Unilag Journal of Medicine, Science and Technology

TENSILE PROPERTIES OF PRE-HEATED BITUMEN QUENCHED DUAL PHASE MEDIUM CARBON STEEL

***Amuda, M.O.H., Osoba, L. O. and Olaniyan, T. A.**
Materials Development and Processing Research Group (MADEPREG)
Department of Metallurgical and Materials Engineering
University of Lagos, Lagos, Nigeria 101017
*Corresponding Author: *mamuda@unilag.edu.ng*

ABSTRACT

The tensile 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 pre-heated to 280°C. Optical and scanning electron microscopy characterisation of the duplex structure revealed 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, hardness and elongation by 42, 35 and 45 %, respectively relative to the values obtained in the normalised sample. But, the elongation of the sample heat treated at 810°C for 60 minutes decreased by 18 % when compared to the elongation in the normalised sample. The values obtained are comparable to previously reported investigation in duplex structure produced using conventional oil quenching. This work suggests that pre-heated bitumen can be exploited for the production of DPSs.

Keywords: Duplex-structure, Martensite, Annealing, Tensile Fractured Surface

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 low carbon footprint (reaction of carbon with other elements especially oxygen to produce carbon (II)oxide which leads to greenhouse effect) discharge without sacrificing on the performance of conventional ferrite-pearlitic steels (Hall, 2011; WorldAutoSteel, 2011; Granbom, 2010; Tsipouridis, 2006). There are several grades of such 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, advancement in these steels has focused mainly on the DPS because of their superior strength and plasticity relative to other high strength low alloy steels (Mazinani and Poole, 2007; Sun and Pugh, 2002; Xu *et al.,* 2008). This is a new class of high-strength low-alloy steel having 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 (Tayanc *et al.*, 2007; Bayram *et al.*, 1999; Rashid, 1981). The martensite content in the steel which usually ranges 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 tensile strength ratio, higher initial strain hardening rate, uniform elongation and higher strain rate sensitivity with better fatigue resistance than the conventional ferrite-pearlite plain carbon steel (Mazinani and Pool, 2007; Bello, 2007; Bhattacharyya *et al.*, 1993). 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 (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.

A major variable in the metallurgy and properties of DPS is the quenching medium through which the steel is produced via the intercritical annealing (MacKenzie, 2011). The quenching medium can be influenced by the viscosity. Oil (such as vegetative or synthetic) for instance has a higher viscosity than water and as such, usually produces a DPS with enhanced microstructural features and properties than water. Thus, oil quenching can represent a more attractive process for producing DPS with improved properties than water quenching.

However there are issues of environmental sustenance and bio-degradability associated with conventional synthetic oil quenchant which has motivated the drive for organic oils. While much success has not been reported with organic oil quenchant, there is still the need to develop alternative means to the conventional synthetic oil quenchant. It is in the light of this that bitumen in the pre-heated state is investigated as a possible quenchant for the production of DPS from medium carbon steel.

MATERIALS AND METHODS

Preliminary Treatment

The dark supercooled bitumen shown in Fig. 1 was heated and melted in a stainless pot fired by a fossil fuel chamber to boiling point. 20 ml of bitumen was poured into a PENSKY MARTENS close-cup flash point tester with facility for temperature reading. The flash point temperature in the tester was noted at 310^0C and recorded. This value is both lower than the boiling point (350^0C) and flash point (378^0C) values available in literature for bitumen obtained from fractional distillation of crude oil (Speight, 1992).

Hot rolled carbon steel supplied as cylindrical rods of ∅12 mm x 1000 mm specification was cut into coupon size of ∅ 12 mm x 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, 1995) show that the material belongs to the medium carbon low alloy steel grade. The normalizing heat treatment was conducted at 870^0C for 45 minutes in a muffle furnace followed by air cooling.

Intercritical Annealing Treatment

The intercritical (A_1 and A_3) 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 and upper critical temperatures calculated using those empirical relationships were found to be 722.4 and 804.2^0C, respectively. Therefore, the lower and upper temperature values of 730 and 810^0C and their mid-point were used for the intercritical treatments at soaking times of 30 and 60 minutes.

