Microstructure characterisation and microhardness of P92 steel heat treated at the transformation temperatures

J Obiko 1,a, L H Chown 1,2,b, D J Whitefield 1,2,c

1 School of Chemical and Metallurgical Engineering, University of the Witwatersrand, 1 Jan Smuts Avenue, Johannesburg, 2000, South Africa
2 DST-NRF Centre of Excellence in Strong Materials, hosted by the University of the Witwatersrand

a Japheth.Obiko@students.wits.ac.za, b Lesley.Chown@wits.ac.za, c David.Whitefield@wits.ac.za

Abstract. Modern power plant components for the transport of superheated steam are manufactured from creep resistant steels, such as ASTM grade P92, that have microstructural stability and excellent mechanical properties at high temperature and pressure. Before application as pipes, tubes and connectors, P92 steel undergoes a heat treatment of austenitisation and tempering to achieve a tempered martensite microstructure, with carbides along the prior austenite grain boundaries and carbonitrides within the matrix. In the power plant, pipes and tubes are joined by welding, inducing residual stresses and inhomogeneous microstructures in the weld joint, which reduce creep resistance. As in most steels, the temperature gradient from the fusion line to the base metal leads to different microstructural zones in the weld joint. The fine-grained heat affected zone (FGHAZ), which forms at temperatures near the transformation region, acts as a weak zone for Type IV cracking during creep. The lower and upper critical transformation temperatures for the P92 steel in this work are 800°C-850°C (Ae1) and 900°C-950°C (Ae3) respectively. As-received samples were heated to temperatures of 830, 850, 900°C to simulate the fine grain heat affected zone, or the very high temperature of 1200°C to simulate the coarse grain heat affected zone (CGHAZ), held at that temperature for 90 minutes and then tempered at 730°C to 780°C for 120 minutes. The microstructures before and after heat treatment were observed by scanning electron microscopy and optical microscopy, and Vickers microhardness measurements were done. After tempering, coarse carbides were seen on the prior austenite grain boundaries (PAGBs) and nitrides were observed in the matrix. In general, the hardness increased with an increase in the austenitisation and tempering temperature combination.

1. Introduction
The use of fossil fuel for electricity generation has received global attention due to the shortage of coal, oil and gas and also due to environmental regulations to reduce carbon dioxide (CO₂) emissions [1]. The thermal efficiency of the power plants has been improved by increasing the steam temperatures and pressures [2,3]. Ultra-supercritical power plants are projected to operate at high temperatures and pressures of ~650°C and 30 MPa [4,5]. Creep resistant 9-12 wt% chromium steels, such as P91, E911 and P92, have been developed to operate at high temperature and pressure [2]. P92 steel was developed from modified P91 steel and exhibits higher creep strength [5]. These steels are mainly used for structural components such as steam boiler tubes and pipes [4]. The high-temperature phase stability of these steels has been achieved by the addition of various micro-alloying elements, such as Mo, W, V,
Nb, Co and Mn [3]. These additions enhance the solid solution and precipitation strengthening, hence improving creep resistance [6,7]. P92 steel also has a lower coefficient of thermal expansion, higher resistance to corrosion, good weldability and higher thermal conductivity than older generation 9%Cr steels [8,9].

After production, P92 steels undergo heat treatments of normalisation and tempering before being supplied for industrial application [5]. The normalisation process is usually done in the temperature range of 1040°C to 1100°C for the duration of 20-30 minutes, followed by tempering at 760°C to 780°C for 30-40 minutes [9,10]. After tempering, the steel exhibits a tempered martensitic microstructure with carbides along the prior austenite grain boundaries (PAGBs) and carbonitrides finely distributed in the matrix [11,12]. The presence of precipitates along the PAGBs and martensite lath boundaries prevents subgrain growth and so improves the strength of the steel [12]. The commonly identified precipitates include M23C6 (containing Fe, Cr, Mo, W and C) carbides and MX (containing V, Nb, C and N) carbonitrides [6]. Nagode et al. [13] showed that MX precipitates finely distributed in the matrix act as a barrier to dislocation movement during creep deformation. Di-Gianfrancesco et al. [14] reported that both M23C6 and MX precipitates enhance creep resistance, as the precipitates hinder dislocation movement during creep deformation [9].

