## **RESEARCH PAPER**

# Effect of post-weld heat treatment (PWHT) time and multiple PWHT on mechanical properties of multi-pass TIG weld joints of modified 9Cr-1Mo steel

H. C. Dey • S. K. Albert • A. K. Bhaduri • G. G. Roy • R. Balakrishnan • S. Panneerselvi

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Abstract The effect of post-weld heat treatment (PWHT) time and multiple PWHT on mechanical properties of modified 9Cr-1Mo steel weld joints has been studied using multipass tungsten inert gas (TIG) weld joints fabricated from 12.5mm-thick plates using matching composition ER90S-B9 filler wire. Radiographically qualified weld joints were subjected to single PWHT at 760 °C for different hold times, viz. 1, 3, 4, 8 and 12 h, as also to multiple PWHT cycles of (1 h+3 h) and (4 h+4 h) for comparing with single PWHT of 4 h and 8 h. respectively. Transverse-weld tensile strength of the weld joints (as also the base material) decreases marginally with increasing heat treatment duration. In all heat-treated conditions, the Charpy V-notch impact toughness of more than 200 J is obtained for the weld and base materials. Multiple PWHT is found to have no adverse effect on the tensile properties and impact toughness of the weld joint, which has been corroborated by microstructural examination and hardness measurements.

Keywords Welding  $\cdot$  Post-weld heat treatment  $\cdot$  Mechanical properties  $\cdot$  Impact toughness  $\cdot$  Microstructure  $\cdot$  Weld metal  $\cdot$  Heat-affected zone

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H. C. Dey · S. K. Albert · A. K. Bhaduri (⊠) · R. Balakrishnan · S. Panneerselvi Indira Gandhi Centre for Atomic Research, Kalpakkam 603102, India e-mail: bhaduri@igcar.gov.in

G. G. Roy Indian Institute of Technology, Kharagpur 721302, India

# **1** Introduction

Modified 9Cr-1Mo (grade 91) steel is widely used in petrochemical and power plants as a structural material for hightemperature applications, owing to its resistance to hightemperature creep deformation, oxidation and stress corrosion cracking in caustic/chloride environment [1, 2]. This steel is used in the normalised (1040-1080 °C) and tempered (730-780 °C) condition [3], where the hard and brittle martensite formed on normalising is tempered to improve ductility and toughness by formation of fine Nb(C,N), V(C,N) and M<sub>23</sub>C<sub>6</sub> precipitates along prior-austenite and martensitic-lath boundaries. Welding thermal cycle introduces gradients of microstructure in the heat-affected zone (HAZ) of 9-12 % Cr ferritic-martensitic steel weld joints. The HAZ is broadly classified according to the extent to which grain growth and austenitisation occur and is referred to as coarse grain HAZ (CGHAZ) adjacent to weld fusion line that is heated above 1100 °C; fine grain HAZ (FGHAZ) that is heated between the upper critical temperature (Ac<sub>3</sub>) and 1100 °C; intercritical HAZ (ICHAZ) that is heated between the lower critical temperature (Ac1) and Ac3; and over-tempered base metal. One of the important high-temperature service-related problems in 9-12 % Cr steel weld joints is their premature failure in the FGHAZ/ICHAZ owing to increased rate of creep void formation that leads to type IV cracking. It has been reported [4] that for 9–12 % Cr steel, the shift in fracture location from the base metal to the HAZ in cross-weld creep tests is dependent on both the applied stress and temperature. The applied stress has a greater influence than temperature, with applied stress less than ~120 MPa being expected to result in type IV failure [4]. However, controlled addition of boron (of 90-130 ppm) to 9-12 % Cr steel has been found to have a beneficial effect in improving resistance to type IV cracking [4].

Welding of grade 91 steel needs special attention because of its tendency to undergo hydrogen-assisted cracking (HAC)

Chemical composition (in wt %)												
С	Mn	Р	S	Si	Ni	Cr	Мо	Al	Nb	V	Ν	Fe
0.114	0.403	0.014	0.0008	0.309	0.22	8.838	0.860	0.013	0.08	0.207	0.0538	Bal.
Tensile properties (at room temperature)										Impact toughness		
Specimen location				0.2 % YS (MPa)		UTS (MPa)		Elongation (%)		At 18 °C (J)		
Rolling direction Transverse direction				548 552		712 718		24.6 25.8		190–211		
ASME specification				415 (min)		585-760		18.0		_		