The coupons were subsequently intercritically treated at 730, 770 and 810°C and holding time of 30 and 60 minutes each respectively followed by rapid quenching in a preheated at 280° bitumen. 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 while final polishing was done with 0.05 μm alpha agglomerated alumina suspension. Etching was conducted with 2% Nital solution for 5-10 seconds 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 500gf 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 100KN Instron machine at a strain rate of $2 \times 10^{-3}s^{-1}$ in accordance with ASTM E8M standards (2013). The specifications of the tensile samples are a gauge diameter of 4 mm, 40 mm gauge length, grip external diameter of 6 mm and grip length of 10 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.

RESULTS AND DISCUSSION

Microstructural Analysis

The microstructure of the normalised sample from optical microscope shown in Fig.2 indicates a matrix of ferrite with grain boundary iron carbides and small islands of pearlite. The network of the grain boundary iron carbide is very wide similar to reported investigation by Rashid (1981) in normalised medium carbon steel. However, most of the published works on normalised medium carbon steel have only reported on the existence of ferrite-pearlite microstructure in such normalised conditions.

Fig. 3 shows the microstructure of samples intercritically treated at 730^{0}C for 30 and 60 minutes, respectively. The microstructures at these conditions consist of ferrite matrix networked by polygonal martensite, formed through diffusionless transformation of austenite after quenching in hot bitumen. 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 minutes is caused by the short heating times resulting in a non-homogeneous austenite. This inhibits complete diffusion of carbon into the austenite phase and a concentration gradient exists. With increasing soaking time, however, carbon diffusion is enhanced and a more homogeneous austenite evolves resulting in higher martensite transformation distribution upon quenching. The morphology of the martensite phase transits from polygonal to fibrous martensite at maximum soaking time of 60 minutes but with apparent increase in grain morphology.

Fig. 4 and 5 are the micrographs obtained at intercritical treatments of 770 and 810^{0}C for holding times of 30 and 60 minutes, respectively. The phase distribution in these micrographs is similar to those obtained at 730^{0}C intercritical treatment; except that the morphology of the martensite is more fibrous at these higher intercritical temperatures than the polygonal obtained at 730^{0}C.

Previous works have reported the additional presence of retained austenite in ferrite-martensite dual phase structure produced through intercritical treatment (Giodarno *et al.*, 1991). In the current study, retained austenite is only observed in samples treated at 730^{0}C for all soaking times but not observable in other heat treatment conditions (see Fig. 3-5). As such, the need for a more extensive study using diffraction technique may help validates the presence of the retained austenite which is an innovative strategy for improving the deformation behaviour of such dual phase structure and 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 Fig 6 revealed fibrous martensite whose morphology become coarser with increasing intercritical temperature and holding time with those treated at 810^{0}C providing the more fibrous network of martensite in a ferritic matrix than the other treatment conditions.

Analysis of Mechanical Properties

The mechanical properties of the normalised sample presented in Table 2 show a bulk hardness of 95 HBN, tensile strength close to 487 MPa and percent elongation of 17%. Fig 7 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 minutes exhibiting the higher hardness value. The hardness between 730 and 810^{0}C range between 100 and 120 HBN for holding time of 30 minutes; and for 60 minutes, it range between 120 and 180 HBN. The hardness values from the quenched samples are higher than that of the normalised sample by about 16-90% across all the treatment conditions (see Table 2 and Fig.7). 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 while the quenched samples consist of dual phase structure of ferrite and martensite. The enhanced hardness is primarily due to martensite distribution in the ferritic matrix (Rashid, 1981). Increasing intercritical temperature and holding time increases the distribution of austenite (as a result of increase in solubility of carbon atoms) in the two phase structure which eventually transforms to martensite on quenching in the preheated bitumen.

Fig. 8 provides the trend of tensile strength in the quenched samples. The strength increases with increase in intercritical temperature up to a temperature of 770^{0}C and subsequently reduces afterwards. The reduction is rapid at holding time of 30 minutes but gradual at holding time of 60 minutes. The minimum tensile strength value range between 501 and 566 MPa in the treatment temperature range 730-810^{0}C for 30 minutes holding time. The range is 535-626 MPa for the same condition of temperature and 60 minutes holding time.

