Materials Research Express

RECEIVED
6 August 2019

REVISED
18 September 2019

ACCEPTED FOR PUBLICATION
24 September 2019

PUBLISHED
4 October 2019

PAPER

Mechanical and microstructural evaluation of dual phase steel, quenched in bitumen and water at an intercritical temperature: effect of holding time

E Olorundaisi , T Jamiru and T A Adegbola

Department of Mechanical Engineering, Mechatronics, and Industrial Design, Tshwane University of Technology Pretoria, South Africa

E-mail: olorundaisiemmanuel@gmail.com

Keywords: microstructure, dual phase steel, mechanical properties, low carbon steel

Abstract

Low carbon steel was heat-treated at an annealing temperature of 800 °C for a different holding time of 30, 40, 50 and 60 min, to investigate the microstructure evolution and the mechanical properties of Dual Phase (DP) steel. The samples were subjected to different quenching medium (bitumen and water) and the result obtained was analyzed. Optical microscope characterization of the DP steel shows the extensive distribution of martensite phase within the ferrite matrix. The DP steel exhibit improved ultimate tensile strength, yield strength, and hardness when compared with the as-received samples. At the holding time of 50 and 60 min, DP steel quenched in water shows better strength and hardness as compared to bitumen quenched samples. The hardness value of water to bitumen quenched at 50 and 60 min is 169.32 HV as against 167.72 HV and 172.46 HV as against 169.67 HV respectively. Likewise, for ultimate tensile strength, the values showed 600.09 MPa as against 594.42 MPa and 611.22 MPa as against 601.33 MPa respectively, and that of yield strength as 304.14 MPa as against 301.27 MPa and 309.78 MPa as against 304.77 MPa respectively. Despite the slight difference in strength and hardness, both quenching medium (water and bitumen) can be exploited for the production of Dual Phases steel.

1. Introduction

The microstructure of Dual-phase (DP) steels is made up of martensite (hard) grain dispersion with few volumes of bainite capsulated in a ductile ferrite (soft) matrix [1]. It is cost-effective and possesses remarkable mechanical properties, such as high tensile strength, enhanced formability, continuous yielding behavior, crashworthiness, high strength-ductility, and high work hardening rates [2–4]. The quest for improved lightweight, safety and considerable energy savings material, gave birth to the production of DP steels [5]. When compared with other conventional steels, DP steel is rated as a reliable material for the automobile and other structural applications. These steels (DP) is of low carbon steels, it's made up of duplex ferritic martensitic microstructure with a chemical composition that is uncomplicated compared to some other conventional steels, which makes it even more advantageous for welding purposes [6, 7].

The properties of DP steel is a function of the ferritic matrix grain size and the martensite particles morphology [8]. Its microstructure is polygonal in shape [9]. However, its mechanical properties are a product of the microstructure made up of different phases (ferrite, martensite, bainite or pearlite), the combination of these phases with varying ductility and strength, formed a new material. Therefore, Temperature, holding time, cooling rates, ferrite grain size and the amount, morphology, composition and martensite distributions, are important parameters that influence the mechanical properties of Dual Phase steels.

Steel with the finer grain rather than the coarse, have shown to possess qualitative mechanical properties [6, 10]. DP steel has tensile strength ranging from 500–1200 MPa and total elongation in the range of 12%–34%. However, a high volume of martensite produces high yield and ultimate tensile strength, alongside a uniform decrease in total elongation [9]. The distribution pattern of martensite also influences the mechanical behavior

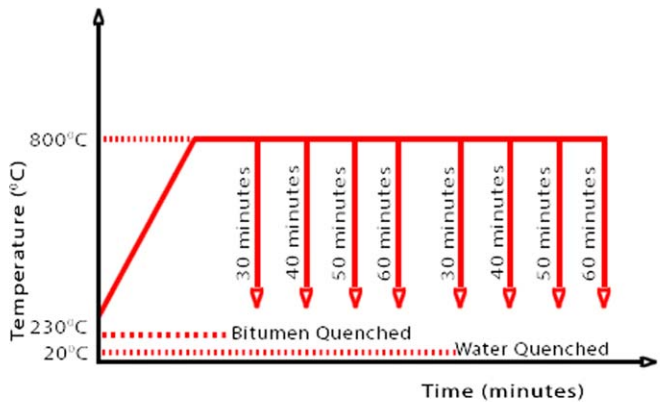

Figure 1. Intercritical annealing process.

Table 1. Chemical composition of the low carbon steel.

