P. Rajesh Kannan*, V. Muthupandi, B. Arivazhagan and K. Devakumaran

Microstructure and Mechanical Properties of Heat-treated T92 Martensitic Heat Resistant Steel

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Abstract: T92 samples were solutionized at 1,050 °C, 1,100 °C and 1,150 °C for 20 min and then tempered at 730 °C, 745 °C and 760 °C for 60 min. Optical microscopy studies were carried out to understand the microstructural evolution due to heat treatment. These heat-treated samples comprised of lath martensite microstructure in all the cases. Prior austenite grain size of the heat-treated samples increased with solutionizing temperature. Tensile properties were evaluated using micro-tensile samples. Hardness values of the heat-treated samples were estimated using Vickers hardness tester. Interestingly, for all the given tempering condition, the hardness values showed an increasing trend with solutionizing temperature while their tensile strength values tend to decrease. Fractograph analysis depicted that increasing the solutionizing temperature led to grain boundary decohesion.

Keywords: T92 steel, heat treatment, microstructure, hardness, tensile test, grain boundary decohesion

Introduction

In thermal power plants, improvement in efficiency and reduction in carbon dioxide emissions [1–6] can be achieved by increasing the operating parameters from the current practice, i. e. by operating at temperature and pressure exceeding 600 °C and 27 MPa, respectively [7–9]. In

view of this, selection of structural materials in power plant construction plays a vital role. The Cr–Mo steels are chosen as the candidate materials for superheater and reheater tubes in power plant applications [2]. In the family of Cr–Mo steels, plain 9Cr–1Mo (T9) is modified as T91 steel by adding Nb: 0.08 wt %, V: 0.2 wt % and N: 0.04 wt % and partially substituting C by N for withstanding high temperature and high pressure. Further, T91 steel is modified to T92 steel by the addition of alloying elements like tungsten (1.8 wt %) and boron (0.005 wt %), and by reducing the amount of molybdenum from 1 wt % to 0.5 wt % [10–12]. Addition of tungsten slows down the recovery process in martensite and thereby reduction in dislocation density, which improves strength at high temperatures. Moreover, tungsten acts as a solid solution strengthening agent and stabilizes $M_{23}C_6$ carbides [2, 8, 12, 13].

T92 steel is a preferred material for applications involving operating temperatures that do not exceed 620 °C [11, 14] as it exhibits higher thermal conductivity and smaller coefficient of thermal expansion than austenitic stainless steels used in high-temperature applications and also has high temperature stability to resist creep deformation [7]. Though its corrosion and oxidation resistance are equal to other 9 % Cr ferritic steels, it exhibits improved creep behaviour and high-temperature strength compared to other boiler grade ferritic steels. Upto 625 °C, creep strength of T92 steel is 20 % more than that of T91 steel and higher or equal to that of 304 austenitic stainless steels [7, 9, 15]. Owing to these excellent properties, T92 steel is used as superheater and reheater tubes, headers and main steam pipes in high-temperature applications [2]. T92 steel is commonly used in normalized and tempered condition. Tempering promotes formation of beneficial precipitates that help in enhancing the creep properties.

For industrial applications, the Cr–Mo steels are received from the manufacturer in solutionized/normalized and tempered condition. In these materials, tempering temperature plays a crucial role in the attainment of microstructure favouring the end properties of the material, as it affects the carbide distribution in the matrix. When the tempering temperature is low, diffusion

*Corresponding author: P. Rajesh Kannan, Department of Metallurgical and Materials Engineering, National Institute of Technology, Tiruchirappalli-620015, India. Tel. + 91-9843080269; Fax No. + 91-431 2500133, E-mail: rajeshmechtce@gmail.com

V. Muthupandi, Department of Metallurgical and Materials Engineering, National Institute of Technology, Tiruchirappalli-620015, India. Tel. + 91-9894050794; Fax No. + 91-431 2500133, E-mail: vmuthu@nitt.edu

B. Arivazhagan, Materials Technology Division, Indira Gandhi Centre for Atomic Research, Kalpakkam 603012, India

K. Devakumaran, Welding Research Institute, Bharat Heavy Electricals Limited, Tiruchirappalli 620014, India

kinetics are slower due to lower thermal activation phenomena and higher tempering temperature leads to the formation of untempered martensite [16].

The manufacturer of T92 steel [17] has suggested a heat treatment cycle comprising normalizing at 1,040–1,080 °C and later tempering at 750–780 °C for better strength and toughness combined with enhanced creep resistance while the temperature ranges specified by the ASME code for T92 steel are between 1,040 °C and 1,080 °C for normalising and in a wider range of temperature between 730 °C and 800 °C for tempering [18].

