

Mechanical Properties of Bitumen Quenched Dual Phase Steel (Sifat Mekanik Bitumen Lindap Keluli Dual Fasa)

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ABSTRACT

The mechanical properties of Dual Phase Steel (DPS)-duplex structure-produced by quenching in pre-heated bitumen have been investigated. Medium carbon steels intercritically heated at different temperatures and holding times were quenched in hot bitumen. Optical and scanning electron microscopy characterisation of the duplex structure showed extensive network of fibrous martensite in a ferritic matrix with occasional presence of polygonal martensite. The duplex phase structure exhibited continuous yielding dynamics, improving the tensile and hardness values by about 42 and 35%, respectively, relative to the normalised structure. But, the elongation and impact values decreased by about 42 and 50%, respectively, when compared to the normalised structure. These values are similar to those obtained in duplex structure produced using conventional oil quenching. The tensile fractured surface showed transition between a predominantly cleavage mode in the lower annealing temperature to a mixed mode in the upper bound of the annealing temperature. These findings suggest that pre-heated bitumen can be exploited for the production of DPS.

Keywords: Duplex structure; fibrous martensite; intercritical annealing; tensile fractured surface

ABSTRAK

Sifat mekanik struktur Keluli Fasa Dual (DPS)-dupleks yang dihasilkan melalui pelindapan dalam bitumen pra-panas telah dikaji. Keluli karbon sederhana dipanaskan secara kritikal pada suhu yang dan masa pegangan berbeza telah dilindap dalam bitumen panas. Pencirian mikroskop optik dan elektron imbasan oleh struktur dupleks telah menunjukkan rangkaian luas oleh martensit berserabut dalam matriks ferit dengan sesekali kehadiran poligon martensit. Struktur fasa dupleks mempamerkan hasil berterusan yang dinamik, menambahbaik nilai tegangan dan kekerasan masing-masing sebanyak 42 dan 35%, relatif kepada struktur normalan. Namun, pemanjangan dan nilai impak menurun masing-masing sebanyak 42 dan 50% jika dibandingkan dengan struktur normalan. Nilai ini adalah sama seperti yang diperolehi dalam struktur dupleks yang dihasilkan dengan menggunakan minyak pelindapan konvensional. Permukaan retak tegangan menunjukkan peralihan antara mod belahan pradominan dalam suhu sepuh lindap rendah kepada mod campuran dalam batas atas suhu sepuh lindap. Kajian ini mencadangkan bahawa bitumen prapanas boleh dieksploitasi untuk pengeluaran DPS.

Kata kunci: Martensit berserabut; permukaan retak tegangan; sepuh lindap antara kritikal; struktur dupleks

INTRODUCTION

In the last three and half decades, stringent energy/service economics and environmental considerations have necessitated the drive for the development of advanced steel materials with potential for lower carbon footprint discharge without sacrificing on the performance of conventional ferrite-pearlitic steels (Granbom 2010; Hall 2011; Tsipouridis 2006; WorldAutoSteel 2011). There are several grades of such better steels that have been developed and commercially available in the market ranging from transformation induced plasticity (TRIP) steels, high strength low alloy (HSLA) steels to dual phase (DP) steels all belonging to a class of steels called *Advanced High Strength Steels (AHSSs)* (Hall 2011). Recently, however, advancement in these steels has focused mainly on the DPS because this grade of steel exhibits superior strength and plasticity relative to other high strength low alloy steels (Mazinani & Poole 2007; Sun & Pugh 2002; Xu et al. 2008). This class of high-

strength low-alloy steel has a microstructure consisting strong martensite and/or bainite colonies dispersed in a soft matrix of ferrite (Ghosh et al. 1991). Instances of the additional presence of retained austenite in the ferrite matrix have also been reported but this has not been shown to have significant influence on the improved properties of the material (Bayram et al. 1999; Krajewski & Nowacki 2014; Rashid 1981; Tayanc et al. 2007). The martensite content in the steel which usually range between 15-25% coupled with its morphology influences the mechanical properties of the steel. But, irrespective of the distribution and forms of the martensite, the DPS is reported to exhibit continuous yielding with no yield point, lower yield strength/tensile strength ratio, higher initial strain hardening rate, uniform elongation and higher strain rate sensitivity with better fatigue resistance relative to conventional ferrite-pearlite plain carbon steel (Bello 2007; Bhattacharyya et al. 1993; Mazinani & Pool 2007; Tasan et al. 2015). These combinations of strengths and formability have made DPS

very attractive to industries, particularly, the automobile sector; and there is indication that this could equally be extended to many other areas particularly those related to structural applications (Fazaeli et al. 2016; Khamedi et al. 2009; ThyssenKrupp Steel Europe 2008). The duplex structure in DPS offers the potential for combining the strength of conventional high strength low alloy steels with a formability approximating that of plain carbon steel.