Elements	C	Mn	Si	Al	Co	Cr	Cu	Ni	S	P
Composition (wt%)	0.183	0.64	0.033	0.036	0.002	0.009	0.014	0.008	0.02	0.016

of DP steel. An isolated martensite region within the ferrite matrix tends to have improved strength and ductility than a distribution of chain-like structure surrounded by ferrite matrix [9].

DP microstructure can be produced by heat treatment. Therefore, Intercritical annealing is a conventional heat treatment to obtained ferrites-martensite microstructure which involves heating at an Intercritical annealing temperature for a particular holding time and quenched rapidly, for austenite to fully transformed to martensite [1, 2]. It invariably means that the transformation of austenite to martensite is subjected to the influential factors of temperature and holding time [11]. At Intercritical annealing, the isothermal transformation of austenite to ferrite occurs, thereby forming an austenite-ferrite mixture. However, when this mixture is quenched rapidly, a microstructure of ferrite and martensite is formed [12].

The volume fraction of martensite can be controlled by adjusting the annealing temperature and time. At higher temperature and holding time, a larger volume fraction of austenite is formed, hence a large volume fraction of martensite is produced after quenching [13]. The ferrites region in DP steel is responsible for the ductility while the martensite region provides the strength. The transformation of austenite either result in, the formation of bainite or martensite depending on the cooling rate. At partial austenitization, the carbon content in the austenite grain varies directly with Intercritical temperature. Hence at a high temperature, more volume fraction of austenite alongside a weaker carbon content is formed. Therefore the carbon and other alloying elements present in DP steel affect its hardness and hardenability [14–16]. Quenching media is very important to hardening, liable on the alloy and other facts such as concern for maximum hardness versus cracking and distortion. The selection of quenchant is influenced by the hardening capability of the alloy, morphology of the material and the rate of cooling required for the desired material [17]. Therefore, quenchant should be able to excerpt heat from at least the exterior part or the whole parts of the quenched material. Quenching medium ranges from water, oil, salt solution, and liquid metal bath. Preference has been given to water and salt solution for steel with low hardenability while oil is preferred for micro-alloyed steel, as it eliminates the tendency for cracks growth in the steel [18–22]. Bitumen being an oil is, however, chosen for this work because of its large deposit in Africa (Nigeria). The present research was carried out to investigate the changes that occur in the microstructure and mechanical behavior of DP steel, has been triggered by different holding time. Using bitumen at a temperature of 230 °C and water at room temperature (20 °C) as a quenching medium, the effect of holding time was examined at an Intercritical annealing temperature of 800 °C (figure 1).

2. Experimental detail

2.1. Material and heat treatment

Table 1 shows the chemical composition of the as-received steel used in the present work. The sample is a sheet and was wire cut to 10 samples at a dimension of 10 × 2 mm before heat-treated (figure 2). Four of the samples was quenched in bitumen (SS 60–70 grade) at a temperature of 230 °C, another four was quenched in water at

Figure 2. Wire cut 10 × 2 mm samples.

Table 2. Mechanical properties of normalized low carbon steel.

Sample designation	Hardness (HV)	Ultimate tensile strength (MPa)	Yield strength (MPa)
Normalized sample	89.45	317.02	160.68

room temperature (20 °C). The remaining (two) samples were used as control and therefore not heat treated. As required the prepared samples were subjected to normalization, then austenitized by heating to Intercritical annealing temperature of 800 °C for 30, 40, 50, and 60 min, quenched rapidly in bitumen for 10 min followed by air cooling to room temperature for the complete removal of any traces of retained austenite and residual stresses. The procedure was repeated for another four samples quenched in water.

The samples used for carrying out the microstructural examination and hardness test were hot mounted using a Struers CitoPress-1 machine. After which the face was thoroughly grinded and polished, using Struers TegraPol-11 550 Machine, with disc grade of 90, 220 and 330 in successions. After the final polishing, the samples were washed in running water and dried. The samples were then etched using 3% nital solution (10 ml Nitric acid of 70% concentration and 100 ml Ethanol). The surface thus prepared is then kept on the Olympus Metallurgical microscope to capture the microstructure of the samples. Hardness was done using Vickers Microhardness Tester (Automatic indentation measuring system ARS900), the samples were indented with a load of 500 gf until a permanent indentation was achieved. In each of the tests performed the average of three consistent readings were taken and presented. Ultimate tensile strength (UTS) and yield strength (YS) was computed using expressions developed and used by [23–27], relating hardness and tensile strength and yield strength in the form of:

$$\mathrm{UTS} = \left(\frac{H}{2.9}\right)\left(\frac{n}{0.217}\right)^{n} \tag{1}$$

$$\mathrm{YS} = \left(\frac{H}{3}\right)(0.1)^{n} \tag{2}$$

Where:

UTS is given as Ultimate Tensile Strength

YS is given as Yield Strength

H is given as Hardness

n is given as the Strain-Hardening Exponent (for low carbon steel it's given as 0.26) [28].