Table 1 Chemical composition and mechanical properties of modified 9Cr-1Mo steel plate

in the weld and HAZ and also the low-weld metal toughness in as-welded condition. Avoiding HAC requires suitable choice of preheat and interpass temperatures as well as postheating after welding. The poor as-welded impact toughness of the weld and HAZ necessitates mandatory post-weld heat treatment (PWHT) at 730-775 °C. For this steel, poor toughness has been found in welds produced by flux-based processes, like shielded metal arc, submerged arc, flux-cored arc welding, etc.; for example, impact toughness of flux-cored arc welds can be less than 65 J compared to base material toughness of 227 J [5]. However, multi-pass tungsten inert gas (TIG) welds exhibit higher toughness than the base material and activated-flux TIG (A-TIG) welds [6, 7]. A study on the impact toughness of HAZ of grade 91 steel, using heat treatments simulating the weld thermal cycle and 750 °C/1 h PWHT, has shown that the highest upper-shelf energy and lowest ductile-to-brittle transition temperature occur in the inter-critical HAZ, and the lowest toughness occurs in the coarse-grained HAZ adjacent to the weld fusion line [8]. A study on the mechanical properties and hydrogen embrittlement (HE) of notched specimens of grade 91 base metal and its laser welds [9] has shown that martensitic structure and coarse grains are more prone to HE. However, impact toughness and resistance to HE increase significantly on tempering at 750 °C [9]. During the solidification of 9-12 % Cr steel welds, there is a tendency to form delta-ferrite ( $\delta$ ) because of incomplete  $\delta \rightarrow$  austenite ( $\gamma$ ) transformation due to the rapid cooling depending on the welding process and weld metal chemistry [10, 11]. The presence of  $\delta$ -ferrite in the weld is reported to impair the impact toughness, promote brittle sigma-phase formation and reduce creep ductility at elevated temperatures. Therefore, the development of 9-12 % Cr steel welding consumable aims at elimination of retained  $\delta$ -ferrite by suitable optimisation of the weld chemistry [10]. Further, in 12Cr-1Mo-0.3V (HT9) steel weld joints, δ-ferrite is present even in the CGHAZ as this region is heated to 1200-1370 °C corresponding to the  $\delta + \gamma$  phase field resulting in localised reduction in hardness compared to the fusion zone and the adjacent HAZ regions [11]. In situ synchrotron X-ray diffraction monitoring of phase transformation in 9Cr-3W-3Co-V- Nb steel subjected to simulated HAZ thermal cycle with 1300 °C peak temperature, at different heating rates (of 10 and 100 K s<sup>-1</sup>) but same cooling conditions, showed that at 1250 °C, 65 %  $\delta$ -ferrite is formed at the slower heating rate (10 K s<sup>-1</sup>) while 45 %  $\delta$ -ferrite is formed at the faster heating rate (100 K s<sup>-1</sup>) [12]. However, after these samples (for both the heating rates) are cooled to room temperature, 4 % each of  $\delta$ -ferrite and austenite are found to be retained.

Grade 91 steel weld joints fabricated by multi-pass TIG welding exhibit very good weld metal impact toughness because the weld metal is almost free of inclusions and has low oxygen content. Hence, the TIG welding process is preferred in applications requiring very high-quality welds. In the Indian 500 MWe Prototype Fast Breeder Reactor, grade 91 steel is used as the material for construction of the steam generators [13, 14], with the recommended process being multi-pass TIG welding. Fabrication of large components of grade 91 steel, like steam generators in nuclear power plants, involves a large number of weld joints of various thicknesses. To avoid HAC and achieve required toughness and ductility, PWHT is given to each of these welds locally immediately after welding as also to the entire welded component to restore mechanical properties [1]. Additionally, in case of weld repair or repair welding of cracked components of grade 91, PWHT is mandatory after repair welding. In such cases, the weld and base materials are subjected to second PWHT after repair welding. Hence, it is important to study the effect of PWHT time and



Fig. 1 Schematic of the modified 9Cr-1Mo weld joint



Fig. 2 Schematic of the single and multiple heat treatment cycles

multiple PWHT on the mechanical properties of grade 91 steel weld joints, which in this work has been made for multi-pass TIG weld joints.

### 2 Experimental

Grade 91 steel plates of 12.5-mm thickness in normalised (1050 °C/12.5 min and air-cooled) and tempered (780 °C/12.5 min and air-cooled) and tempered (780 °C/12.5 min and air-cooled) condition were used in this study; the chemical composition and the tensile and impact properties are given in Table 1. Weld pads were fabricated by multi-pass TIG welding using single-V joint geometry of 70 ° included angle (Fig. 1), matching composition ER90S-B9 filler wire and preheat and interpass temperature of 250 °C. After radio-graphic examination, the weld joints were subjected to single PWHT at 760 °C for different hold times, viz. 1, 3, 4, 8 and 12 h, and also to multiple PWHT cycles of (1 h+3 h) and (4 h+4 h) for comparing with single PWHT of 4 h and 8 h, respectively (Fig. 2). For comparison, the base material was also subjected to the same heat treatment cycles.