Hald [15] found that the microstructural stability of 9 wt% Cr steels largely depends on precipitate stability for long term exposure to high temperature and pressure. An optimised normalisation and tempering process of P92 steels leads to phase transformation from austenite to a refined tempered martensite structure [11]. Normalisation is usually done above the upper critical transformation temperature (Ae₃), which is ~900-950°C for P92 steel, depending on the specific composition [16]. The effect of heat treating P92 between the lower and upper critical temperatures followed by tempering has not been widely reported.

This paper presents the microstructure evolution of two P92 steels with differing amounts of chromium and tungsten, which were subjected to different combinations of normalisation and tempering temperatures. The ‘normalisation’ temperatures were selected to be similar to those attained during welding: 850-950°C in the fine grained heat affected zone (FGHAZ) and ~1200°C in the coarse grained heat affected zone (CGHAZ). After the different heat treatment cycles, the precipitate distribution was compared and the hardness variation was also reported.

2. Materials and Methods

The two P92 steels in this study are named A and B, and their chemical compositions are given in Table 1. P92 steels are usually supplied in the as-normalised and tempered condition. Samples were cut from the as-received P92 steels and were heat treated (N) for 90 minutes and tempered (T) for 120 minutes. Four sets of N+T temperatures were used: at 830°C (N)/730°C (T), 850°C (N)/750°C (T), 900°C (N)/760°C (T), and 1200°C (N)/780°C (T), to produce various microstructures. The FGHAZ reaches peak temperatures of ~900°C.

Specimens for microhardness and microstructure analysis were prepared using standard metallographic procedures. After grinding and polishing, the specimens were etched with Villella’s reagent (5 ml HCl + 1 g picric acid + 100 ml ethanol) for microstructure investigation using a Leica DM 6000M optical microscope and a Zeiss Sigma field emission scanning electron (FEGSEM) microscope. Microhardness of the as-received and heat treated samples were measured using a 1 kgf load and a dwell time of 10 seconds with a FUTURE-TECH FM-700 microhardness tester.

Thermo-Calc thermodynamic simulation software with the TCFe5 database [17] was used to determine the equilibrium Ae₃ and Ae₁ transformation temperatures.

**Table 1. Chemical composition of the as-received steels (wt %).**

| Steel | C  | Mn  | S  | P  | Si  | Cr  | Mo  | Ni  | Cu  | Al  | V  | Nb  | W  | Co  |
|-------|----|-----|----|----|-----|-----|-----|-----|-----|-----|----|-----|----|-----|
| A     | 0.10 | 0.39 | 0.011 | 0.014 | 0.20 | 8.29 | 0.65 | 0.19 | 0.08 | 0.012 | 0.16 | 0.093 | 2.07 | 0.015 |
| B     | 0.11 | 0.51 | 0.005 | 0.010 | 0.22 | 9.37 | 0.50 | 0.17 | 0.27 | 0.006 | 0.19 | 0.130 | 1.76 | 0.028 |
3. Results and Discussion

3.1. ThermoCalc analysis

The thermodynamic calculations were done to determine the phase transformation temperatures ($A_{\varepsilon 1}$ and $A_{\varepsilon 3}$) and dissolution temperatures of precipitates using ThermoCalc with the TCFe5 database. For the two P92 steels in this work, the equilibrium phase transformation and dissolution temperatures of $M_23C_6$ and MX precipitates are given in Table 2.

Table 2: ThermoCalc predicted transformation temperatures and precipitate dissolution temperatures (in °C)

| Steel | $A_{\varepsilon 1}$ | $A_{\varepsilon 3}$ | $M_23C_6$ | MX   |
|-------|--------------------|--------------------|-----------|------|
| A     | 830                | 945                | 870       | 1201 |
| B     | 816                | 917                | 888       | 1250 |

