



# Effect of post weld heat treatment soaking time on microstructure and mechanical properties of TIG welded grade 91 steel

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## Abstract

Development of new alloy materials is in progress to improve thermal efficiency of supercritical boilers. One of such material is modified 9Cr-1Mo (Grade 91) martensitic alloy steel referred as T/P91. This material is extensively used in high temperature applications such as fabrication of superheater, reheater and economizer sections of a boiler. The present study is made to find the effect of post weld heat treatment (PWHT) soaking time on microstructure and mechanical properties of TIG welded ASTM A213 Grade 91 steel plate. Experiments were conducted for PWHT at 760°C for different soaking time such as 2, 4 and 6 hours to get the desired mechanical properties. The investigated results suggest that PWHT of 2 hours at 760°C is optimal to regain the strength of Grade 91 steel after welding.

## 1. Introduction

The thermal efficiency of High-Pressure Boiler Plants (HPBP) is intensely depends on steam temperature and pressure. So it is essential to develop such advanced material which can withstand steam temperature of 500 - 600°C and pressure of 180 - 300 bar [1]. In order to suit this requirement, advanced materials should have enough strength, good oxidation and corrosion resistance at elevated temperature. This led to the development of 9Cr-1Mo steel in Oak Ridge National laboratory in 1970. This material allows high operating temperature (550 - 650°C) and improves the corrosion resistance [2,3].

After welding, high hardness values were obtained in the heat affected zone (HAZ) and weld zone than base metal. This high hardness is due to occurrence of untempered martensite in the weldment at high cooling rate of operation. It is hard and brittle in nature and it will have high hardness level [4]. However, these differential hardness levels result in premature failure and also high hardness welds may lead to stress corrosion cracking (SCC) in the presence of the moisture. Moreover, the impact strength of as-welded condition does not exhibit adequate resistance which further results in crack initiation at high temperature service operation [5,6]. This demands the use of PWHT to ensure the desired material properties. If the PWHT is carried out at suitable temperature and time, the welded joint exhibits acceptable mechanical properties [7,8].

PWHT is necessary to improving material service life and to ensure adequate toughness during hydrostatic

testing. The PWHT process should be performed within the range of 1350 - 1420°F (730 - 770°C). The maximum temperature at any point in the PWHT process should not exceed 1420°F (770°C) which is below lower critical temperature for Grade 91 type materials [9]. The lower critical temperature is indicated by the A<sub>1</sub> line in the iron-carbide diagram, it is the temperature at which austenite (γ-Fe) to pearlite (ferrite (α-Fe) + cementite (Fe<sub>3</sub>C)) transformation on cooling, below this temperature austenite does not exist. If this temperature exceeds, Grade 91 material shows an erratic behavior [5]. However, high-performance Cr-Mo steels develop their properties by means of tempering process. This results in the precipitation of carbides which provides superior elevated-temperature performance characteristics to these materials. If the lower critical temperature exceeds, the carbide matrix is destroyed and the material loses its elevated temperature strength. And further not possible to reform tempered microstructure using local heating. As can be referred to ASME Section VIII, the minimum holding temperature during PWHT of Grade 91 weld is 730°C with a minimum holding time of 1 hour per inch thickness. Though, if the PWHT temperature is too low, the weld joint displays insufficient toughness due to unsatisfactory tempering effect. At the same time, if the PWHT temperature is too high, the tensile strength at ambient and elevated temperatures becomes inadequate due to over tempering effect [10,11].

**Table 1.** Chemical composition of ASTM A213 Grade 91 steel.

Composition (wt. %)											
C	Mn	Si	P	S	Cr	Mo	V	Nb	Ni	N	Al
0.11	0.47	0.32	0.014	0.003	8.50	0.85	0.22	0.076	0.13	0.038	0.018

**Table 2.** Chemical composition of AWS ER90S-B9 electrode.

Composition (wt. %)											
C	Mn	Si	P	S	Cr	Mo	V	Nb	Ni	N	Al
0.09	0.49	0.20	0.004	0.003	8.70	0.90	0.19	0.08	0.66	0.07	0.006

**Table 3.** Mechanical properties of A213 Grade 91 steel as per ASTM.

Yield strength (MPa)	Tensile strength (MPa)	Hardness (HV)	Elongation (%)
≥ 415	≥ 585	265	≥ 20

Much research work was conducted in the Hybrid weld joints (combination of multiple welding). However, the amount of literature available on complete TIG weld joint is limited and hence it requires further investigation. Therefore, an effort has been made to review the comparative study of microstructural characteristics and mechanical properties of TIG welded Grade 91 steel at different PWHT soaking conditions. In this present study, Grade 91 plates of 15 mm thickness have been welded and subjected to PWHT at a constant soaking temperature of 760°C with different conditions of soaking time such as 2, 4 and 6 h. The major focus is given to impact toughness, hardness and microstructure characteristics of the weld joints.