Wang et al. [12] performed solutionizing and tempering in the range of 1,030–1,090 °C and 750–790 °C, respectively, on P92 material for a range of soaking periods both for solutionizing and tempering and suggested that, better mechanical properties could be attained when the samples were normalized at 1,090 °C for 20 min and tempered at 790 °C for 60 min.

To meet the optimum combination of microstructure and mechanical properties, there is a need to identify heat treatment cycle(s) that may differ from the recommended standards (such as ASTM A335/A355M-11 and ASME code Sec. II) or existing industrial practice. Though the standards recommend the heat treatment cycle based only on chemical composition, there is a need to optimize the heat treatment cycle based both on chemical composition and thickness of the material. Hence, this forms the basis for present scope of work.

The objective of the present work is to identify a safe window of heat treatment operation (for T92 steel of 5 mm thickness) to achieve the prescribed mechanical properties in terms of ultimate tensile strength and yield strength. In the present study, the proposed heat treatment cycles involve solutionizing in the range of 1,050–1,150 °C and tempering in the range of 730–760 °C. Detailed microstructure analysis and mechanical properties evaluation are carried out to arrive at the optimum mechanical properties.

Experimental

Sections cut from a T92 steel plate of 12 mm thickness were hot rolled to plates of 5 mm thickness. The chemical composition of the T92 plate analysed by optical emission spectroscopy is given in Table 1. Samples of 50 mm × 18 mm × 5 mm dimensions were cut from the rolled plates and were subjected to heat treatment.

Samples were heat treated for nine different conditions in an electrically heated muffle furnace provided with a programmable temperature controller. The combinations

Table 1: Chemical composition of T92 steel.

Element	C	Si	Mn	S	P	Cr	Mo	Ni
Wt %	0.10	0.35	0.58	0.01	0.01	8.96	0.45	0.22
Element	Al	V	W	Nb	Ti	Fe	B	N
Wt %	0.001	0.21	1.86	0.05	0.006	Bal	*	*

*B and N are not detected by optical emission spectroscopy. The standard range for B and N are 0.001–0.006 % and 0.03–0.07 %, respectively.

Table 2: Heat treatment performed on rolled T92 steel samples.

Sample designation	Solutionizing temperature (°C)	Soaking period (min)	Tempering temperature (°C)	Tempering period (min)
S _A T _X	1,050	20	730	60
S _A T _Y	1,050	20	745	60
S _A T _Z	1,050	20	760	60
S _B T _X	1,100	20	730	60
S _B T _Y	1,100	20	745	60
S _B T _Z	1,100	20	760	60
S _C T _X	1,150	20	730	60
S _C T _Y	1,150	20	745	60
S _C T _Z	1,150	20	760	60

*A, B and C denote the solutionizing temperatures, i. e. 1,050 °C, 1,100 °C and 1,150 °C, respectively. X, Y and Z denote the tempering temperatures, i. e. 730 °C, 745 °C and 760 °C, respectively.

of solutionizing and tempering temperatures employed in this study are shown along with the designations assigned for the heat-treated samples in Table 2. Solutionized samples were quenched in water then followed by tempering then air cooling to room temperature.

The as-rolled and the heat-treated samples were mechanically polished using conventional metallographic sample preparation techniques and then etched with picral (2 g picric acid in 100 mL ethanol). Microstructures of these samples were observed with an optical microscope fitted with a digital image capturing facility. From the recorded images, prior austenite grain size (PAGS) was estimated by linear intercept method.

Microhardness values of the samples were measured in terms of Vickers hardness with 1-kg load and 10 s dwell time using a Wilson microhardness tester. The reported microhardness values are the average of at least 15 indentations. Flat micro tensile samples of 6-mm gauge length, 4-mm gauge width and 0.5-mm thickness were prepared by electrical discharge machining. Tensile test at room temperature was performed in a Tinius Olsen HK50S Universal testing machine at a constant crosshead speed of 0.02 mm/s.

Results and discussion

Microstructure

Microstructure of the samples solutionized at 1,050 °C, 1,100 °C and 1,150 °C and water quenched are presented in Figure 1(a), (b) and (c), respectively.