These strength values are greater than the 487 MPa obtained in the normalised sample. The reduction in tensile strength at treatment temperature beyond 770^{0}C can be predicated on the development of coarse martensite observed in Figs. 5 and 8 as a result of high temperature treatment. Odusote *et al.* (2012) and Ekrami and Bahrehbarpoor (2005) 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^{0}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 Fig 9. It emerged from the figure that the percent elongation decreases with increase in intercritical temperature although the influence of the holding time was generally inconsistent. Intercritical treatment at 730^{0}C for 30 minutes exhibits the highest percent elongation of 44% whilst that at 810^{0}C is just about 20% for the same holding time. Similar trend is observed across the intercritical temperatures at holding time 60 minutes except that there was a fall in elongation at 810^{0}C. 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 minutes. In this instance, the percent elongation is marginally lower by about 2-3 units.

In general, 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.

Tensile Surface Fractograph

The fractographs shown in Fig. 10 revealed 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 730°C while the mixed mode is prevalent in intercritical temperature 810°C. The cleavage mode was facilitated by the greater distribution of martensite in the microstructure at these treatment conditions (see Fig. 3-5 and Fig. 10) coupled with presence of microvoids which acted as site for the initiation of brittle fracture. Türkmen and Gündüz (2011) indicated in their work that increasing martensite distribution change the fracture pattern from ductile to brittle. Such fibrous martensite has also been reported to facilitate continuous yielding in DPSs (Alaneme *et al.*, 2010).

CONCLUSION

The tensile properties of DPS produced by quenching in preheated bitumen have been evaluated and the following emerged from the investigation:

i. The tensile strength and hardness of the DPS are about 42% and 35% higher than that of the normalised samples while the percent elongation increased by 45% except at 810°C and holding time of 60 minutes.
ii. This contrasting finding could be due to the increased transformation of ferrite to austenite which eventually yield hard but brittle martensite in the dual phase structure formed during quenching.
iii. The tensile fractured surface revealed transition between a predominantly cleavage mode in the lower annealing temperature to a mixed mode in the upper bound of the annealing temperature.
iv. These findings suggest that pre-heated bitumen can be exploited as an alternative to more expensive oil quenchant for the production of DPSs.

ACKNOWLEDGMENT

The authors are grateful to the management of AKS Steels Limited, Ikorodu, Lagos, Nigeria for the supply of the research material; and Mr. Adediran, Adeolu Adesoji for providing the source bitumen used as the quenching medium.

REFERENCES

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*. 9:621-633.

American Iron and Steel Institute (1995). AISI-SAE Classification of steels: SAE Standard J403f. Accessed from http://www.blueshield.ca/en/docs/pdf/techinfo/databook/e-fsection218to228.

ASTM E8/E8M (2013). Standard test methods for tension testing of metallic materials. ASTM International, *Pennsylvania, USA.*

Bayram, A., Uguz, A. and Ula, M. (1999). Effects of mechanical properties of dual – phase steels. *Mater. Charact.* 9:259 – 269.

Bello, K.A. (2007). Effect of intercritical and tempering heat treatment on the microstructure and mechanical properties of a high martensite dual phase steel, M.Sc. Dissertation, Metallurgical Engineering Department, Ahmadu Bello University Zaria, Nigeria.

Bhattacharyya, A., Sakaki, T. and Weng, G. J. (1993). Influence of martensite shape, concentration, and phase transformation strain on the deformation behaviour of stable dual – phase steels. *Metall. Mater. Trans. A.* 24 (2): 301 – 314.

Ekrami, A. and Bharehbapoor, M. (2005). High temperature behavior of dual-phase steel. *Int. J. Integr. Soc. Sci. Innov.* 2(2): 30-35.

Ghosh, P. K., Gupta, P. C. and Ramavtar; Jha, B. K. (1991). Weldability of intercritical annealed dual phase steel with resistance spot welding process. *Weld. J.* 70(1): 7s-14s.

Giodarno, L., Matteazzi, P., Tiziani, A. and Zambon, A. (1991). Retained austenite variation in dual phase steel after mechanical stressing and heat treatment. *Mater. Sci.and Eng.A.* 131(2): 215-219.