Transverse tension test specimens (of 5-mm diameter and 25-mm gauge length), fabricated from the base materials and weld joints in all the heat-treated conditions, were tested at room temperature using two specimens for each condition

Fig. 3 Variation of a yield strength and b tensile strength with heat treatment time



#### **3** Results and discussion

The results of tensile tests at room temperature (Fig. 3) show that the yield strength (YS) and ultimate tensile strength (UTS) of the weld joint in as-welded condition are marginally higher (by 10–15 MPa) than the as-received base material due to restraint effect of the high-strength martensitic structure in the weld and HAZ. In contrast, after PWHT, the YS and UTS





of the weld joint are lower than the base material subjected to corresponding heat treatment cycle. Both the YS and UTS of the weld joint and base material decrease gradually with an increase in heat treatment time up to 4 h, beyond which there is no significant change/degradation in strength. This indicates that maximum tempering/ageing is achieved in about 4 h at 760 °C. Multiple PWHTs of (1 h+3 h) and (4 h+4 h) do not have any adverse effect on strength of the weld joint and base material after multiple PWHT, especially after (1 h+3 h), is marginally higher than that after the single PWHT for 4 h. The ductility of the weld joint (Fig. 4) in all heat-treated conditions



Fig. 5 Tensile-tested samples of weld joints showing fractured location: a typical photograph of failed samples, b for PWHT of 1 h and c for PWHT of 12 h

is comparable with the base material. Figure 5a shows a typical failure location for transverse weld tensile test samples in the as-welded and all the PWHT conditions. To find out the exact failure locations, failed samples were metallographically polished along the length and etched using Villella's reagent. Figure 5b, c shows the macrograph of etched samples and failure location for PWHT time of 1 h and 12 h, respectively. It is clear that tension test samples were fractured in the base metal adjacent to heat-affected zone. These tensile properties of the weld joint and base metal in all heat-treated conditions are acceptable as per ASME section IX requirements (Table 1), with no significant reduction in strength of the weld joint and base material with increase in heat treatment time even up to 12 h.

The minimum toughness value obtained from the three impact tests carried out for each condition was considered here as it is the most conservative value (Fig. 6). In the as-welded condition, the weld has a low impact toughness of 77 J with 0.9 mm lateral expansion, due to its hard and brittle martensitic structure. However, PWHT for 1 h significantly increases the weld toughness to 264 J due to tempering and softening of the martensite. The impact toughness of weld is about 25–50 J higher than that of the base material in all the



Fig. 6 Variation in Charpy V-notch impact toughness at  $18 \text{ }^{\circ}\text{C}$  with heat treatment time (0 h refers to as-received base material and weld joint in as-welded condition)

Fig. 7 Microstructures of a base metal, b intercritical HAZ, c coarse grain HAZ near fusion line and d weld metal in the as-welded condition weld joint



heat-treated conditions, except after the multiple (1 h+3 h) PWHT when the toughness of the weld is marginally lower than the base material. The scatter in the weld toughness values with PWHT time can be attributed to the variation in the local microstructure ahead of notch tip. However, in all the heat-treated conditions, the base material and weld metal have

impact toughness values greater than 200 J and very good lateral expansion of 1.85–2.40 mm.

The microstructure of the base metal (Fig. 7a) shows the presence of tempered martensite within prior-austenite grains and fine precipitates along prior-austenite grain boundaries and martensite lath boundaries. The microstructure of the

Fig. 8 Microstructures of a base metal, b intercritical HAZ, c coarse grain HAZ near fusion line and d weld metal in the weld joint after multiple (4 h+4 h) PWHT