Converting hardness value from Vickers Pyramid Number (HV) to Megapascal (MPa) is provided as:

$$\mathrm{MPa} = (HV)(9.806) \tag{3}$$

3. Results and discussion

3.1. Mechanical analysis

The mechanical properties of the normalized sample presented in table 2 show a bulk hardness of 89.45 HV, the tensile strength of 317.02 Mpa and yield strength of 160.68 Mpa. The result of the samples quenched in bitumen and water is presented in table 3.

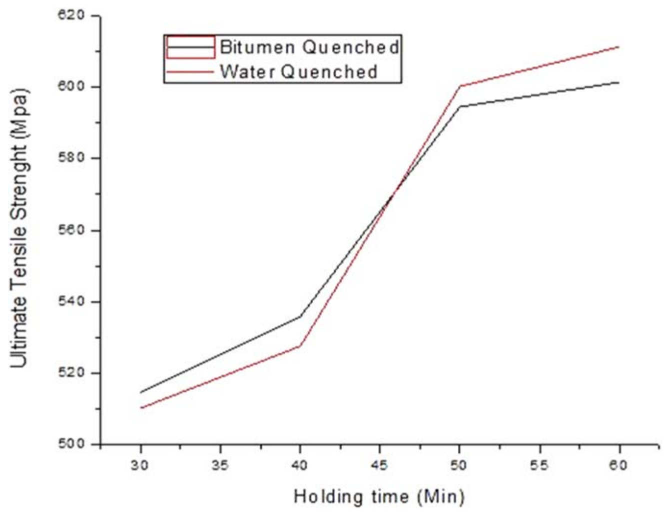

Figure 3. Variation in UTS of DP steel quenched in bitumen and water at different holding time.

Table 3. Mechanical properties of Low carbon steel quenched in bitumen and water.

	Hardness (HV)		Ultimate tensile strength (MPa)		Yield strength (MPa)	
Holding time (Min.)	Bitumen	Water	Bitumen	Water	Bitumen	Water
30	145.23	143.98	514.71	510.28	260.87	258.63
40	151.22	148.87	535.94	527.61	271.63	267.41
50	167.72	169.32	594.42	600.09	301.27	304.14
60	169.67	172.46	601.33	611.22	304.77	309.78

3.1.1. Ultimate tensile strength

There is a uniform increase in strength as the holding time increases for bitumen and water quenched samples. DP steel quenched in water shows higher strength at a holding time of 50 and 60 min as compare to bitumen quenched samples. The reduction in the Ultimate tensile strength (UTS) at a lower holding time, could be as a result of higher percentage volume of austenite formation and reduction in the ferrite matrix or may be due to the effect of residual stress induced while quenching [16]. The value of UTS agrees with literature, which states that UTS of DP steel ranges between 500 to 1200Mpa [9]. Figure 3 shows the variation in the UTS of bitumen and water quenched samples. Water quenched gives a better strength at a higher holding time than bitumen quenched (figure 4). Water quenched samples shows 61.68% strength higher than the as-received while the bitumen quenched shows 61.88% at a holding time of 30 min (figure 5).

3.1.2. Hardness

Figures 6 and 7 shows hardness variation which is in line with the tensile test result except for considerable deviation in the values of the quenched samples increases as holding time increases. As discussed in the tensile test result, the reason might be as a result of residual stress been introduced while quenching.

3.1.3. Yield strength

The result of the yield strength (figure 8) is comparable with that of the tensile strength. For the as-quenched bitumen samples, the YS increases with an increase in holding time. But for water quenched the value of yield strength at a holding time of 50 and 60 min increased more. A prolong holding time shows improved toughness due to the formation of more austenite transforming into martensite.

3.2. Further study

From figures 3, 7 and 8, it was observed from holding time of 40 to 50 min, the curve slope changes with a different rate. It is therefore expedient to carry out a further study between 40 to 50 min holding time. At an Intercritical annealing temperature of 800 °C and holding time of 42, 44, 46 and 48 min, the samples were heat-treated and quenched rapidly in bitumen at a temperature of 230 °C. this procedure was repeated for another four samples and quenched in water at room temperature. The result obtained is given in table 4.