Gorni, A. A. (2006). Steel forming and heat treating handbook. Vol. 2, pp 4. Sao Vacente SP, Brazil.

Granbom, Y. (2010). Structure and mechanical properties of dual phase steels – An experimental and theoretical analysis. Doctoral Thesis, Royal Institute of Technology. Sweden.

Hall, J. N. (2011). Evolution of advanced high strength steels in automotive applications; Power Point Presentation in Great Design in Steel Seminar, pp 1-27. Accessed from http://www.autosteel.org.

Khamedi, R., Fallahi, A. and Zoghi, H. (2009). The influence of morphology and volume fraction of martensite on AE signals during tensile-loading of dual phase steels. *Int. J. Recent Trends in Eng.* 1(5): 30-34.

Leslie, W. C. (1981). The physical metallurgy of steel. Pp 257. McGraw-Hill Series in Materials Science and Engineering, Hempisphere Publishing Corp. Washington DC.

MacKenzie, D. S. (2011). Heat Treating: Selecting the right quenching oil. Accessed from http://www.slideshare.net/HoughtonInternational/ on Friday August 22, 2014.

Mazinani, M. and Poole, W. (2007). Deformation behavior of martensite in a low-carbon dual – phase steel. *Adv. Mater. Res.* 15 – 17:774 – 779.

Odusote, J. K., Ajiboye T. K. and Rabiu A. B. (2012). Evaluation of mechanical properties of medium carbon steel quenched in water and oil. *Assumpt. Univ. J. Technol.* 15(4): 218-224.

Rashid, M. S. (1981). Dual phase steels. *Annual Rev. Mater. Sci.* 11: 245-266.

Speight, J. G. (1992). Asphalt in – Kirk Othmer Encyclopedia of Chemical Technology, 4th edition, Vol. 3, pp 689 – 724, John Wiley and Sons, New York..

Sun, S. and Pugh M. (2002). Properties of thermomechanically processed dual – phase steels containing fibrous martensite. *Mater. Sci. Eng. A.* 335(1-2): 298 – 308.

Tayanc, M., Aytac, A. and Bayram, A. (2007). The effect of carbon on the fatigue strength of dual phase steels. *Mater. Des.* 28(6): 1827-1835.

ThyssenKrupp Steel Europe (2008). DP-W® and DP-K® dual phase steels for the manufacture of complex high-strength structural elements. Accessed from http:// www.thyssenkrupp-steel-europe.com/upload/.../Dual_phase_steels.pdf

Tsipouridis, P. (2006). Mechanical properties of dual phase steels. Doctoral Thesis, Technical University Munchen, Germany.

Türkmen, M. and Gündüz S. (2011). Martensite morphology and strain ageing behaviours in intercritically treated low carbon steel. *Ironmak. Steelmak.: Prod. Appl.* 38(5): 346-352.

WorldAutoSteel (2011). Future steel vehicle: overview report, pp 1-78. Accessed from http://www.autosteel.org.

Xu U., Yang W. and Sun Z. (2008). Mechanical properties of fine-grained dual phase low –carbon steels based on dynamic transformation, *Int. J. Miner. Metall. Mater.* 15(5): 556 –560.