Literature indicate that these steels are produced either by heating low carbon/micro alloyed steel into the intercritical range between the A_1 and A_3 temperatures on the iron-carbon constitution diagram within the continuous transformation cycle for a holding period usually less than one hour, follow by rapid cooling to freeze the high temperature microstructure (quenching) or directly off the hot rolling mill by the control of composition and processing conditions (Khamedi et al. 2009; Rashid 1981). Traditionally, quenching heat treatment is employed to strengthened, hardened or softening carbon steel and alloys depending on the carbon content. The A_1 represents the lower critical temperature on the iron-carbon constitution diagram below which austenite is not stable; and also the temperature of eutectoid transformation. This temperature corresponds to 723°C on the diagram; while the upper critical temperature at 910°C on the diagram is the temperature below which ferrite starts to form from austenite. However, considerable research and production activity have been reported more for the continuous heating than controlled rolling (Fallahi 2002; Khamedi et al. 2009). This arose because continuous heating (annealing) offer some attractions compared to controlled rolling such as lower manganese content, lesser variation in mechanical properties, production of well dispersed low-carbon martensite in a ferrite matrix (Kuziak et al. 2008). Furthermore, the process offers inherent control of volume fraction and composition of the ferrite and austenite as dictated by the equilibrium phase diagram. It is equally reported that the high dislocation density at the interface with martensite facilitates the continuous yielding behaviour of the steel (Gbasemi-Armaki et al. 2014). During quenching heat treatment, the choice of quenching media is critical to the ensuing microstructure aside the possibility of defects such as distortion and/ or cracking. The morphology and volume fraction of martensite formed during quenching from the intercritical temperatures are dependent on the chemistry of the quenching medium. Therefore, quenching medium ranging from water, oil, salt solution and liquid metal bath has been exploited for the production of DPS (Rashid 1981). Water or brine solution quenching is recommended for steel with lower hardenability while oil is preferable for micro-alloyed steel (Alaneme et al. 2010). In the recent past, however, oil quenching seems to be the choice for the production of DPS because it eliminates the tendency for the development of quench cracks in the steel. Thus, extensive works have been reported on dual phase structure in micro alloyed low carbon produced via oil quenching (Akay et al. 2009; Kumar 2008; Son et al. 2005); but very little has been reported on dual phase

structure in medium carbon steel (Alaneme et al. 2010). This is attributed to the high incidence of retained austenite or bainite in intercritically annealed medium carbon steel which affects the deformation behaviour of the steel (Ersoy 2013). However, Xiurong et al. (2012) reported that dual phase structure in medium carbon steel with higher martensite volume exhibits superior tensile strength and wear resistance compared to low carbon micro-alloyed steel. Adamczyk and Grajcar (2007) reported similar findings. This suggests that dual phase structure could be produced in medium carbon steel.

Different investigators have utilized various oils ranging from synthetic to organic oils such as palm kernel and red oil (palm oil) as quenchant for the production of DPS (Adeyemi & Adedayo 2009; Herring 2010). The attraction in the organic oils is their bio-degradability and compatibility with the environment. However, these organic oils are food materials and using them as quenchant may create consumption deficit. This therefore demands sourcing alternatives to these organic oils.

Nigeria is credited with a vast deposit of bitumen stretching between 4-6 km in the east-west belt in Ondo and Ogun states (Adegoke 1980). Bitumen is a dark highly viscous supercooled hydrocarbon based oil mineral that could be exploited as a quenchant for the production of DPS (Ause 2008). Information on such possibility in literature is scarce except for the work of Alabi et al. (2013) which investigated the influence of hot bitumen austempering on the mechanical properties of medium carbon steel. Therefore, in this work, pre-heated bitumen is investigated for the production of DPS by characterising the associated mechanical properties in relation to normalized steel samples of the same composition. This is essentially an exploratory study whose findings are imperative for a wide scale adoption of the material as a possible substitute to organic vegetable oil as a quenchant for the production of DPSS.

MATERIALS AND METHODS

PRELIMINARY TREATMENT

The dark supercooled bitumen shown in Figure 1 was heated and melted in a stainless pot fired by a fossil fuel chamber to boiling point. Small quantity of about 20 mL was poured into a PENSKEY MARTENS close-cup flash point tester with facility for temperature reading. The temperature at the point the orifice in the tester released a flash of fire was noted as 310°C and recorded as the flash point. This value is both lower than the boiling point (350°C) and flash point (378°C) values available in literature for bitumen obtained from fractional distillation of crude oil (Speight 1992). This showed that the liquid bitumen must maintain a temperature level below 310°C during the quenching cycle otherwise boiling and flashes of fire would occur resulting in fire incidence. Thus, the bitumen quenchant was maintained at a pre-heated temperature level of 280°C for the quenching cycle.