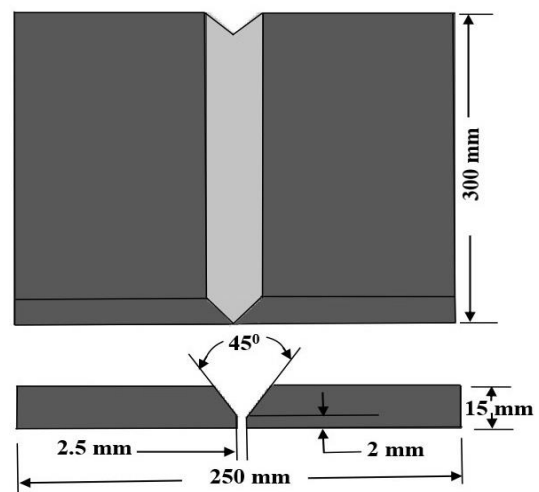
## 2. Experimental

For the present work, the base material was taken from ASTM A213 (ASME SA213) Grade 91 steel plate with a thickness of 15 mm and electrode of AWS ER90S-B9 having diameter of 1.2 mm was used. In order to get the desired properties of weldment, Grade 91 base metal was welded with similar chemical composition of electrode. In the present study ER90S-B9 was selected as a filler rod due to similar composition of Grade 91 base metal. The chemical compositions of ASTM A213 Grade 91 steel [12] and AWS ER90S-B9 electrode [13] is given in the Table 1 and 2 respectively. Table 3 shows mechanical properties of A213 Grade 91 steel as per ASTM standard [12]. For welding, Grade 91 steel plate has been cut into a size of 300 × 125 × 15 mm by machining process, and these specimens are welded with a root gap of 2.5 mm, root face of 2 mm and a groove angle of 45°. Figure 1 shows schematic view of weld bead geometry and Figure 2 (a) and 2 (b) shows single V-groove butt joint preparation before and after welding respectively.

The entire weld joint was made using TIG welding process in 07 layers and 15 weld passes with an

average heat input of 1.50 kJ·mm<sup>-1</sup>. The welding process parameters are given in Table 4.

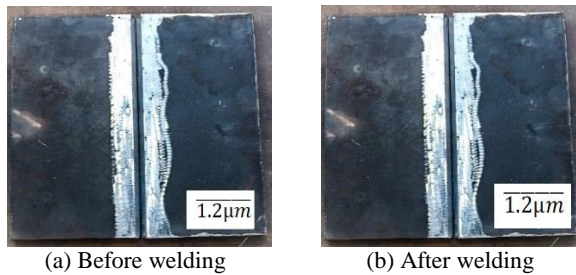
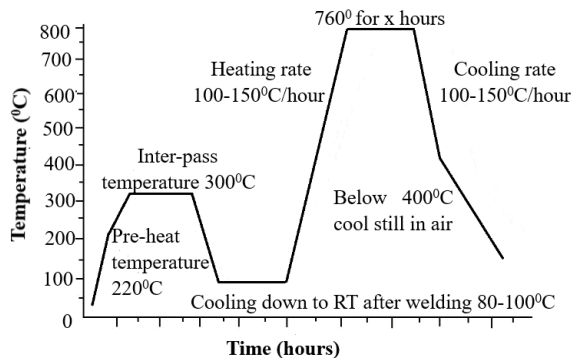
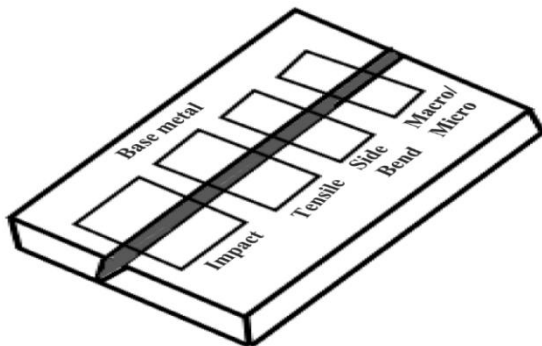
Four plates of 15 mm thickness have been welded with different specimen configurations as-weld, 2, 4 and 6 h soaking. After welding, the quality of the joint was assessed by using X-ray radiography test as per ASME Sec V code [12]. Weld joints free from any imperfections were considered for PWHT. Grade 91 plates of 15 mm thickness have been welded and subjected to PWHT at a constant soaking temperature of 760°C with different conditions of soaking time such as 2, 4 and 6 h. PWHT cycle adopted for this study as shown in Figure 3 [6, 14]. After post-treatment, the weld bead was cut in order to analyze the weldment for tensile test, hardness test, impact test, side bend test and microstructural study. From each plate, two specimens for tensile test, five specimens for Charpy impact test, one specimen for side bend test and one specimen for both macro-hardness test and microstructure analyses have been taken in the fashion shown in Figure 4.