Solutionizing temperature was expected to have an effect on the grain size and the degree of dissolution of the preexisting precipitates. Effect on grain size was clearly reflected in the microstructure of the solutionized samples. PAGS increased with increase in solutionizing temperature. Fully martensitic structure was formed on quenching and the resulted lath martensitic structure was due to the low carbon content of the steel. PAGS influenced the lath width and also the martensite packet

size (Figure 1). Larger the PAGS, the coarser was the martensite packets formed.

Microstructures of the heat-treated samples solutionized at 1,050 °C, 1,100 °C and 1,150 °C and tempered at 760 °C are presented in Figure 2. The grain boundaries of the prior austenite grains are clearly delineated by the tempering process; the effect of solutionizing temperature on the PAGS is clearly visible.

During solutionizing, the extent of dissolution of the precipitates depends on the solutionizing temperature. Dissolution of $M_{23}C_6$ precipitates starts at 780 °C while only at 900 °C MX precipitates starts dissolving. Further, dissolution goes to completion at 940 °C and 1,200 °C for the respective precipitates [12]. Therefore, at the solutionizing temperatures, viz. 1,050 °C, 1,100 °C and 1,150 °C, the $M_{23}C_6$ precipitates in the as-rolled T92 steel could be completely dissolved while MX precipitates could remain

Figure 1: Microstructure of T92 steel samples solutionized at (a) 1,050 °C, (b) 1,100 °C and (c) 1,150 °C.

Figure 2: Microstructure of T92 steel samples solutionized at (a) 1,050 °C, (b) 1,100 °C, (c) 1,150 °C and tempered at 760 °C.

as partially dissolved leaving some remnant undissolved MX precipitates in the matrix [8]. Generally, the equilibrium grain size is smaller for an alloy solutionized at a lower temperature than that of solutionized at higher temperature. However, it can be expected, in the present case, remnant MX precipitates also could have contributed on grain size by preventing grain coarsening. Thus, the PAGS of the samples solutionized at three different temperatures falls in the following order: grain size at $1,150\text{ }^{\circ}\text{C} > \text{at } 1,100\text{ }^{\circ}\text{C} > \text{at } 1,050\text{ }^{\circ}\text{C}$. It is to be stated here that light optical microscope does not resolve lath width of martensite and fine precipitates (limit of resolution of light microscope is restricted to about 200 nm).

For ferritic steels, usually the tempering temperature should be below the AC_1 temperature. Tempering being a subcritical treatment does not expect to alter the PAGS. But it can lead to the occurrence of two main phenomena. First, reduction in dislocation density occurs by recovery process and formation of subgrains by the rearrangement of dislocations [5, 8, 19, 20]. Second, formation of carbide and/or carbonitride precipitates, viz. $M_{23}C_6$, MX, etc., which can enhance the strength of the alloy by precipitation hardening [10]. These precipitates promote microstructural stability at high temperature by slowing down the grain growth [19].

The precipitation sequence in Cr–Mo steels is influenced by the composition also. Sung Ho et al. [21] have indicated the sequence of precipitate formation in 2.25Cr–1Mo steel is

while in 9–12 Cr steel is

It is not clear if the same sequence is possible in the modified 9Cr steel and what could be the precipitates formed for the practised temperature and the treatment duration in the present study as these fine precipitates are not resolved by the light optical microscope.

Grain size vs. Heat treatment

Grain size of the heat-treated samples was estimated by intercept method in order to analyse the effect of solutionizing temperature on PAGS of T92 steel and the estimated average grain diameter values are given in Table 3. It can be seen from the values, tempering has no effect on the grain size, as expected. In general, fine grain microstructure offers higher yield stress than coarse grain microstructure. But coarse grain structure is preferred

Table 3: Prior austenite grain size for each heat-treated condition.

Sample designation	Solutionizing temperature ($^{\circ}\text{C}$)	Prior austenite grain size (μm)	Average grain size (μm)
$S_A T_X$	1,050	27.26 ± 2	27.61
$S_A T_Y$		27.75 ± 2	
$S_A T_Z$		27.84 ± 2	
$S_B T_X$	1,100	55.33 ± 2	54.95
$S_B T_Y$		53.21 ± 2	
$S_B T_Z$		56.30 ± 2	
$S_C T_X$	1,150	110.23 ± 3	110.31
$S_C T_Y$		110.06 ± 3	
$S_C T_Z$		110.65 ± 3	

for high-temperature applications, due to their creep resistance. So, optimum grain size should be achieved to have a combination of good tensile properties and improved creep strength, as well.