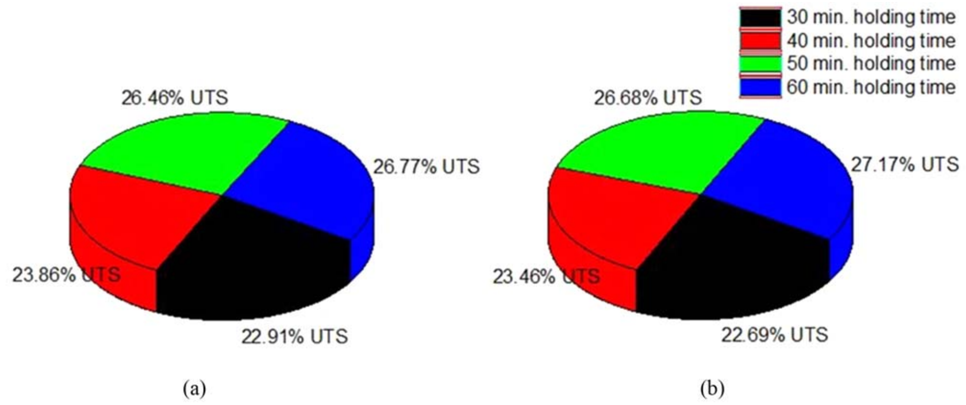

Figure 4. % Change in value of UTS of DP steel at different holding time (a) bitumen quenched (b) water quenched.

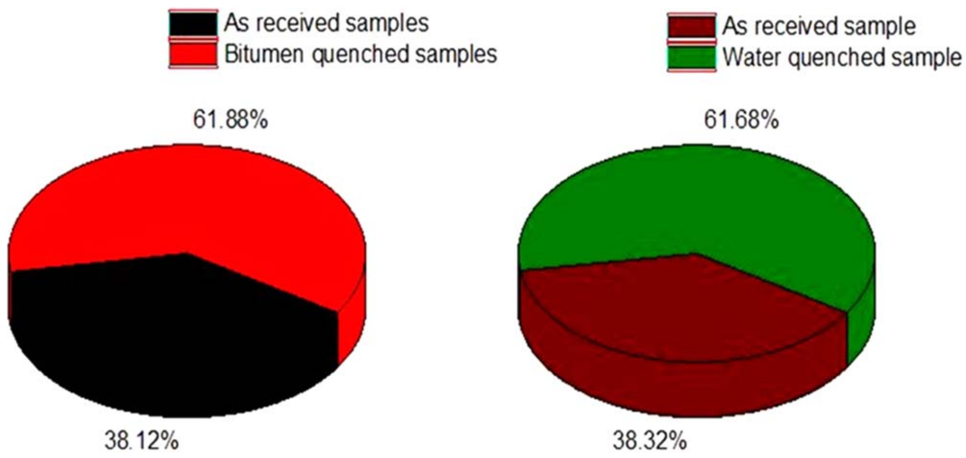

Figure 5. % Variation in UTS for DP steel (quenched in bitumen and water) and as-received samples.

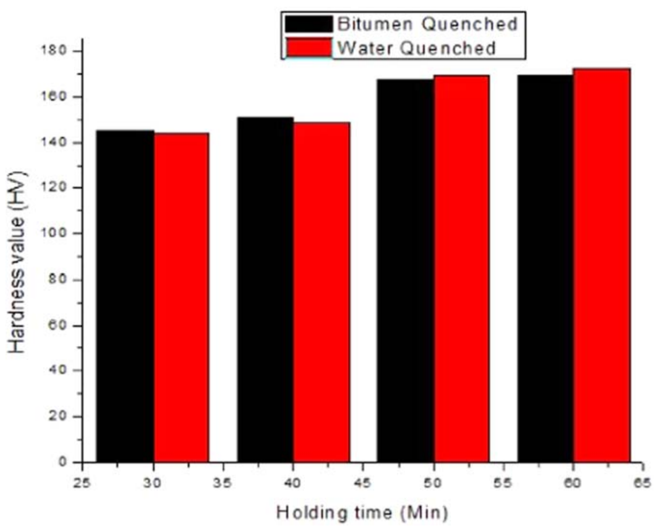

Figure 6. Variation in Hardness of DP steel quenched in bitumen and water at different holding time.