**Figure 1.** Schematic view of weld bead geometry.

**Table 4.** Process parameters of TIG welding.

Current type	DCEN
Welding Current	150 A
Voltage	12 V
Welding speed	1.20 mm·sec <sup>-1</sup>
Weld geometry	Single V-Groove Butt Joint
Power	1800 Watts
Heat input	1500 (J·mm <sup>-1</sup> )
Total number of passes (Root, Hot and Filler)	15

**Figure 2.** Single V-groove butt joint specimen of Grade 91 steel plates.**Figure 3.** PWHT cycle for Grade 91 steel.**Figure 4.** Schematic illustration of test specimen locations in the weld plate after PWHT.

The tensile test was carried out to ensure the quality of the weld. The specimen dimension for the

tensile test was as per AWS B4.0 [13]. The bend test was carried out to evaluate both the ductility and soundness of the weld joint. The bend test was conducted on UTM with 180° bend angle according to AWS B4.0 procedure [13]. The Vickers Hardness test was carried out as per ASTM E92 (2003) procedure. The hardness values were taken from each position of the weld bead horizontally (base metal, HAZ and weld zone) from the center of the weld to either side.

The Charpy impact test was conducted to analyze the ability of different microstructures to absorb energy during the process of fracture. The specimen dimension for impact test was according to AWS B4.0 (2015). The Charpy impact test specimens of size 55 × 10 × 10 mm have been cut from the transverse cross section of joints, with the notch located at the center of the weld.

For the microstructural study the specimen was milled, ground, polished and then etched using the Vilella's reagent (1g picric acid, 5 ml HCl, 100 ml methanol) and inspected under the metallurgical microscope. Scanning Electron Microscopy (SEM) analysis was carried out for impact tested specimens to examine the detailed information on the mechanism of fracture by microscopic examination of fracture surfaces.

### 3. Results and discussion

#### 3.1 Mechanical properties of grade 91 steel

##### 3.1.1 Tensile test

Variation of UTS with PWHT at 760°C for a different soaking time as shown in Figure 5. As-welded specimen has a maximum value of Ultimate Tensile Strength (UTS) due to the occurrence of  $\delta$ -ferrite in the weldment [8] [10,11]. The formation of  $\delta$ -ferrite is an important phenomenon during welding of martensitic (9Cr) steels [5]. During welding, the corresponding weld joint is heated to solidification temperature and reaches its melting point. In the heat affected zone (HAZ) and weld zone it is indeed possible to form small amount of  $\delta$ -ferrite at high peak temperatures [15]. This formation of  $\delta$ -ferrite could be the reason for as-welded specimen has a maximum value of UTS [16]. And there is a slight decrease in UTS value was observed after PWHT with different soaking time. However, it can consider as marginal only. This is due to rapid cooling of austenite after PWHT (at 760°C, within the austenitic region) with different soaking time [15]. It has been observed that all the tested specimens (as-welded and PWHT with different soaking time) were found to fracture at a base metal position which ensures weld joint is strong.

##### 3.1.2 Hardness test

Differences of hardness values throughout the weld cross section of all the four different configuration

specimens (as-welded and PWHT at 760°C for 2, 4 and 6-h soaking time) as shown in Figure 6.

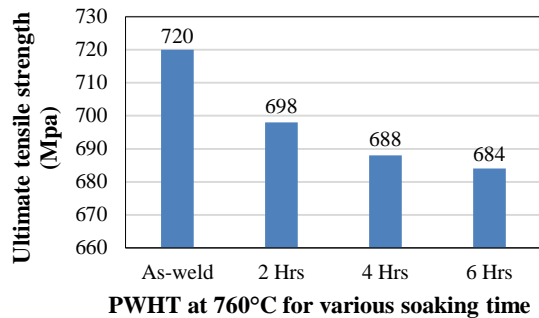


Figure 5. UTS of specimen at PWHT 760°C for various soaking time.

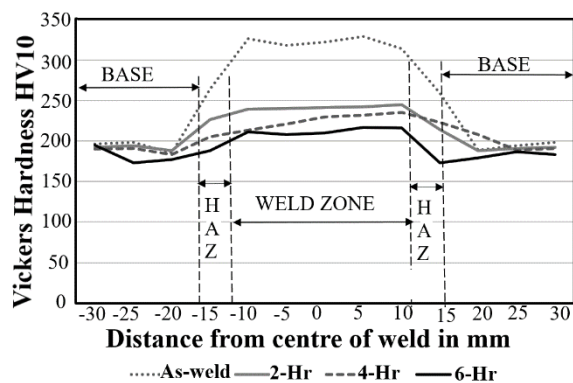


Figure 6. Hardness values along cross section of the weld metal.

After welding, high hardness values were obtained in the heat affected zone (HAZ) and weld zone than base metal. This is due to the occurrence of alloying element which produces martensite in weld zone and HAZ [4]. At this hardness level, if hydrogen entraps in the weldment, it can produce hydrogen induced crack (HIC) [4]. During welding due to high solidification temperature,  $\delta$ -ferrite forms in the weldment [4]. This occurrence of  $\delta$ -ferrite restricts the grain size and grain growth of the weldment microstructure [4]. After PWHT (at 760°C, within the austenitic region) with an increase in soaking duration from 2 to 6 h, the hardness of Grade 91 metal weld zone and HAZ reduced due to the phase transformation from austenite to tempered martensite [4,5].