FIGURE 1. Dark supercooled bitumen before melting

Hot rolled carbon steel supplied as cylindrical rods of $\varnothing 12 \times 1000$ mm specification was cut-into coupon size of $\varnothing 12 \times 80$ mm long. The composition of the steel, as provided by the manufacturer and validated via in-house spectroscopy, presented in Table 1 in comparison with published data (Alaneme 2010; American Iron and Steel Institute 2014) shows that the material belongs to the medium carbon steel grade. The coupons were degreased in ethanol solution; descaled using metal brush and subsequently subjected to normalizing treatment to

remove the thermal and mechanical history of the steel. The normalizing treatment was conducted at 870°C for 45 min in a muffle furnace followed by air cooling.

$$A_1 = 723 - 10.7(\%Mn) - 16.90(\%Ni) + 29.1(\%Si) + 16.9(\%Cr) + 290(\%As) + 6.38(\%W) \quad (1)$$

$$A_3 = 910 - 203(\%C)^{1/2} - 15.2(\%Ni) + 44.7(\%Si) + 104(\%V) + 13.1(\%W) \quad (2)$$

INTERCRITICAL ANNEALING TREATMENT

The intercritical (A_1 and A_3 , Figure 2) annealing temperatures for the coupons were estimated from the chemical composition using the empirical relationship provided by Gorni (2006), Leslie (1981) and represented by Khamedi et al. (2009). The lower critical temperature was calculated using (1) while (2) was used to determine the upper critical temperature and these were found to be 722.4 and 804.2°C, respectively. Therefore, based on these values, five levels of intercritical temperatures and three levels of holding times presented in Table 2 were considered for the heating cycle.

TABLE 1. Chemical composition of the hot rolled steel

Material specification	Composition (wt. %)						
	C	Si	Mn	P	S	Fe	Trace elements
Medium carbon steel	0.327	0.231	0.73	0.036	0.033	96.3	Balance

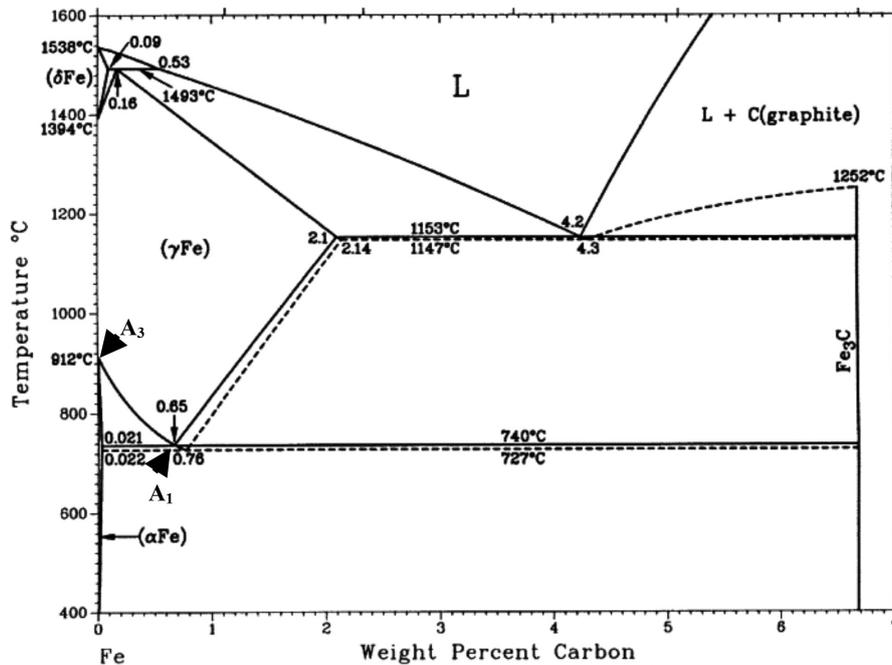


FIGURE 2. Iron-carbon constitution diagram with the indication of both the lower (A_1) and upper (A_3) critical temperatures (Adapted from: Massalski et al. 1990)

TABLE 2. Combination of temperature and holding time for intercritical annealing

Treatment Temp (°C)	Holding time (min)		
	30	45	60
730	X	X	X
750	X	X	X
770	X	X	X
790	X	X	X
810	X	X	X

The coupons were subsequently intercritically treated at the various combinations of temperature and holding time listed in Table 2 and then rapidly quenched in hot bitumen maintained at a preheated temperature of 280°C. The surface of the quenched pieces was then cleaned off and then minimally ground to remove oxidised surface scales before metallographic preparation.

MICROSTRUCTURAL AND MECHANICAL CHARACTERISATION

The treated coupons were prepared for microstructural evaluation by grinding successively in a series of emery papers of grit sizes 240, 320, 400 and 1000. Final polishing was done with 0.05 µm alpha agglomerated alumina suspension. Etching was conducted with 2% Nital solution for 5-10 s just before microscopic examination.

Macrohardness of the coupons were evaluated using a Brinell hardness tester. Polished surface of the samples were indented with a load of 500 gf until a permanent indentation was achieved. The diameter of the indentation was measured with a Brinell reading microscope and the corresponding hardness number was obtained from the conversion table. This test was repeated thrice and the average was taken as representative value.

Tensile testing repeated thrice was conducted at room temperature on appropriately machined sample in an Instron machine rated 100 kN at a strain rate of $2 \times 10^{-3} \text{ s}^{-1}$ in accordance with ASTM E8M standards (2013). The specifications of the tensile samples are a gauge diameter of 4 mm, gauge length 10 mm and specimen length 40 mm. Morphological characterisation and fractography of

the tensile fractured surface were conducted using scanning electron microscope in the secondary electron imaging mode at an applied voltage of 15 kV.

The impact specimens were prepared in accordance with standard procedures having specifications $\varnothing 10 \times 60$ mm, 2 mm depth notch at an angle of 45° and the test was repeated three times.