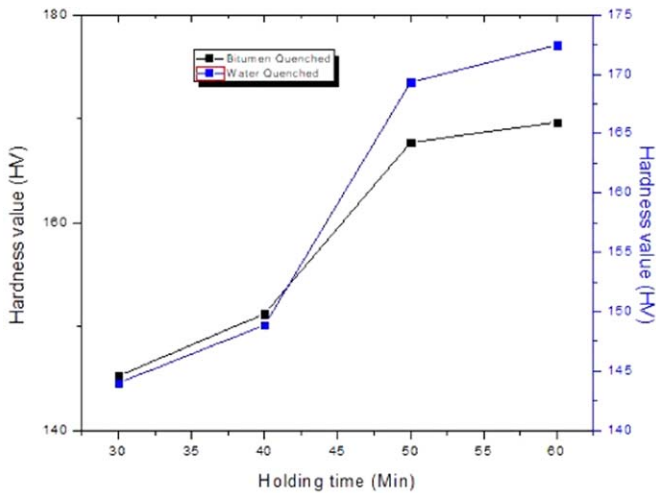

Figure 7. Variation in Hardness of DP steel quenched in bitumen and water at different holding time.

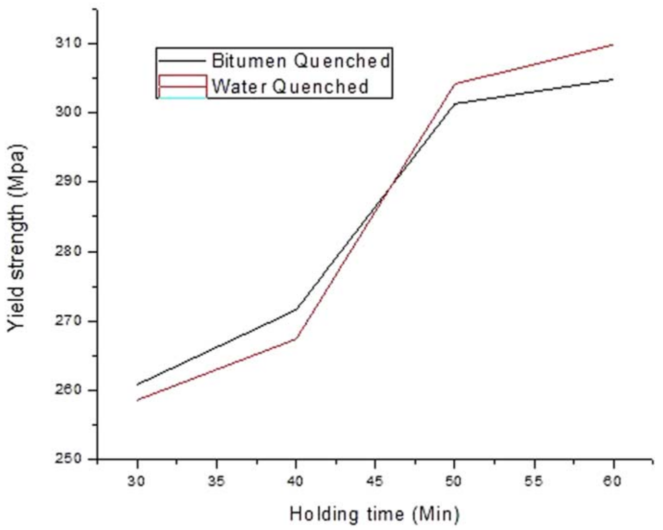

Table 4. Mechanical properties of Low carbon steel quenched in bitumen and water.

	Hardness (HV)		Ultimate tensile strength (MPa)		Yield strength (MPa)	
Holding Time (Min.)	Bitumen	Water	Bitumen	Water	Bitumen	Water
42	153.64	152.76	544.52	541.40	275.98	274.40
44	158.28	160.45	560.96	568.65	284.31	288.21
46	164.39	165.09	582.62	585.10	295.29	296.55
48	165.87	168.04	587.86	595.55	297.95	301.84

Figures 9–11 gives a representation of Hardness, Ultimate tensile strength, and the yield strength. It was observed that as the holding time increases the rate at which the strength increases dropped. As stated earlier the reason could be attributed to reduction in the ratio of percentage volume of austenite transforming into martensite, and the structural arrangement of martensite and ferrite.

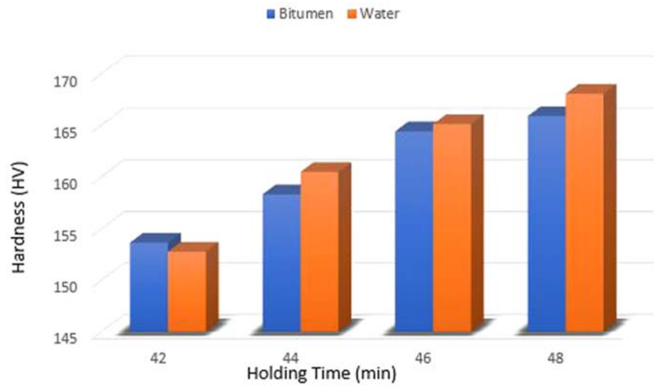

Figure 9. Variation in Hardness of DP steel quenched in bitumen and water at different holding time.

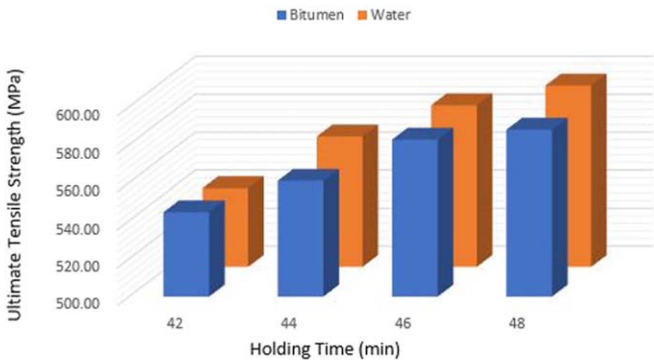

Figure 10. Variation in UTS of DP steel quenched in bitumen and water at different holding time.