### 3.1.3 Charpy impact test

Variation of impact strength with PWHT soaking duration as shown in Figure 7. In supercritical boilers, generation of high-pressure steam at above 600°C is essential. Impact toughness of Grade 91 material is very much essential for ensuring hydro test. Impact toughness of Grade 91 weldment is dropped rapidly in as-welded condition. The weld zone shows poor toughness as compared to base metal. This may be due to  $\delta$ -ferrite formation in the martensite matrix, which is untempered martensite [11]. After PWHT (at 760°C

within the austenitic region) with increase in soaking time from 2 to 6 h, the tempering gets completed. This results tempered martensite phase formation, which is the reason impact toughness value improved from 193 to 225 Joules [11].

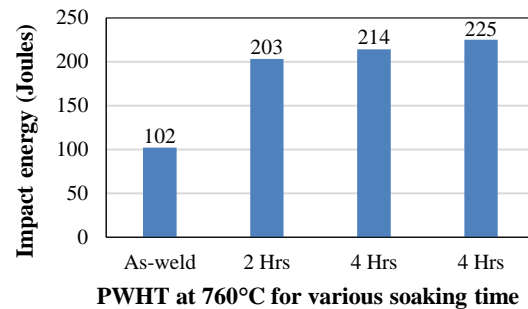


Figure 7. Impact energy of specimen at PWHT 760°C for various soaking time.

### 3.1.4 Larson-Miller Parameter (LMP)

The Larson-Miller parameter (LMP) is extensively used as extrapolation technique for predicting creep life of materials. In this technique, the Larson-Miller parameter (LMP) is empirically expressed as  $LMP = T [\log (t) + C]$  where, T is the soaking temperature in Kelvin and t is the soaking time in hours and C is the material specific constant, assumed to be value of 20 [10,17].

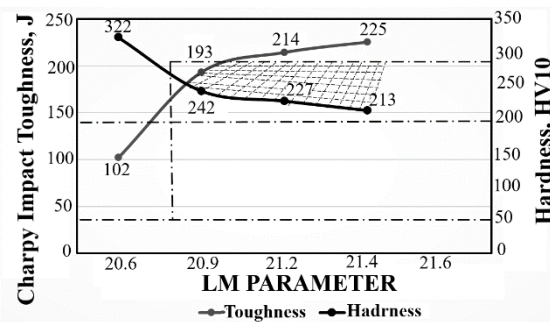
Based on the Larson-Miller Parameter (LMP), soaking time should be optimized for desired mechanical properties with constraints in impact toughness value more than 47 J (As per European specification BS EN 1599:1997, impact toughness of 47 Joules at room temperature of 20°C) is mandatory for the Grade 91 weld joint for a successful hydro test [10] and hardness band of 200 - 290 HV [9]. The average impact toughness has increased with soaking time from 2 to 6 h and it has a maximum value of 225 J with 6 h soaking time. But at this soaking time (6 h), the weld zone hardness value dropped to 213 HV and HAZ hardness value fall to 181 HV, which is lower than the parent metal. However, it should be noted that, the measurement of impact toughness only does not give a single definite trace of weld quality.

The hardness value range need to be considered for effective sound welding. Hence, the impact toughness value needs to be optimized for a particular hardness band [9,10]. After PWHT process, it is mandatory that hardness level of a Grade 91 material should be in the range of 200 - 290 HV. This hardness band helps to achieve required mechanical characteristics of weldment i.e. increase in impact toughness. So, the soaking time should be optimized with constraints in impact toughness value more than 47 J and weld zone hardness band between 200 HV and 290 HV, which in this study is satisfied by 2, 4, and 6 h of soaking time. Considering the soaking temperature and time duration, the Larson-Miller Parameter (LMP) has been calculated and shown in Table 5.

**Table 5.** Calculation of Larson-Miller Parameter.

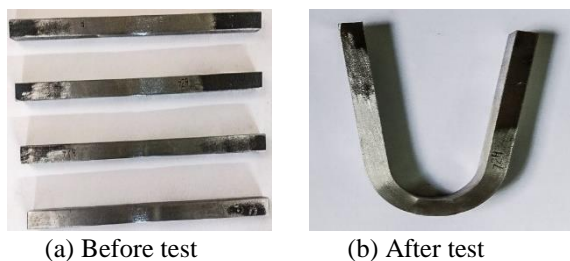
Work Samples	$LMP = T [\log(t) + C] \times 10^{-3}$	Larson-Miller Parameter
As-welded	$(760+273) [20] \times 10^{-3}$	20.66
2-h soaking	$(760+273) [\log(2) + 20] \times 10^{-3}$	20.97
4-h soaking	$(760+273) [\log(4) + 20] \times 10^{-3}$	21.28
6-h soaking	$(760+273) [\log(6) + 20] \times 10^{-3}$	21.46

Figure 8 shows a plot between Charpy impact toughness and hardness against Larson-Miller parameter to know the variation of the two properties with LMP. Considering a weld zone hardness band of 200 to 290 HV and toughness higher than 47J, a feasible region has been obtained.