RESULTS AND DISCUSSION

MICROSTRUCTURAL ANALYSIS

The microstructure of the normalised sample from optical microscope shown in Figure 3 indicates a matrix of ferrite with grain boundary iron carbides and small islands of pearlite. The network of grain boundary iron carbide is very wide. Rashid (1981) reported similar microstructural features in normalised medium carbon steel whereas most published works have only reported on the existence of ferrite-pearlite microstructure in such normalised steels.

Figure 4 shows the microstructure of samples intercritically treated at 730°C for 30, 45 and 60 min, respectively. The microstructures at these conditions consist of ferrite matrix networked by polygonal martensite. The distribution of martensite estimated using graduated eye piece show increasing volume fraction of martensite with increasing soaking time. It is presumed that the low distribution of martensite at lower soaking time of 30 min is caused by the short heating times resulting in a non-homogeneous austenite. This inhibits complete diffusion of carbon into the austenite phase and

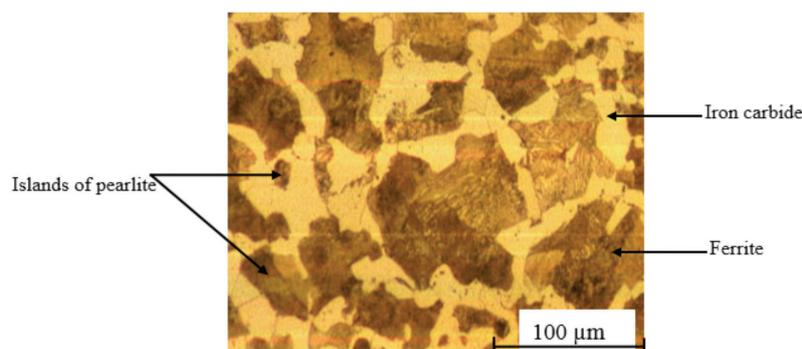


FIGURE 3. Microstructure of normalised sample before intercritical treatment

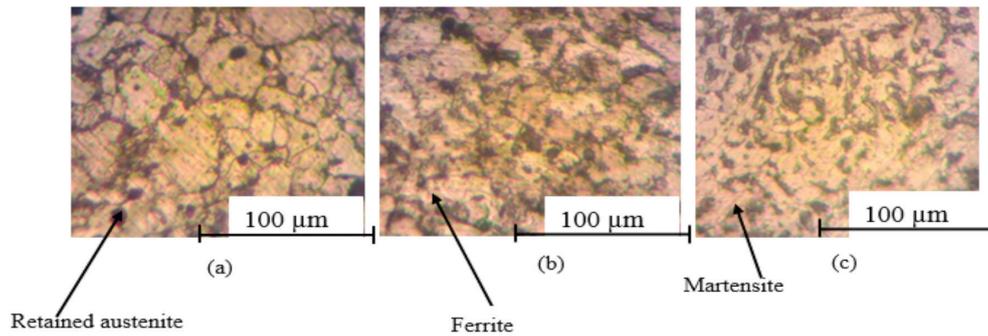


FIGURE 4. Microstructures of samples treated at 730°C at different holding times (a) 30, (b) 45 and (c) 60 min

a concentration gradient exists. With increasing soaking time, however, carbon diffusion is enhanced and a more homogeneous austenite evolves resulting in higher martensite distribution. The morphology of the martensite phase transits from polygonal to fibrous martensite at maximum soaking time of 60 min but with apparent increase in grain morphology.

Figures 5 to 8 are the micrographs obtained at intercritical treatments of 750, 770, 790 and 810°C for holding times of 30, 45 and 60 min, respectively. The phase distribution in these micrographs is similar to those obtained at 730°C intercritical treatment; except that the morphology of the martensite is more fibrous at these higher intercritical temperatures than the polygonal obtained at 730°C.

Previous works reported the additional presence of retained austenite in ferrite-martensite dual phase structure produced through intercritical treatment (Giodarno et

al. 1991) but such retained austenite is only observed in samples treated at 730°C for all soaking times but this is not seen in other treatment conditions (Figures 4-8). Therefore, there is a need for more extensive study in this regard with diffraction technique; because if this is validated, it presents an innovative strategy for improving the deformation behaviour of such dual phase structure resulting in improved formability of the steel.

The optical micrographs at the various intercritical treatment conditions were reinforced with scanning electron microscopy for detailed morphological analysis. These electron micrographs shown in Figure 9 shows fibrous martensite whose morphology become coarser with increasing intercritical temperature and holding time with those treated at 810°C and 45 min providing the most fibrous network of martensite in a ferritic matrix. The distribution of the martensite network also increased with increasing intercritical annealing temperatures.