intercritical HAZ (Fig. 7b) shows that the weld thermal cycle has transformed some of the original tempered martensite laths to fine, equiaxed austenite grains, while some of the original prior-austenite grain boundaries with substantial amount of fine precipitates are also present. The presence of prior-austenite grain boundaries and incomplete dissolution of original precipitates in the intercritical HAZ indicates that during welding, incomplete transformation has occurred as the upper critical  $(Ac_3)$  temperature has not been exceeded. The microstructure of the HAZ close to fusion line shows transformation of the tempered martensite to fine equiaxed grains of austenite due to the welding heat (Fig. 7c), with no clear evidence of the presence of the original prior-austenite grains indicating thereby that there has been almost complete dissolution of the original fine precipitates along with complete transformation of tempered martensite to austenite. The microstructure of the weld metal (Fig. 7d) consists of martensite laths within prior-austenite grains, with the prioraustenite grains being coarser compared to that in the base material. It is important to note here that the microstructure of the weld metal in multi-pass welds of grade 91 steel is heterogeneous from the root pass to final pass, with each subsequent welding pass creating a HAZ in the weld metal of previous pass. Consequently, a banded microstructure is observed across the multi-pass TIG weld, similar to that also observed in multi-pass submerged arc welds [16]. The microstructure and properties of weld metal HAZ are different from that of the base metal HAZ due to differences in their original microstructures. Due to this banded/heterogeneous structure in the multi-pass weld metal, the mechanical properties vary from point to point within the weld metal itself as reported earlier [16] and confirmed by significant variation in measured hardness of as-welded weld metal presented subsequently in this paper.

After PWHT, the microstructure of the intercritical HAZ (Fig. 8b) shows the presence of fine precipitates developed during the heat treatment, while that of the coarse grain HAZ (Fig. 8c) shows completely tempered martensite with the prior-austenite grains being finer than in the base metal. The microstructure of the weld metal after PWHT (Fig. 8d) shows tempered martensite with significant amount of fine precipitates that contribute to the reduction in hardness and strength of the as-welded weld metal accompanied by an increase in ductility and toughness.

The hardness profile of the weld joint in as-welded condition (Fig. 9a) shows that the hardness of the weld metal varies across the weld joint by about 60 Vickers hardness number (VHN) due to microstructural variations as discussed earlier. The hardness of the as-welded HAZ near the fusion line is similar to the hardness of the weld metal indicating complete dissolution of tempered martensite to austenite during weld heating cycle followed by transformation of the austenite to martensite during the weld cooling cycle. In the as-welded



Fig. 9 Hardness variation across the weld joint in **a** as-welded condition and after single PWHT and **b** after multiple PWHT in comparison with single PWHT

joint, the hardness reduces gradually from that in the HAZ to that in the base metal due to partial/incomplete transformation of the tempered martensite during the weld thermal cycle. The hardness profiles across the weld joint after PWHT for different duration (Fig. 9a) show that the hardness of the weld metal and the high hardness of the HAZ in the as-welded condition reduce significantly due to tempering of the martensite, which also results in an increase in toughness of the weld metal. It is also evident that PWHT for 1 h is adequate for the restoration of mechanical properties from that in the as-welded condition and, in general, an increase in duration of PWHT results in only marginal reduction in hardness. It is also observed that on PWHT, there is a drop in hardness in the over-tempered region of base metal and in the intercritical HAZ. However, this drop in hardness is very limited and does not significantly alter or reduce the tensile properties of the weld joints, with the transverse-weld tensile properties meeting the ASME section IX requirements.

The hardness profile after multiple PWHT compared with the single PWHT of same duration (Fig. 9b) shows a hardness variation across the weld metal, HAZ and base metal of about 25 VHN, and there is no significant change in hardness distribution due to multiple PWHT.

# 4 Conclusions

The following are the important conclusions from this study on the effect of duration of PWHT and multiple PWHT on mechanical properties of multi-pass TIG-welded modified 9Cr-1Mo steel using matching filler wire:

- 1. Transverse-weld tensile strength of weld joints decreases by 25–30 MPa with an increase in PWHT duration from 1 to 12 h. The ductility of the weld joint in all heat-treated conditions is comparable with the base material. Tensile properties of base material subjected to the same heat treatment cycle as that of the weld joint show marginally higher tensile strength compared to weld joint. Multiple PWHT does not have any adverse effect on tensile strength and ductility. Transverse-weld tension test samples in as-welded and after PWHT were fractured in the base metal adjacent to heat-affected zone. The tensile properties of weld joint and base material for all the heat treatment conditions studied meet the ASME section IX requirements and are acceptable.
- 2. Charpy V-notch impact toughness at  $18 \,^{\circ}$ C of the weld metal and base metal after all PWHTs studied (including the multiple PWHTs) is more than 200 J, with lateral expansion of  $1.85-2.40 \,$  mm.
- 3. Hardness across the weld joints after PWHT shows that PWHT for 1 h is adequate to restore the mechanical properties from that in the as-welded condition. Also, the multiple PWHTs did not have adverse effect on the hardness distribution across the weld joint.
- 4. Microstructural examination of the different regions across the weld joint in the as-welded condition and after PWHT showed that the phase transformations taking place across the weld joint during welding and after

PWHT correlate well the variations in the mechanical properties including hardness.

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