3.2. Microstructural analysis

3.2.1. As-received base material.

Micrographs of the two P92 steels in the as-received (heat treated + tempered) condition are shown in Figure 1 a-d. Both steels had a tempered martensite microstructure with well-defined prior austenite grain boundaries. It has been reported that the creep strength of P92 steel is enhanced by the presence of precipitates such as $M_23C_6$ ($M = Fe, Mo, W, Cr$) carbides and MX ($M = V, Nb; X = C, N$) carbonitrides [18]. $M_23C_6$ carbides nucleate along the PAGBs and lath boundaries, while MX precipitates are usually randomly distributed in the matrix [8]. The SEM micrographs in Figure 1c-d show precipitates on the lath boundaries which are assumed to be $M_23C_6$ carbides.
Figure 1. Optical and SEM micrographs of as-received Grade P92 steel: a) and c) steel A, b) and d) steel B.

3.2.2. Optical microstructure after heat treatment.

The optical images for steels A and B after various heat treatment procedures are shown in Figure 2. Figure 2 a and b show optical micrographs of alloy A and B after heat treatment at 830°C and tempering at 730°C. It was observed that this heat treatment had changed the initial tempered martensite lath structure of the as-received samples into finer martensitic lath structures. Similar behaviour was also observed after heat treatments of 850°C/750°C and 900°C/760°C (Figure 2 c - f). The tendency of 9-12% Cr to form fresh lath martensitic structures and fine grain sizes even at low cooling rates has been reported in literature [4]. During normalisation, there is a complete dissolution of precipitates into the matrix resulting in a homogeneous austenitic microstructure [19]. However, in this work, two of the initial heat treatment temperatures were below the precipitate dissolution temperatures of $\text{M}_2\text{C}_6$ carbides (870°C (steel A) and 888°C (steel B)) which would have led to only partial dissolution. The tempering stage reduces the dislocation density, breaking of laths and reduction of solid solution strengthening due to the precipitation and coarsening of the carbides [20]. Figure 2 g-h show the microstructures of steel A and steel B after normalisation at 1200°C (far above the $A_{et}$ temperature and the $\text{M}_2\text{C}_6$) and tempering at 780°C. After this treatment, both steels A and B showed tempered lath martensite microstructures with coarse grain sizes.

Figure 2 e) shows the formation of ferrite containing coarse carbides within the matrix of tempered martensite. The ferrite forms either due to the incomplete transformation of ferrite into austenite on heating and finally martensite on cooling or the contribution of alloying elements. [21] Ryu and Yu [22] reported that ferrite retention is a function of alloying elements that affect the expansion and contracting of the austenite field. Wang et al. [23] investigated the role of cobalt in 9-12 wt% Cr steels, and observed that δ-ferrite was suppressed by adding 1% Co and reducing the nickel content to 0.4% depending on the presence of the ferrite and austenite stabilisers. Similarly, Sireesha et al. [24] reported that δ-ferrite retention is likely to occur in steels containing ferrite stabilisers. They proposed using an empirical formula for the Cr equivalent and the Kaltenhauser ferrite factor (FF) to determine the tendency of ferrite retention in the matrix (in wt %):

\[
\text{Cr}_{eq} = \text{Cr} + 6\text{Si} + 4\text{Mo} + 1.5\text{W} + 11\text{V} + 5\text{Nb} + 12\text{Al} + 8\text{Ti} - 40\text{C} - 2\text{Mn} - 4\text{Ni} - 2\text{Co} - 30\text{N} - \text{Cu}
\]

\[
\text{FF} = \text{Cr} + 6\text{Si} + 4\text{Mo} + 8\text{Ti} + 2\text{Al} + 4\text{Nb} - 2\text{Mn} - 4\text{Ni} - 40\text{C} + \text{N}
\]

The $\text{Cr}_{eq}$ is used to determine the ferrite formation in 9-12 wt% Cr since it gives the amount of chromium percentage in Fe-Cr phase at which equal amount of δ-ferrite will form at the same temperature [22]. The retention of ferrite has been reported to occur preferentially along the grain boundary and at triple
points [22] when Cr<sub>eq</sub> is greater than 10 and FF is greater than 8. For the current study, steel A had a Cr<sub>eq</sub> of 11.1 and an FF value of 7.0, while steel B had 11.4 and 7.2. The prediction of Equations 1-2 is correct as ferrite phase was observed in the microstructure as shown in Figure 2(e) even though FF was slightly below the reported empirical value. Even though steel B is more prone to form ferrite, the presence of ferrite phase in steel A may be attributed to the cooling rate. The formation of ferrite along the grain boundary seen in Figure 2e may be attributed to the amount of ferrite and austenite stabilisers in the two steels. However, this requires further analysis.