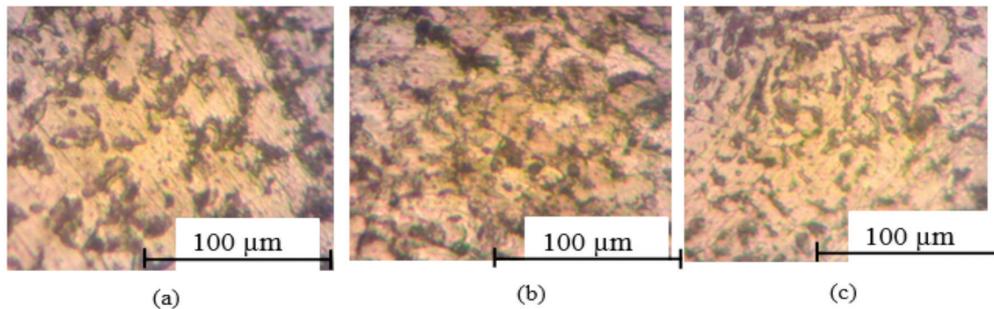


FIGURE 5. Microstructures of samples treated at 750°C at different holding times (a) 30, (b) 45 and (c) 60 min

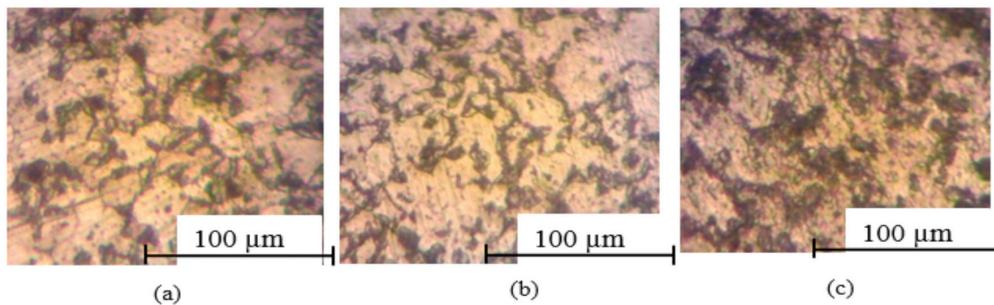


FIGURE 6. Microstructures of samples treated at 770°C at different holding times (a) 30, (b) 45 and (c) 60 min

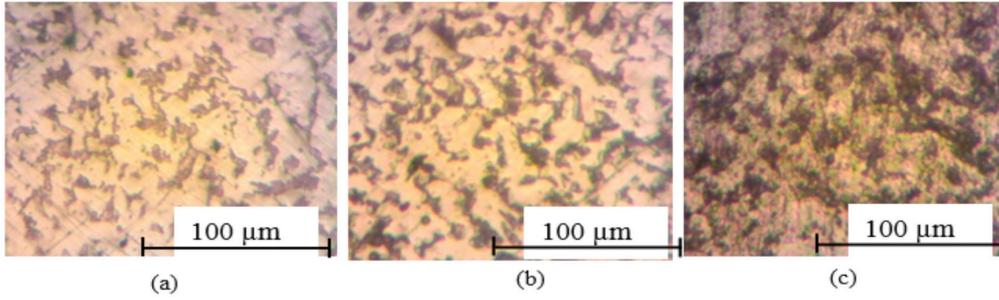


FIGURE 7. Microstructures of samples treated at 790°C at different holding times (a) 30, (b) 45 and (c) 60 min

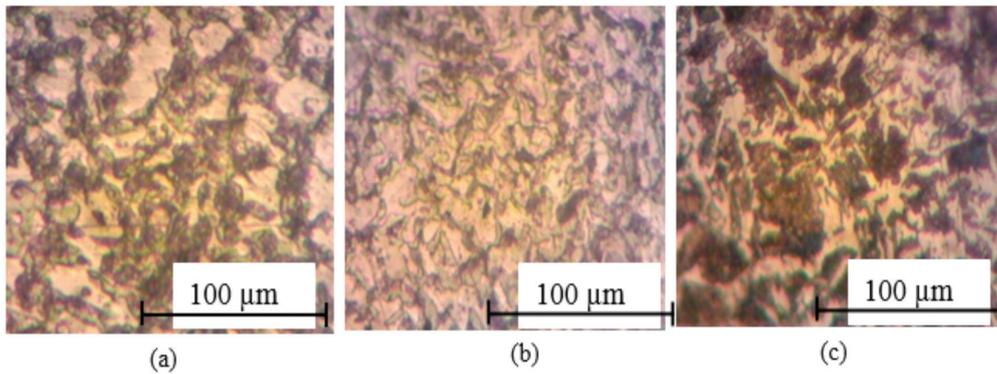


FIGURE 8. Microstructures of samples treated at 810°C at different holding times (a) 30, (b) 45 and (c) 60 min