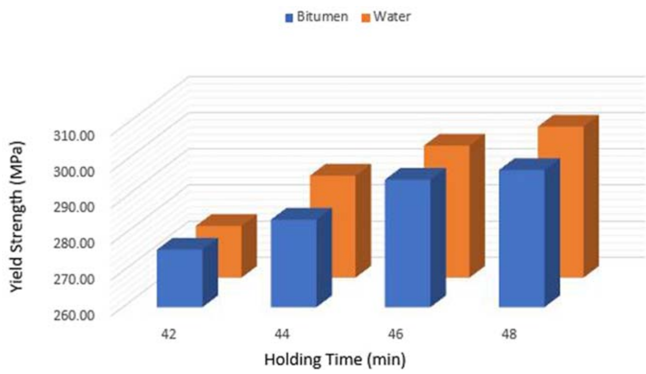

Figure 11. Variation in Yield Strength of DP steel quenched in bitumen and water at different holding time.

3.3. Microstructure analysis

Figure 12 (A)–(C) represents the microstructure obtained for different dual phase conditions. Lower holding time represents samples with finer and lesser dark regions (martensite phase). As the holding time increases there is a considerable increase in the grain size of austenite formed, the amount and size of martensite formation also increase [18, 29]. Figures 12(A) and 9(B) (a), (b), (e), (f)) represent samples quenched in bitumen while (c), (d), (g), (h) represent samples quenched in water. The distribution pattern of martensite of the water quenched samples shows isolation patterns within the ferrite matrix at a holding time of 50 and 60 min. The higher percentage volume of austenite formation transforming into martensite could be observed and a considerable reduction in the ferrite matrix. This makes the sample tends to have improved strength and ductility than the chain-like distribution pattern exhibited by the bitumen quenched samples, as seen in figures 3–8. The result agreed with the preposition of Xiong *et al* (2015) [9]. However, the dual phase formed at