3.2.3. SEM microstructure after heat treatment.

The SEM secondary electron images showing the microstructure of heat treated and tempered samples of steel A and B are shown in Figure 3 and Figure 4 respectively. The steels exhibited a martensitic structure with distinct prior austenite grain boundaries (PAGBs) delineated by the coarse M<sub>23</sub>C<sub>6</sub> precipitates with some also on subgrain boundaries. Figure 3 a) and b) show precipitate distribution along the former PAGBs of the steel before heat treatment, while Figure 3 d) shows the newly formed PAGBs decorated with precipitates after the heat treatment.

From Figure 4, steel B exhibited a tempered martensitic microstructure with fine prior austenite grain sizes. The evolution of precipitates clearly delineates the newly formed grain boundaries. Maddi et al. [11] reported that M<sub>23</sub>C<sub>6</sub> carbides precipitate along the austenite and lath boundaries. The M<sub>23</sub>C<sub>6</sub> carbides impede dislocation movement during creep deformation, hence improving creep strength [25].
Figure 2. Optical micrographs after heat treatment at different heat-treated (N) + tempering (T) temperatures.

Figure 3. Secondary electron micrographs of steel A after heat treatment.
3.2.4. Quantitative image analysis.

Quantitative analysis of the larger, visible precipitates evolved during heat treatments for the two steels was done using the Image J image analysis program. The results are shown in Figure 5-7. Figure 5 shows the variation in the average particle size, expressed as particle area (μm²), with increase in the normalising temperature (and thus tempering temperature). The particle area continuously increased for steel B as the normalising-tempering temperatures increased. While for steel A the particle area increase at slow rate and then dropped as the normalisation temperature increase to 1200°C. The percentage area fraction of particles for steel A increased between 830°C to 900°C normalisation temperatures then drastically decreased as the normalisation-tempering temperature increased (1200°C/780°C). For steel B, the percentage area fraction of particles increased rapidly from 830°C to 900°C as shown in Figure 6. Further increases in the normalisation temperature gave only an insignificant change in percent area fraction. It was observed that the population density of precipitates decreased as the normalisation temperature increased for steel B, which can be attributed to the formation of large grain sizes as the heat treatments temperature increases hence, reducing the number of grain boundaries for the precipitation of carbides [8]. While for steel A, it was noticed that the population density of particles decreased from 830°C to 900°C and then started to increase with further increase in temperature to 1200°C. This increase in the particle population density can be attributed to the formation of smaller grain sizes which leads to more grain and lath boundaries per unit area available for the precipitation of carbides.

![Precipitates](image1)

![Precipitates](image2)

![Precipitates](image3)

![Precipitates](image4)

**Figure 4.** SEM micrographs of steel B after heat treatment.
3.3. Microhardness Analysis

The microhardness values of as-received steels A and B were 239 ±7 HV\textsubscript{1} and 230 ±7 HV\textsubscript{1} respectively, which is within the design specifications range of 210-265 HV\textsubscript{0.3} [16]. The variation in microhardness for the as-received and heat treated steels is shown in Figure 8. It was observed that for steel A the microhardness increased as the heat treatment temperatures increased. However, steel B showed unique behaviour after the heat treatment of 830°C/730°C, with a high hardness of 241 HV\textsubscript{1}, similar to that of the as-received material. After this heat treatment (830°C/730°C), full dissolution of carbides would not have taken place. The hardness of Steel B then increased with increasing heat treatment temperature from 850-1200°C. The subsequent tempering process would have led to coarsening of the precipitates, which reduces the precipitate nucleation sites and lowers the area fraction of precipitates, thus leading to solid solution hardening [26]. At 830°C, steel B had low area fraction of precipitates (Figure 5), indicating high solid solution hardening due to incomplete precipitation of carbides hence, and high hardness. At 850°C, steel B had the lowest hardness value of 216 HV\textsubscript{1} compared to 227 HV\textsubscript{1} at 900°C and 245 HV\textsubscript{1} at 1200°C as shown in Figure 8.
The variation in hardness values was attributed to the microstructural changes that occur during the heat treatment processes. It has been reported that grain size, solid solution strengthening, precipitation of carbides and carbonitrides and thus the reduction of N and C from the solid solution during tempering account for the hardness variations in 9-12% Cr steels [5,19]. Pandey et al. [27] reported that an increase in normalisation temperature transforms the microstructure into single phase austenite which on cooling transforms to martensite. The formed martensite structure increases the microhardness due to an increase in dislocation density. In the current study, there was an increase of 11.5 ± 7 % in the microhardness with an increase in the heat treatment temperatures.