**Figure 8.** Charpy impact toughness and hardness plotted against Larson-Miller Parameter.

### 3.1.5 Side Bend Test

All the four tested specimens (as-welded and PWHT at 760°C of 2, 4 and 6-h soaking time) displays no trace of visible cracks until the applied load angle reached 180° at a bend radius of 0.04 m. This confirms that all the welded joints exhibit optimum ductility and sound welding. Figure 9(a) and 9(b) shows specimen before and after testing.

**Figure 9.** Bend test specimen.

### 3.2 Fracture characteristics

The fractured surfaces of broken impact tested specimen are shown in Figures 10(a) - 10(d). As-welded impact fractured specimen shows mainly cleavage dominated fracture with some large size of voids (Figure 10 (a)) had an average impact energy of 102 Joules and it shows cleavage fracture with flat facets [18]. This formation of completely cleavage flat facets could be the

reason for exhibiting low impact energy in as-welded condition. After PWHT at 760°C, the impact fractured specimen shows dimple fracture with some micro voids [19]. As the soaking time increasing from 2 to 6 h, there was increase in quantity of dimples (Figure 10 (b) to 10 (d)) [5]. This is due to increase in volume fraction of fine grain boundary precipitates [5], which could be the reason for exhibiting high average impact energy from 193 to 225 Joules.

### 3.3 Microstructure characteristics

Microstructure study was conducted by using Metallurgical microscope at a magnification of 500X. The microstructure of as-received Grade 91 steel as shown in Figure 11(a) by optical microscope and Figure 11(b) by transmission electron microscope. It consists of fully tempered martensite with precipitates along grain boundaries [6]. Alloying elements promote the formation of Cr-rich  $M_{23}C_6$  precipitate (where M stands for Fe, Cr or Mo) and V-rich MX precipitate (where M stands for V or Nb and X stands for C or N) [6]. After TIG welding, base metal microstructure (Figure 12(a)) consists of the tempered martensite phase [6]. HAZ (Figure 12(b)) and weld zone (Figure 12(c)) microstructure matrix consist of untempered martensite and retained austenite (austenite that does not transform to martensite during quenching is called retained austenite) [20]. This austenite ( $\gamma$ -Fe) phase formation is due to heating of martensite at high temperature near weld zone and coarse grain microstructure was observed in weld zone than the HAZ [16,20].

After PWHT at 760°C for 2, 4 and 6 h, all the obtained microstructures were in tempered martensite (Figure 13(b), 13(c), 14(b), 14(c), 15 (b) and 15(c)), which consists of carbide precipitation along grain boundaries and there is no significant difference in microstructural characteristics of all samples [16,20]. However, the refinement of grain structure was observed after PWHT with different soaking duration [6]. Weld zone microstructure (Figure 13(c), 14(c), and 15(c)) consist of more coarse grain size compared to HAZ (Figure 13(b), 14(b), and 15(b)) and base metal microstructure (Figure 13(a), 14(a), and 15(a)) [6]. This martensite phase formed due to PWHT of austenite phase with rapid cooling. Since cooling takes place at rapid rate, insufficient time for all excess carbon to diffuse out of the crystal structure to form cementite [16,20]. Martensite is a metastable phase, when steel is heated to a temperature within the austenitic region and is then cooled, the bigger austenite ( $\gamma$ -Fe) grain structures would retransform to bigger martensite grain structures, which is tempered martensite [4].