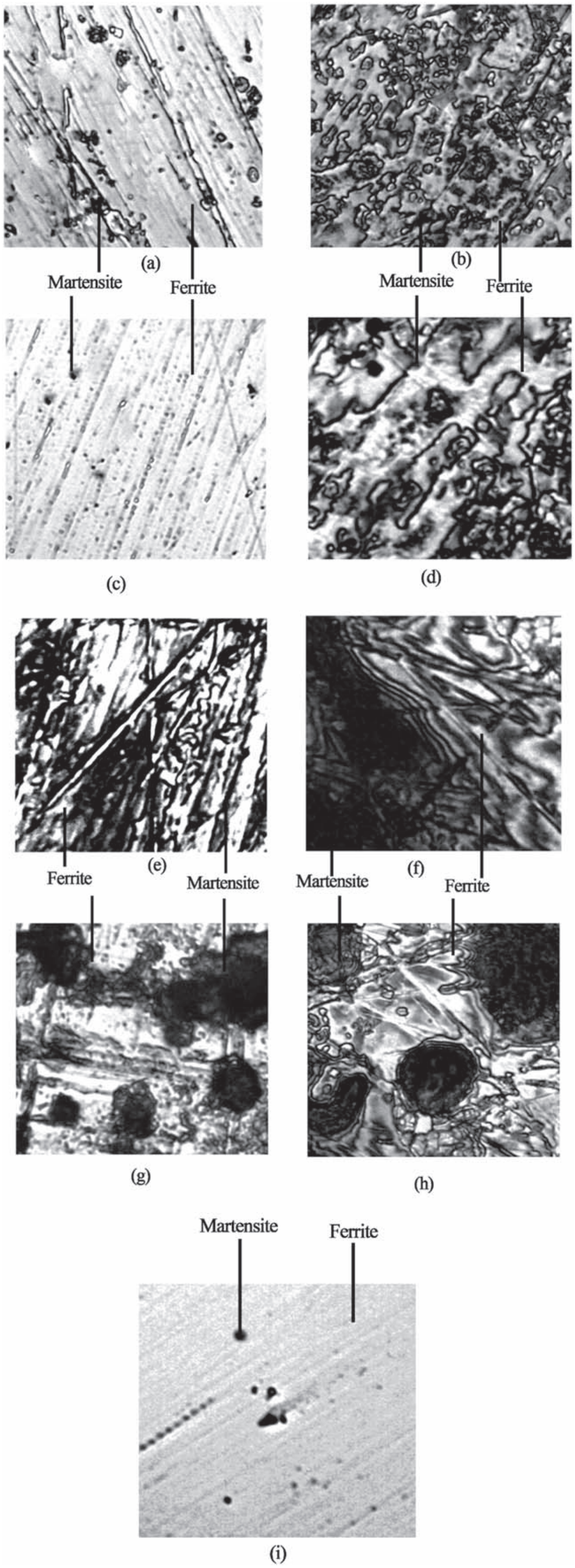

Figure 12. (A) Microstructure of specimens austenitized at 800 °C at [500×]. (a) Dual phase quenched in bitumen at holding time 30 min. (b) Dual phase quenched in bitumen at holding time 40 min. (c) Dual phase quenched in water at holding time 30 min. (d) Dual phase quenched in water at holding time 40 min. (B) Microstructure of specimens austenitized at 800 °C at [500×] (e) Dual phase quenched in bitumen at holding time 50 min. (f) Dual phase quenched in bitumen at holding time 60 min. (g) Dual phase quenched in water at holding time 50 min. (h) Dual phase quenched in water at holding time 60 min. (C) Microstructure of specimens austenitized at 800 °C at [500×]. (i) As-bought steel normalized.

800 °C, and hold for a period of 50 and 60 min shows not only finer but also larger total dark regions (figures (e)–(h)). Figure 12(C) shows the typical structure of normalized hypo-eutectoid steel with a larger region of ferrite and dispersed martensite which makes it softer than the austenitized samples.

4. Conclusion

The heat treatment was effectively used as a means of altering the microstructure and mechanical properties of the DP steel, at an annealing temperature of 800 °C and holding time of 30, 40, 50 and 60 min were separately quenched in water and bitumen. The following conclusions are drawn from the experimental work.

- The lower holding time of the dual phase steel shows lower strength, hardness when compared to that of higher holding time.
- In bitumen quenched samples, the prolonged holding time condition shows a reduction in strength when compared with that of water quenched samples.
- 61.88% reduction in the UTS has observed in as-received samples compare to the bitumen quenched at a holding time of 30 min.
- 61.68% reduction in the UTS has observed in as-received samples compare to the water quenched at a holding time of 30 min.
- Hardness variation in both quenching mediums is minimal.
- Water quenched samples at 50 and 60 min gives better hardness value as compare to bitumen quenched.
- The microstructure of dual phase steel shows a dark region of martensite phases within the ferrite matrix.
- Prolonged holding time duration shows a considerable effect on the microstructure.

It could, therefore, be concluded that the mechanical properties of DP steels vary directly with the microstructure, a change in the structure influence its mechanical properties. From the observation above, the distribution pattern of martensite within the ferrite matrix plays an important role (An isolated martensite region within the ferrite matrix shows an improved strength and ductility as observed in figures 12(B) (g) and (h) than a distribution of chain-like structure surrounded by ferrite matrix isolation patterns within the ferrite matrix as seen in figures 12(B) (e) and (f)).

The fund provided by the National Research Foundation (NRF) program, South Africa, is greatly appreciated.

ORCID iDs

E Olorundaisi https://orcid.org/0000-0002-6842-6134

References

[1] Li Z, Wu D, Lü W, Yu H, Shao Z and Luo L 2015 Effect of holding time on the microstructure and mechanical properties of dual-phase steel during intercritical annealing *J. Wuhan. Univ. Technol. Mater. Sci. Ed.* **30** 156–61
[2] Zuo X, Chen Y and Wang M 2012 Study on microstructures and work hardening behavior of ferrite-martensite dual-phase steels with high-content martensite *Mater. Res.* **15** 915–21
[3] Radwanski K, Kuziak R and Rozmus R 2019 Structure and mechanical properties of dual-phase steel following heat treatment simulations reproducing a continuous annealing line *Arch. Civ. Mech. Eng.* **19** 453–68
[4] Jamei F, Mirzadeh H and Zamani M 2019 Synergistic effects of holding time at intercritical annealing temperature and initial microstructure on the mechanical properties of dual phase steel *Mater. Sci. Eng. A* **750** 125–31
[5] Ch Darabi A, Chamani H R, Kadkhodapour J, Anaraki A P, Alaie A and Ayatollahi M R 2017 Micromechanical analysis of two heat-treated dual phase steels: DP800 and DP980 *Mech. Mater.* **110** 68–83
[6] Alibeyki M, Mirzadeh H and Najafi M 2018 Fine-grained dual phase steel via intercritical annealing of cold-rolled martensite *Vacuum* **155** 147–52
[7] Xiong Z, Andrii K, Nicole S and Elena V P 2014 Effect of holding temperature and time on ferrite formation in dual phase steel produced by strip casting *Mater. Forum* **38** 44–8
[8] Zamani M, Mirzadeh H and Maleki M 2018 Enhancement of mechanical properties of low carbon dual phase steel via natural aging *Mater. Sci. Eng. A* **734** 178–83
[9] Xiong Z P, Kostryzhev A G, Stanford N E and Pereloma E V 2015 Microstructures and mechanical properties of DP and TRIP steels after laboratory simulated strip casting *Mater. Des.* **88** 537–49
[10] Wu S *et al* 2019 Strength-toughness improvement of martensite-austenite dual phase deposited metals after austenite reversed treatment with short holding time *Mater. Sci. Eng. A* **755** 57–65