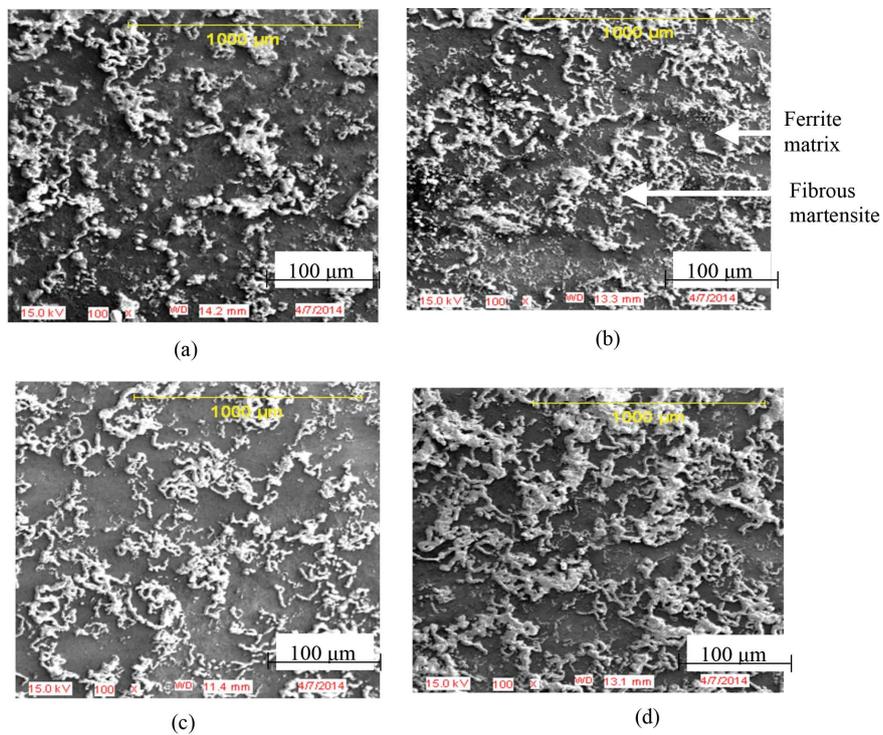


FIGURE 9. SEM morphology of samples intercritically treated at different temperatures at a holding time of 45 min: (a) 750, (b) 770, (c) 790 and (d) 810°C

ANALYSIS OF MECHANICAL PROPERTIES

The mechanical properties of the normalised sample presented in Table 3 shows a bulk hardness of 95 HBN,

tensile strength of almost 487 MPa, percent elongation of 17% and impact energy of 227 J.

TABLE 3. Mechanical properties of normalised medium carbon steel

Sample designation	Tensile strength (MPa)	Elongation (%)	Impact energy (J)	Hardness (HBN)
Normalised sample	486.90	17.06	227	95

Figure 10 shows the change in bulk hardness of bitumen quenched samples at different intercritical treatment temperatures. The bulk hardness increases with increasing holding time across all treatment temperatures with those held for 60 min exhibiting the highest hardness value. The hardness between 730 and 810°C range between 100 and 120 HBN for holding time of 30 min; and for 60 min, it range between 120 and 180 HBN. The values for those held at 45 min lies within these two extreme holding times. The hardness values from the quenched samples are higher than that of the normalised sample by about 16-90% across all the treatment conditions. The higher values in the quenched samples is predicated on the microstructure in the samples, wherein, the normalised samples consist of ferrite and iron carbide phases the quenched samples consist of dual phase structure of ferrite and martensite.

The higher hardness is primarily due to the martensite distribution in the ferrite matrix. Increasing intercritical temperature and holding time increases the distribution of austenite in the two phase structure which eventually transforms to martensite on quenching in the preheated bitumen. Martensite is the known phase responsible for hardness in the dual phase steel (Rashid 1981).

Figure 11 provides the trend of tensile strength in the quenched samples. The strength increases with increase in intercritical temperature up to a temperature of 770°C and subsequently reduces afterwards. The reduction is rapid at holding times of 30 and 40 min but gradual at holding time of 60 min. The minimum tensile strength value range between 501 and 566 MPa in the treatment temperature range 730-810°C for 30 min holding time. The range is 529 - 612 MPa for the same condition of temperature and 45 min holding time while for 60 min, the strength range