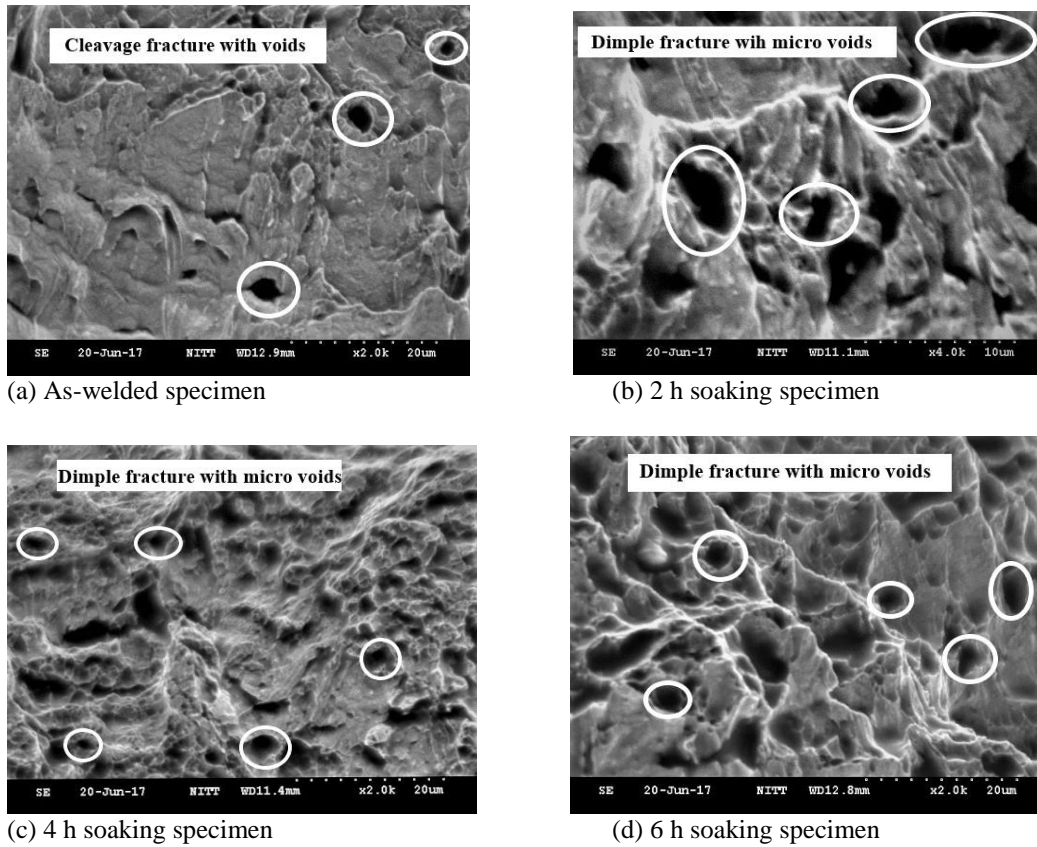


Figure 10. SEM fractograph of impact tested specimens.

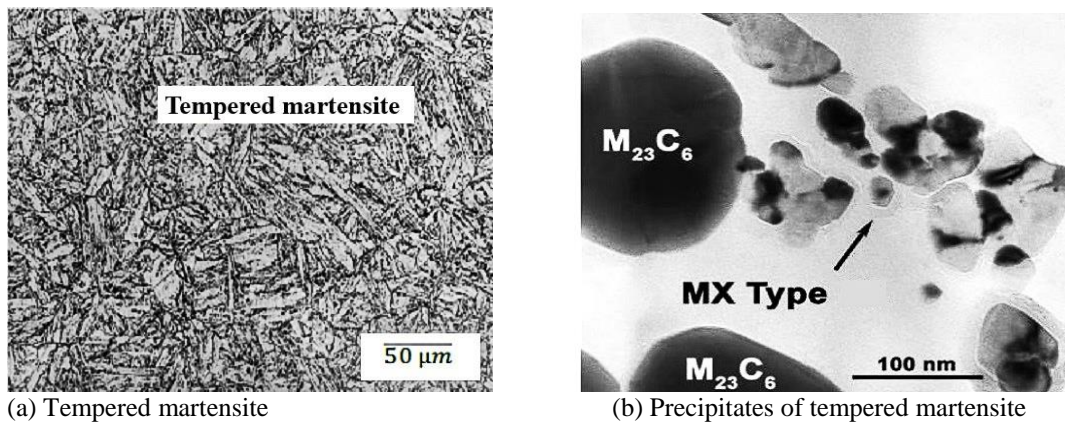


Figure 11. Microstructure of As-received specimen.