[11] Zeytin H K, Kubilay C and Aydin H 2008 Investigation of dual phase transformation of commercial low alloy steels: effect of holding time at low inter-critical annealing temperatures *Mater. Lett.* **62** 2651–3
[12] Ashrafi H, Shamanian M, Emadi R and Saeidi N 2017 Examination of phase transformation kinetics during step quenching of dual phase steels *Mater. Chem. Phys.* **187** 203–17
[13] Xiong Z, Kostryzhev A G, Zhao Y and Pereloma E V 2019 Microstructure evolution during the production of dual phase and transformation induced plasticity steels using modified strip casting simulated in the laboratory *Metals (Basel).* **9** 449
[14] Hofinger M, Staudacher M, Ognianov M, Turk C, Leitner H and Schnitzer R 2019 Microstructural evolution of a dual hardening steel during heat treatment *Micron* **120** 48–56
[15] Balbi M, Alvarez-Armas I and Armas A 2018 Effect of holding time at an intercritical temperature on the microstructure and tensile properties of a ferrite-martensite dual phase steel *Mater. Sci. Eng. A* **733** 1–8
[16] Gurumurthy B M, Sharma S S and Kini A 2018 Ferrite-bainite dual phase structure and mechanical characterization of AISI 4340 steel *Mater Today Proc.* **5** 24907–14
[17] Chaudhari RYPASC D B 2015 Effect of Quenching parameters on material characteristics of specimen- an overview *Int. J. Mech. Prod. Eng. Res. Dev.* **5** 25–32
[18] Amuda M O, Olaniyan T, Osoba L and Akinlabi E 2017 Mechanical properties of bitumen quenched dual phase steel *Sains Malaysiana.* **46** 743–53
[19] Alaneme K K, Ranganathan S and Mojisola T 2010 Mechanical Behaviour of Duplex Phase Structures in a Medium Carbon Low Alloy Steel *J. Miner. Mater. Charact. Eng.* **9** 621–33
[20] Kumar A, Singh S B and Ray K K 2008 *Influence of Bainite/Martensite-Content on the Tensile Properties of low Carbon dual-Phase Steels.* **474** 270–82
[21] Il Y, Kook Y and Park K 2005 *Ultrafine Grained Ferrite—Martensite Dual Phase Steels Fabricated via Equal Channel Angular Pressing: Microstructure and Tensile Properties.* **53** 3125–34
[22] Sudersanan P D, Kori N, Aprameyan S and Kempaiah U N 2012 *The Effect of Carbon Content in Martensite on the Strength of Dual Phase Steel.* **2** 1–4
[23] Adebiyi D I and Popoola A P I 2015 Mitigation of abrasive wear damage of Ti–6Al–4V by laser surface alloying *Mater. Des.* **74** 67–75
[24] Cahoon J R, Broughton W H and Kutzak A R 1979 The determination of yield strength from hardness measurements *Metall. Trans.* 1970–1
[25] Ujah C O, Popoola A P I, Popoola O M and Aigbodion V S 2019 Electrical conductivity , mechanical strength and corrosion characteristics of spark plasma sintered Al-Nb nanocomposite *Int. J. Adv. Manuf. Technol.* **101** 2275–82
[26] Masanori F and Keiichi K 2016 An evaluation of mechanical properties with the hardness of building steel structural members for reuse by NDT *Metals* **6** 247
[27] Pavlina E J and Van Tyne C J 2008 Correlation of Yield strength and Tensile strength with hardness for steels *J. Mater. Eng. Perform.* **17** 888–93
[28] Callister W D 2016 *Fundamentals of Materials Science and Engineering.* 5th edn (New york city united States of America: Wiley) (https://doi.org/10.1016/B978-0-444-82877-4.50019-6)
[29] Adediran A A, Aribo S and Amuda M O H 2015 Mechanical properties of dual phase steel quenched in bitumen medium *Leonardo Electron J Pract Technol.* **14** 1–16