Grain size of the heat-treated steel samples is presented in a graphical form in Figure 3. An empirical relation (eq.1) relating the grain size to the solutionizing temperature has been developed for the curve shown in Figure 3.

$$Y = 0.0056X^2 - 11.5018X + 5,926.09 \quad (1)$$

Figure 3: Effect of solutionizing temperature on grain size of T92 steel test samples.

where Y is grain size in μm and X is the solutionizing temperature in $^{\circ}\text{C}$.

Using this empirical equation, the grain size of the heated treated samples can be obtained, for solutionizing temperatures from $1,050\text{ }^{\circ}\text{C}$ to $1,150\text{ }^{\circ}\text{C}$.

The slope of the curve (Figure 3) is steeper at higher temperatures suggesting that grain growth kinetics is faster at higher temperatures. At high temperatures, available activation energy is more and amount of left over undissolved precipitates is also less. Both of these conditions aid in accelerating the kinetics of grain growth. While at low temperatures, presence of undissolved precipitates hinders the grain coarsening.

Microhardness

Hardness of the T92 steel is influenced by the alloying elements (nature and quantity) and by the precipitates (nature, size, location and morphology). Measured microhardness values of the heat-treated T92 steel samples are presented as a bar chart in Figure 4. Hardness value of the T92 steel in the as-rolled condition is also presented for comparison. The finishing temperature of the rolling process should have been well below the solutionizing temperature resulting in fine grain structure with large amount of dislocation along with all the precipitates formed which would have resulted in very high hardness value of the steel in the as-rolled condition (547HV). This value is nearly double of the heat-treated samples. Subsequent solutionizing has resulted in grain growth, reduction in dislocation density and dissolution of the precipitates and brought down the hardness values. Similarly, tempering influences the type and extent of carbide formation and decides final hardness of the heat-treated samples.

Figure 4: Hardness of T92 steel under different heat-treated conditions (heat treatment details are mentioned in Table 2).

On close observation, it can be seen from the presented data that for any tempering condition, hardness value increases with increase in solutionizing temperature and similarly for any solutionizing temperature hardness decreases with increase in tempering temperature.

The degree of dissolution of the precipitates present in the as-rolled condition is a function of solutionizing temperature. More the dissolution of the precipitate, more will be the dissolved solute in the matrix. Solid solution strengthening by both substitutional and interstitial solutes occurs. At 1,050 °C, though M₂₃C₆ precipitates are dissolved, remnant NbC precipitates will be present. However, the size of the undissolved precipitates can be larger in size than precipitates formed due to tempering operation. The undissolved NbC precipitates act as a nucleation site for forming of plate-like nitrides during tempering operation and also it leads to forming the complex V-wing precipitates [22].

With increase in solutionizing temperature, more amount of solute is dissolved in the matrix and the size of the remnant precipitate is also diminished. Thus, dissolution of the M₂₃C₆ and MX precipitates and dissolution of solutes in the matrix leading to the solution strengthening cause the increase in hardness of the solutionized sample with increase in solutionizing temperature.

Tempering involves formation of precipitates along the lath and grain boundaries, and within the grains. But the extent of precipitation is controlled by the temperature and duration of tempering treatment and also their position in the time–temperature–precipitation (TTP) curve for this alloy. (TTP diagram for this alloy is not available from the published literature). If the chosen temperatures are above the nose of the C-curve of TTP diagram, more precipitate will form for a lower tempering temperature than for a higher tempering temperature, whereas for temperatures below the nose of the C-curve, precipitation will be slowed down with drop in temperature. Further during tempering, formation of precipitates is by drawing the solute atoms from the matrix causing reduction in solid solution strengthening. At the same time, precipitates formed can hinder the movement of dislocations and result in increase in strength. The dominant factor between the two mutually competing processes also will play a role in deciding the final hardness of the tempered samples.

In the present case, for any given solutionizing condition, the hardness is inversely proportional to the tempering temperature. It indicates that the tempering temperatures should be below the nose of the C-curve and reduction in hardness due to depletion of solute from the matrix plays a dominant role than the increase in hardness due to the formation of precipitates.