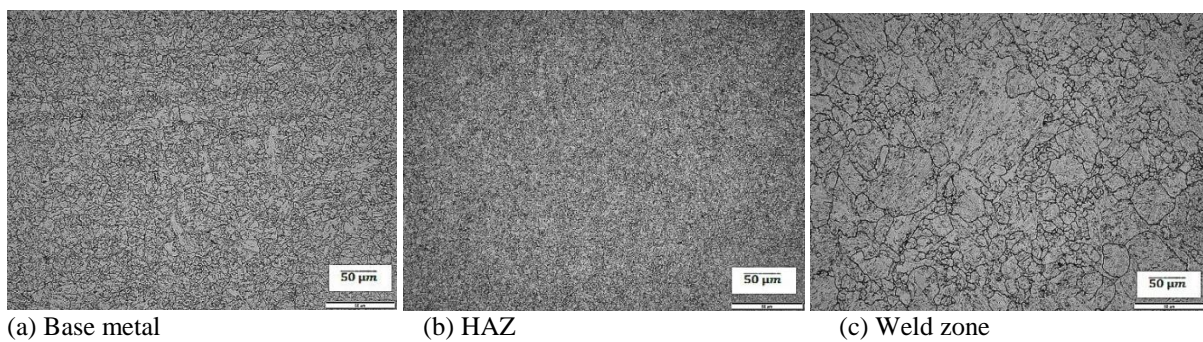
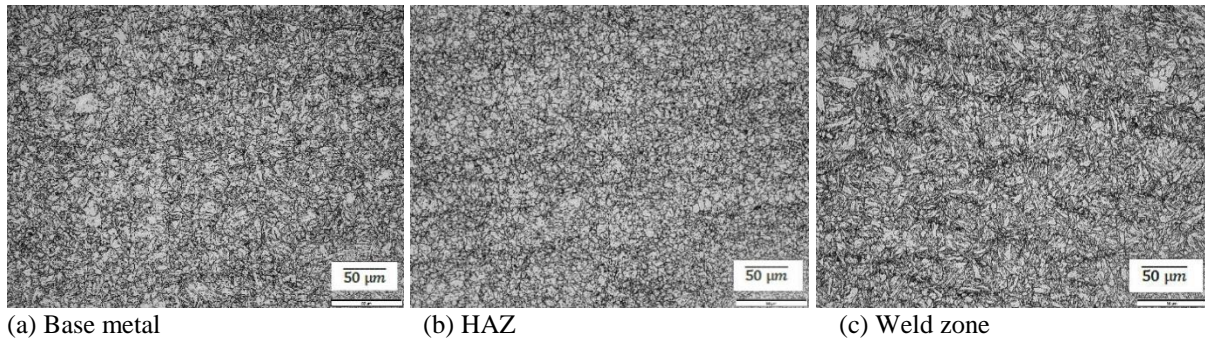
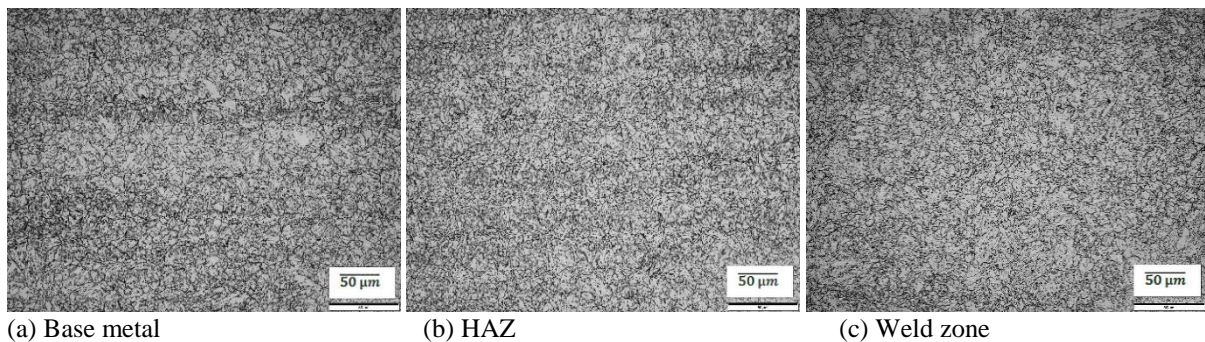


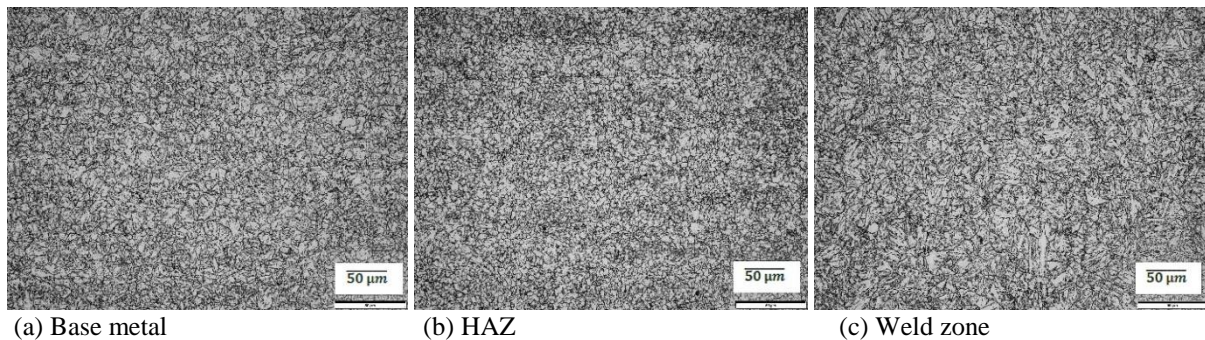
Figure 12. Microstructure of As-welded specimen.