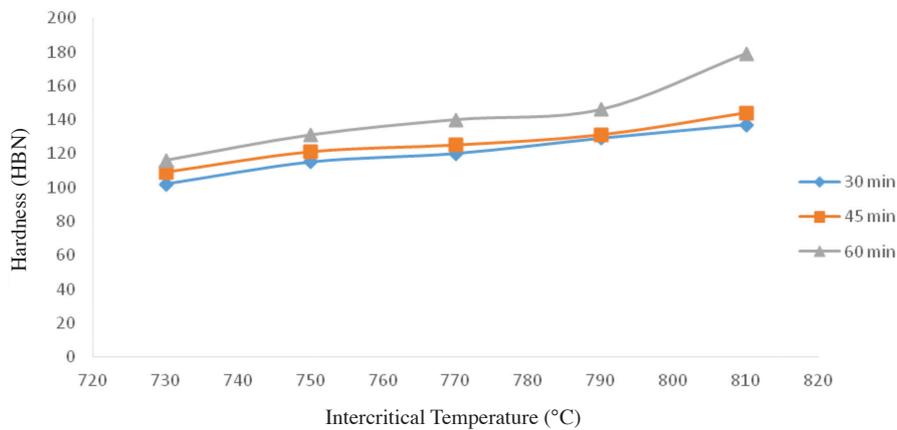


FIGURE 10. Hardness variations with intercritical treatment conditions

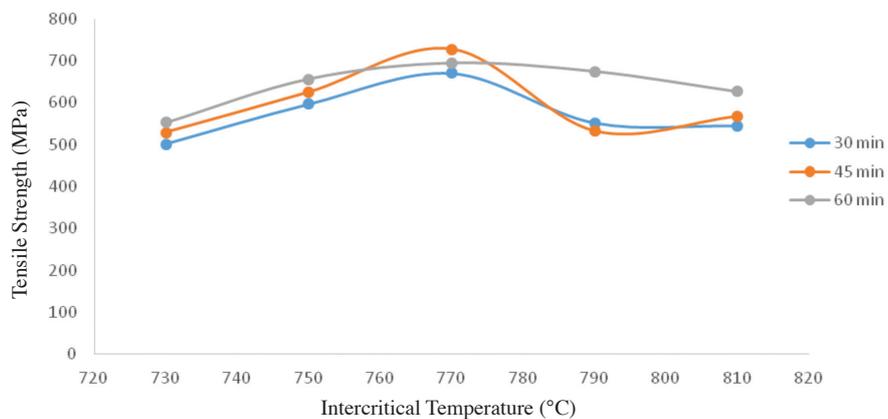


FIGURE 11. Tensile strength of dual phase steel at different intercritical temperatures and holding times

between 535 and 626 MPa. The maximum tensile strength for the quenched samples ranges between 670 and 693 MPa at a specific treatment temperature of 770°C.

Insert Figure 11 here

These strength values are greater than the 487 MPa obtained in the normalised sample. The reduction in tensile strength at treatment temperature beyond 770°C can be predicated on the development of coarse martensite observed in Figures 6 and 9 as a result of high temperature treatment. Ekrami and Bahrehbarpoor (2005) and Odusote et al. (2012) reported similar findings of drastic decrease in tensile property. But Alaneme et al. (2010) reported continuous increase in the tensile strength of oil quenched dual phase steel up to intercritical temperature of 780°C. This contrasting information might be due to the chemistry of the steel samples since the samples investigated in the reported works were not of the same composition. In general, increase in hardness with increase in soaking time of treatment are accompanied with increase in strength, however, the formation of coarse martensite at intercritical temperature could alter the trend as observed in the current study.

The plastic flow trend in the dual phase structure with changes in intercritical temperature and holding

time characterised in term of the percent elongation is shown in Figure 12. It emerged from the figure that the percent elongation decreases with increase in intercritical temperature but the influence of the holding time were generally inconsistent.

Intercritical treatment at 730°C for 30 min exhibits the highest percent elongation of 24% whilst that at 810°C is just about 19% for the same holding time. Similar trend is observed across the intercritical temperatures at holding times of 45 and 60 min. The percent elongation in the quenched dual phase steel is greater than that of the normalised samples except for the value at 810°C for a holding time of 60 min. In this instance, the percent elongation is marginally lower by about 3 units.

It is believed that higher intercritical temperature promotes the transformation of ferrite to austenite and this increases the distribution of martensite in the final dual phase structure. Thus, with increased martensite distribution in the matrix the plastic flow decreases and the percent elongation is consequently reduced.

Figure 13 shows the impact energy of the dual phase steel as a function of intercritical temperatures and holding times. The figure is similar to that of the percent elongation decreasing beyond 730°C across the intercritical all

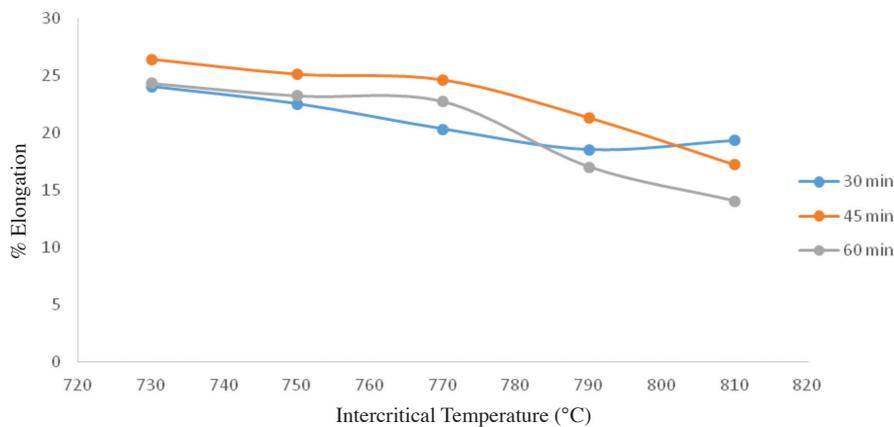


FIGURE 12. Percent elongation of dual phase steel at different intercritical temperatures and holding times

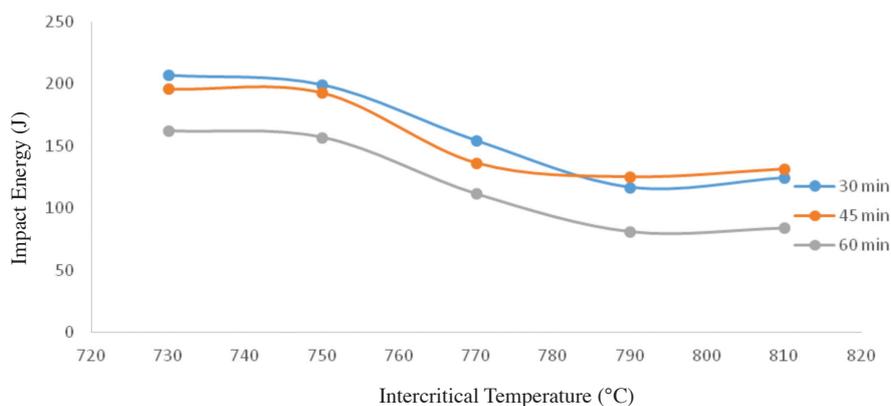


FIGURE 13. Impact energy at different intercritical temperatures and holding times

temperatures for all the holding times with the minimum energy being about 150 J at 730°C and holding time of 60 min while the maximum is about 220 J at 30 min. The impact energy in the quenched dual phase steel is lower than that of the normalised sample.