Tensile test

Tensile properties of the heat-treated samples were evaluated using microtensile samples. The values reported in Table 4 are the average of at least three samples. The data indicate that with tempering, the tensile properties of the heat-treated samples are reduced. The changes in microstructure of the T92 steel have a direct effect on mechanical properties. Large grain size and the low carbon content of the steel facilitate the formation of lath martensite at rapid cooling from solutionizing temperatures. The martensite lath width and packet size are reported to have direct relation with the PAGS. Further, it is known that quenching from a higher temperature can introduce more dislocation density in an alloy than if quenched from a lower temperature. Thus, an alloy solutionized at the highest temperature and quenched in water is expected to have coarse grain, wide laths, large packets and increased dislocation density. Tempering promotes reduction in dislocation density by recovery and subgrain formation. $M_{23}C_6$ and MX carbides and/or carbonitrides are precipitated during tempering process. The extent of precipitation is directly proportional to the tempering temperature and duration. Elements dissolved as substitutional and interstitial solutes are pulled out of solutions causing reduction in solid solution strengthening. The microstructural changes that have opposite influence on the mechanical properties occur simultaneously during tempering process. The net effect decides the final mechanical property of the heat-treated T92 steel. The accepted minimum level of tensile properties for T92 material is 620 MPa for tensile

strength and 440 MPa for yield strength. All the heat-treated samples are meeting the tensile strength requirements, whereas yield strength of the samples tempered at 745 °C and above is lower than the prescribed level of 440 MPa. However, solutionizing at 1,050 °C and tempering up to 760 °C could result in acceptable level of tensile properties. Low-temperature solutionizing leads to lesser extent of dissolution of precipitates and therefore, the matrix having more amounts of undissolved precipitates is lean with respect to the precipitate forming elements. This affects the kinetics of precipitation during tempering which results in higher yield strength.

Fractography analysis

Fractographs of tensile-tested samples are shown in Figures 5–7. Fine dimples with shear lips and also elongated dimples are seen for the sample solution treated at 1,050 °C, irrespective of the tempering conditions (Figure 5). With increase in solutionizing temperature from 1,050 °C to 1,100 °C and 1,150 °C, it can be seen from Figures 6 and 7 that grain boundary decohesion has led to the fracture though the grain interior which exhibits fine dimple structure. Presence of fine precipitates in the dimples could also be noticed. Grain boundary decohesion has led to the intergranular fracture and the effect is clearly revealed in Figures 6 and 7. Generally, cracks encounter more grain boundary barriers and get deflected in alloys having smaller grains [23]. Cleavage cracks are predominately observed (Figures 6 and 7) in alloys having coarse grain structure and it offers less barrier for crack deflection.

Table 4: Tensile results of heat-treated test samples of T92 steel.

Solutionizing temperature (°C)	Tempering temperature (°C)	Yield strength (MPa)	Ultimate tensile strength (MPa)	Elongation (%)
1,050	730	562	950	27.50
1,050	745	482	877	29.10
1,050	760	448	679	23.90
1,100	730	517	879	27.80
1,100	745	418	856	28.50
1,100	760	394	754	26.80
1,150	730	503	872	21.60
1,150	745	385	795	25.30
1,150	760	338	662	23.80

Conclusion

The studies made on the effect of heat treatment, i. e. solutionizing and tempering on microstructural and mechanical properties of T92 steel, are leading to the following conclusions:

1. PAGS increases from 27.61 μm to 110.31 μm with increase in solutionizing temperature from 1,050 °C to 1,150 °C.
2. Owing to the extent of dissolution of preexisting precipitates increases with increase in solutionizing

Figure 5: Fracture features of tensile-tested T92 steel samples solutionized at 1,050 °C and tempered at (a) 730 °C, (b) 745 °C and (c) 760 °C.

Figure 6: Fracture features of tensile-tested T92 steel samples solutionized at 1,100 °C and tempered at (a) 730 °C, (b) 745 °C and (c) 760 °C.

temperatures, higher hardness values are witnessed at higher solutionizing temperatures.

3. Reduction in hardness during tempering is the result of mutually competing processes such as reduction in hardness due to recovery process leading to increase in lath width and corresponding reduction in dislocation density, and increase in hardness due to the formation of various new precipitates.
4. Solutionizing temperature has an inverse relationship with ultimate tensile strength. However, even solutionizing at 1,150 °C can yield an acceptable ultimate tensile strength.
5. Similarly, yield strength decreases with increase in tempering temperature. The present findings suggest that tempering should be restricted to a maximum of 730 °C, especially if the solutionizing temperatures

Figure 7: Fracture features of tensile-tested T92 steel samples solutionized at 1,150°C and tempered at (a) 730 °C, (b) 745 °C and (c) 760 °C.

are above 1,050 °C, to attain an acceptable level of yield strength (440 MPa).

6. Solutionizing at higher temperature reduces the grain boundary barrier for cleavage cracks leading to grain boundary decohesion.

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