**Figure 13.** Microstructure of 2 hour soaking specimen.



**Figure 14.** Microstructure of 4 hour soaking specimen.

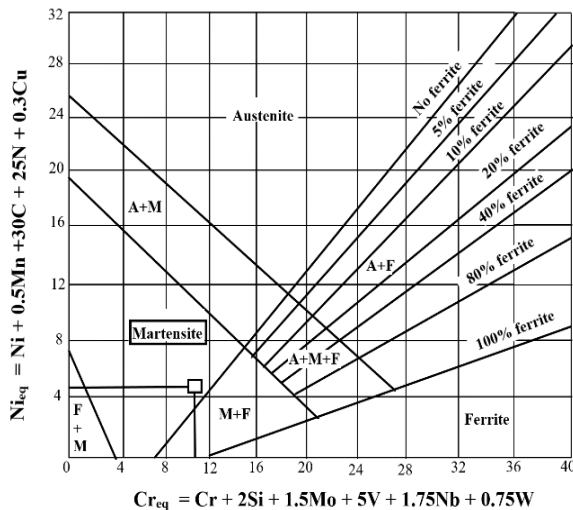


**Figure 15.** Microstructure of 6 hour soaking specimen.

### 3.4 Influence of chemical composition for predicting ferrite levels

Depending on the weight % of alloying elements in the metal, the schaeffler diagram (shown in Figure 16) provides information on the various phases (structures) present [15]. The chromium equivalent is calculated from the weight percentage of ferrite-forming elements (Cr, Si, Mo, V, Nb, W) and the nickel equivalent is calculated from the weight percentage of austenite-forming elements (Ni, Mn, C, N, Cu). The ferrite-forming tendency was evaluated by using schneider formula and obtained ferrite factor for the base metal is 7.033 [5,15,21]. From the literature study, this range of ferrite factor (<8.5) represents fully tempered martensite phase. Based on above condition and calculations, as-received Grade 91 base metal at room temperature consist of fully tempered martensite. For such low ferrite factor (<8.5), it

has been observed from literature that  $\delta$ -ferrite hardly would form during solidification of welds and which imparts high strength (hardness) and poor toughness to the weld joint [15,21]. In order to get the desired properties of weldment, Grade 91 base metal was welded with similar chemical composition of electrode. Ferrite factor of weldment may marginally change due to solidification of electrode chemical composition during welding process [15]. After welding, the obtained microstructure of Grade 91 weldment is untempered martensite and retained austenite. This confirms that weight percentage of ferrite-forming elements higher than austenite forming elements. Though electrode constitutes both ferrite and austenite forming elements, ferrite forming elements dominates in composition of weldment. Hence weldment may contain high V and Nb, which reduces toughness and increase the hardness [5,15].



**Figure 16.** Schaeffler stainless steels constitution diagram.

It has been observed from the literature that all the desirable mechanical properties and microstructural characteristics of the weldment is achieved only after post weld heat treatment with different soaking time. It has been inferred from the literature that, after welding, the strength of the weldment increased and toughness property deteriorated. In order to get the optimal values of both strength and impact toughness, the authors adopted post weld heat treatment (PWHT) process. As explained earlier in order to get the desired mechanical properties, soaking time is optimized with constraints of impact toughness value more than 47 J and hardness band of 200 - 290 HV. In present study TIG welded Grade 91 material with PWHT at 760°C of 2 h soaking time is sufficient for achieving the desired mechanical properties and this condition may not be suitable for another welding process even though same material grade is used or vice-versa. Hence the obtained mechanical properties (hardness level, impact toughness and UTS) mainly depends on the selection of material grade, welding process, type of electrode used, PWHT temperature and soaking duration.

#### 4. Conclusions

The effect of post weld heat treatment at 760°C for 2, 4 and 6-h soaking time on mechanical properties and microstructure characteristics of TIG welded Grade 91 steel by using ER90SB9 as filler metals were studied. The following conclusions are drawn.

1. Post weld heat treatment (PWHT) is essential to improve the homogeneous microstructure and mechanical properties in weld zone. PWHT process helps to reduce hardness and also impart required level of ductility in the weldment. This improves the strength and resistance to brittle fracture and increase the lifespan of material.

2. After welding, high hardness values were obtained in the heat affected zone (HAZ) and weld zone. This high hardness is due to occurrence of untempered martensite in the weldment at high cooling rate of operation. The difference in hardness levels between base metal, heat affected zone and weld zone may lead to crack initiation which further results in failure at high temperature service operation.

3. After PWHT at a temperature of 760°C, the hardness of Grade 91 material weld zone and HAZ reduced and impact toughness increased significantly. And with an increase in PWHT holding time from 2 to 6 h, the hardness of weldment dropped whereas the impact toughness was improved. This is due to the tempered martensite phase and grain coarsening.

4. Soaking time is optimized based on the Larson-Miller Parameter (LMP) with constraints of minimum impact energy (above 47 J) and weld zone hardness band (200 to 290 HV). It is found that all the acceptable hardness values obtained only after post weld heat treatment (PWHT) at 760°C for 2 to 6 h.

5. The most suitable PWHT condition for Grade 91 TIG welded joint was 760°C of 2 h soaking duration. This condition provides the hardness value 242 HV within the hardness band and also impact energy 193 J which is above 47 J. PWHT at 760°C of 4 h soaking is best suitable for maintaining the invariable hardness levels in base metal, HAZ and weld zone. But due to the reason of economy, PWHT at 760°C of 2 h soaking is sufficient to achieve the required mechanical properties.

#### 5. Acknowledgements

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