TENSILE SURFACE FRACTOGRAPH

The fractograph shown in Figure 14 shows different fracture mode ranging from purely cleavage fracture to mixed mode of cleavage-dimple fracture. The cleavage mode is predominant at the intercritical temperature of 750°C and below while the mixed mode is prevalent in intercritical temperature above 750°C. The fracture mode in the pre-750°C temperature range is presumed to be due to the presence of polygonal martensite while the mixed mode is influenced transition of the polygonal martensite to fibrous martensite. Such fibrous martensite has been reported to facilitate continuous yielding in dual phase steels (Alaneme et al. 2010).

CONCLUSION

The mechanical properties of DPS produced via quenching of medium carbon steel in pre-heated bitumen have been investigated. The followings emerged from the investigation: The martensite morphology in the

DPS is more influenced by the intercritical annealing temperatures than holding times. The morphology is fibrous at higher temperature while polygonal martensite is sparsely distributed at temperature around and below 750°C. The tensile strength and hardness of the DPS are about 42 and 35% higher than that of the normalised samples but the percent elongation and impact energy are lower by about 42 and 50%, respectively. This contrasting finding is due to the increased transformation of ferrite to austenite which eventually yielded hard but brittle martensite in the dual phase structure. The tensile fractured surface showed transition between a predominantly cleavage mode in the lower annealing temperature to a mixed mode in the upper bound of the annealing temperature. These findings approximate the behaviour of DPS produced from conventional oil quenching procedures. This suggests that pre-heated bitumen can be exploited as an alternative to more expensive oil quenchant for the production of DPSs.

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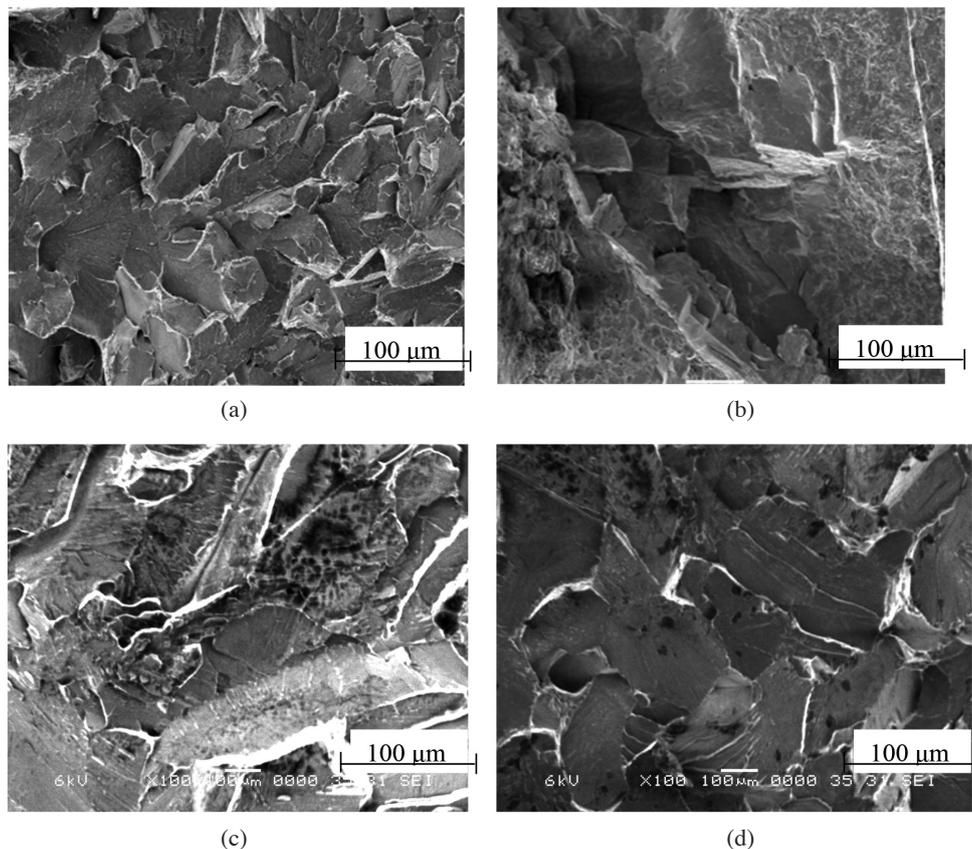


FIGURE 14. Tensile surface fractograph in dual phase steel intercritically treated at (a) 750, (b) 770, (c) 790 and (d) 810°C at holding time of 45 min

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