forging) to introduce dislocations which act as nucleation sites for the MX. Following forging, aus-aging resulted in a dense MX precipitation before the austenite matrix transformed to martensite. The resulting material exhibited almost two to three orders of magnitude longer creep life at 650°C and 138MPa [19]. Since MX precipitates were stable and would not be dissolved into the matrix at the FGHAZ during welding as discussed in the previous section [17,18], the aus-forging/ aus-aging could be worth trying for a potential improvement of high temperature creep properties at the FGHAZ.

Figure 7 shows the CCT/TTT combined diagram of the modified 9Cr-1Mo steel made in the current study. The samples were heated to 1050°C, aus-forged 5-10% and aus-aged at 700°C, 800°C, and 900°C, followed by air-cooling to RT. It should be noted that the aus-forging/ aus-aging temperatures were selected to be higher than the than M_S (400°C). Similar heat treatment without aus-forging was also made for comparison purpose. It was found that there was a C-curve of the ferrite formation with a nose around 700°C and 1h when no forging was applied, and the curve sifted toward higher temperature and shorter time by applying aus-forging. The SEM back scatter electron images of the sample processed at 800°C and 700°C (**7b** and **7c**) clearly showed the transition of the microstructure from martensite to ferrite.

Figure 8 shows the hardness color maps of the aus-forged/ aus-aged samples at 900°C and 800°C with the standard tempering at 760°C/2h, followed by GTAW and PWHT. The sample processed at 900°C (**8a**) showed almost the same hardness distribution as the standard heat treated sample with a distinct hardness dip at the FGHAZ. On the other hand, the sample processed at 800°C (**8b**) exhibited very high hardness in the base metal together with much less hardness reduction at HAZ. Although it is still difficult to estimate the high temperature creep properties from the RT hardness test results, the 800°C aus-forged / aus-aged sample reveals a processing window with significant promise in potentially improving the creep properties, when compared with standard heat treated samples. Property screenings of the TMT samples, as well as optimization of the thermo-mechanical treatment, are currently in progress.

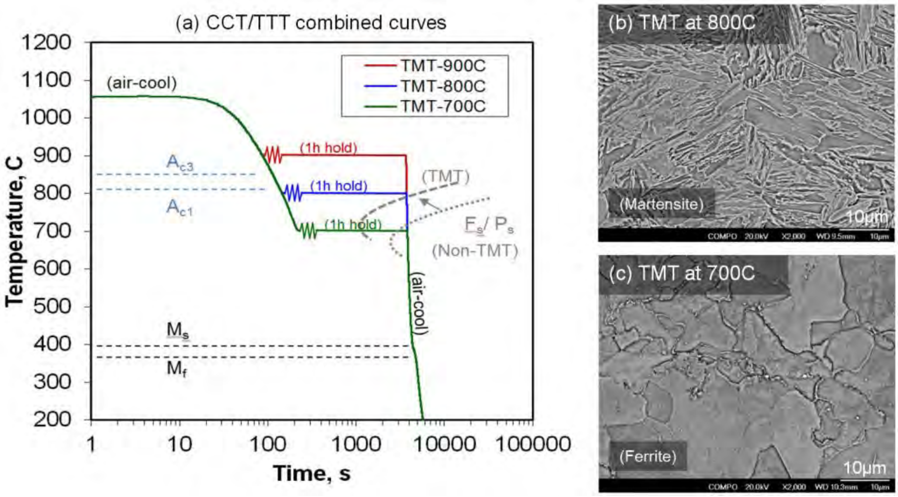

Figure 7. (a) CCT/TTT combined diagram showing the experimental heat history of aus-forging and aus-aging, together with the estimated the C curves of ferrite formation with and without aus-forging; (b) and (c) SEM-back scattered electron images of the aus-forged/ aus-aged sampled processed at 800 and 700°C, respectively.

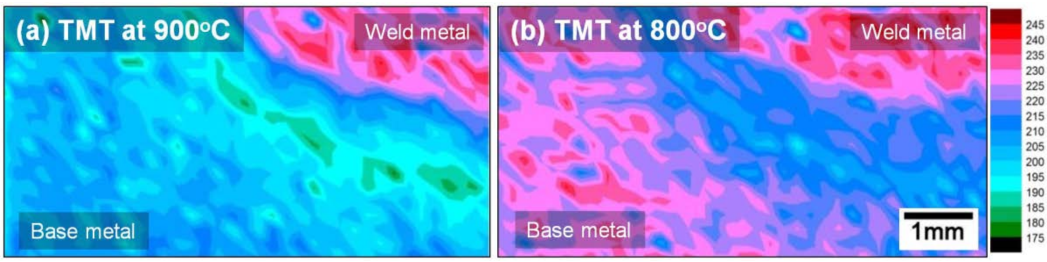

Figure 8. Hardness color maps of TMT samples after GTAW and PWHT showing the hardness distribution across the weld metal and the base metal; (a) TMT processed at 900°C and (b) at 800°C prior to the welding.

CONCLUSIONS

Significant improvement of creep properties of the modified 9Cr-1Mo (Grade 91) steel weldment was observed by applying non-standard pre-weld heat treatments (lower temperature pre-weld tempering), compared to the standard heat treated sample weldment. Controlling $M_{23}C_6$ refinement after PWHT at the FGHAZ is the key for improving the creep properties, although lower temperature pre-weld tempering sacrifices the room temperature ductility of the base metal. Two different thermo-mechanical treatments, two-step tempering and aus-forging/aus-aging, were attempted to control the refinement or dense dispersion of $M_{23}C_6$ and MX, respectively. Optimization of the processing routes is currently in progress.

ACKNOWLEDGMENTS

The authors thank Mr. Tom Muth for his review and comments on this manuscript, and Dr. Pete Tortorelli for his thoughtful supports on the project. Research sponsored by the U.S. Department of Energy, Office of Fossil Energy, Fossil Energy Advanced Research Materials Program, under contract DE-AC05-00OR22725 with UT-Battelle, LLC. ORNL's Shared Research Equipment (ShaRE) User Program, sponsored by the Office of Basic Energy Sciences, the U.S. Department of Energy, is also acknowledged.

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