Lower hardness values were observed at heat treatment temperatures of 850°C/750°C and 900°C/760°C for steel A and B than the as-received steel samples, as the tempering process softens the formed brittle martensite structure after austenitisation. Similar low hardness values have been observed to occur in the Fine Grained Heat Affected Zone (FGHAZ) of the weld joint subjected to peak temperature near the \(\text{Ac}_3\) transformation temperature (which is slightly higher than the \(\text{Ae}_3\)) for 9-12 wt% Cr steels [25], [28]. Wang et al. [19] reported that hardness lowering characteristics can be attributed to the recovery of the prior austenite grain boundaries leading to the formation of coarse sub-grains and the reduction in dislocation density. According to Abe and Tabuchi [29] lower hardness was observed when the base metal was heat treated at peak temperatures close to \(\text{Ac}_1\) (~850°C for P92 steel). High hardness was observed at 1200°C for both steels which exhibited microstructures similar to that of the Coarse Grained Heat Affected Zone (CGHAZ) after welding. This is why steel A and B were subjected to normalisation temperatures for P92 which are experienced within the weld joint.

The hardness of steel B after heat treatment at 830/730°C was similar to the as-received steel as there would have been negligible change to the strengthening precipitates on heating up to the \(\text{Ae}_1\). The low hardness values for both steels after the 850/750°C treatment was likely due to coarsening and dissolution of \(\text{M}_2\text{C}_6\) precipitates, which would have been completely dissolved by 870°C (steel A) and 888°C (steel B). With further increase in the normalising temperature to 900°C, the \(\text{M}_2\text{C}_6\) would be fully dissolved for both steels and could precipitate, increasing the hardness. The MX precipitates of steel A would have been fully dissolved at 1201°C (A), so would precipitate on cooling, further increasing the hardness. The trend in hardness with heat treatment temperature from 850 - 1200°C was similar for the two steels, with steel A having the lower values. Although the predicted \(\text{Ae}_1\) of steel A (830°C) coincided with the heat treatment temperature of 830°C, the high hardness seen in steel B was not observed.

![Figure 8. Hardness variation at different heat treatment conditions.](image)
4. Conclusions

- From the ThermoCalc analysis of the two P92 steels, results showed that the lower and upper critical phase transformation temperature (Ae1) range from 800-830°C and 900-945°C. $\text{M}_2\text{C}_6$ carbide dissolution temperature was determined to be 870°C (steel A) and 888°C (steel B) while MX precipitates dissolved at 1201°C (steel A) and 1250°C (steel B).
- After normalising and tempering, both steels contained martensite lath structures. From the SEM images, coarse $\text{M}_2\text{C}_6$ precipitates were seen to occur along the prior austenite grain boundaries.
- Quantitative analysis showed that the heat treatment temperatures had an influence on the precipitate formation and distribution. As the temperatures increased, the population density of the precipitates decreased. Steel B showed a more rapid increase in the area fraction and particle size with an increase in the heat treatment temperature compared with steel A. However, area fraction and particle size of steel A decreases at 1200°C.
- The microhardness values for the two steels increased with an increase in the heat treatment temperatures. Steel B had higher hardness values than steel A in all test conditions which was attributed to the solid solution hardening effect.

5. Acknowledgements

The authors would like to acknowledge and appreciate with gratitude the DST-CSIR Interbursary Support (IBS) programme for financial support.

6. References

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