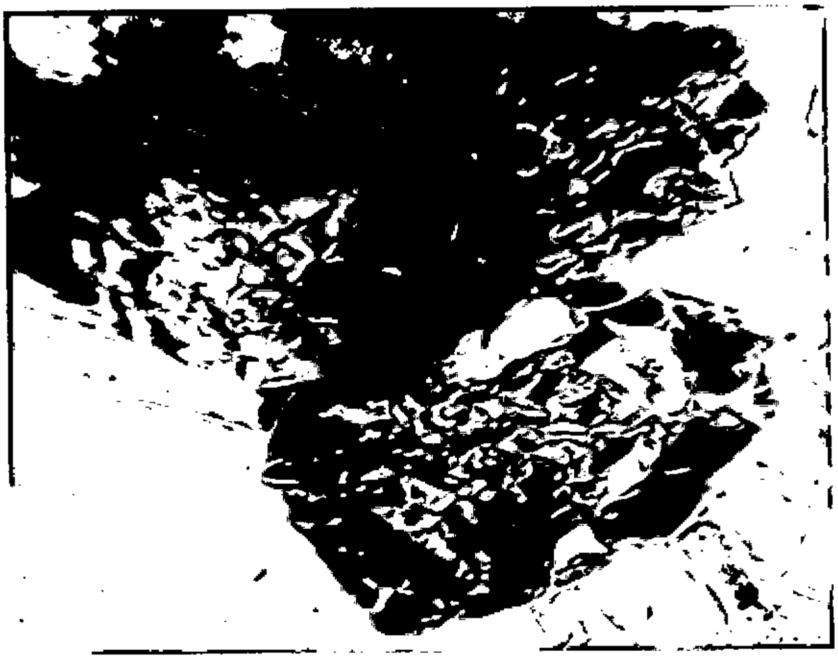


Fig. 1: Dark supercooled bitumen before melting

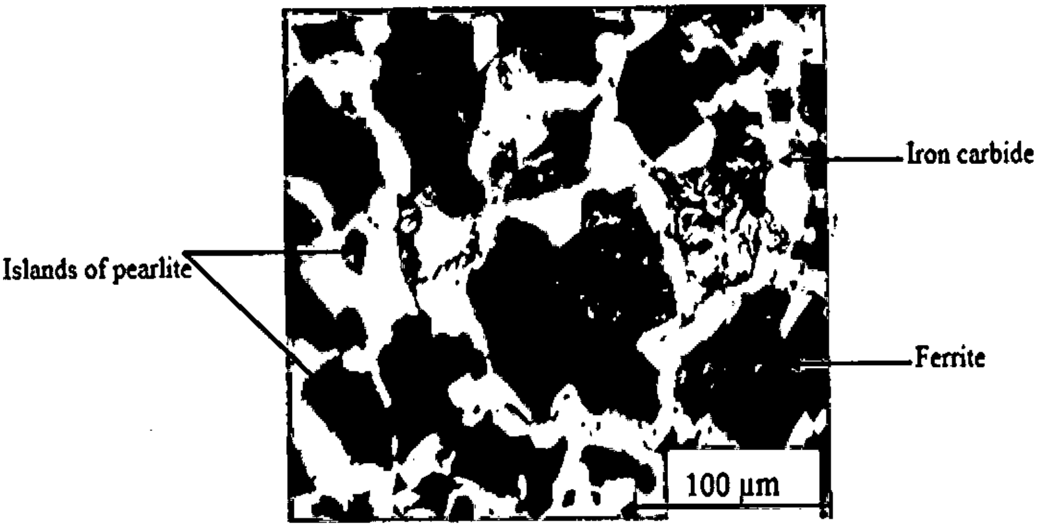
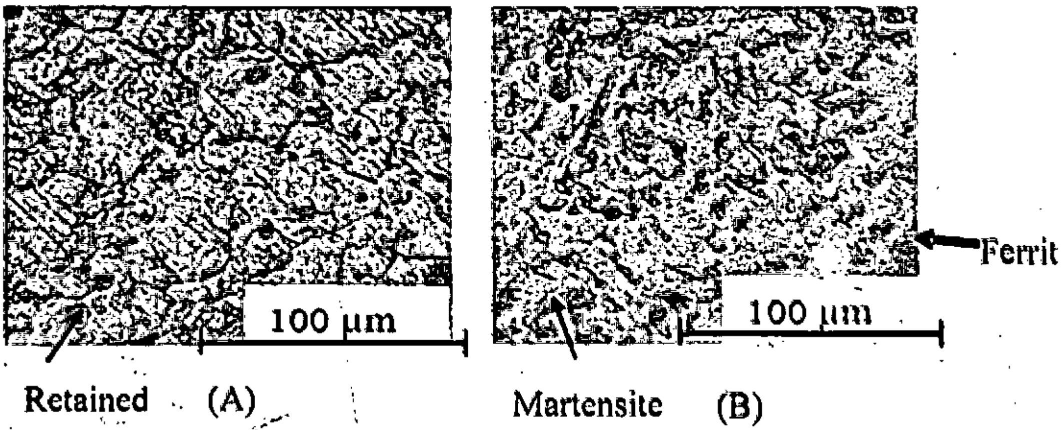
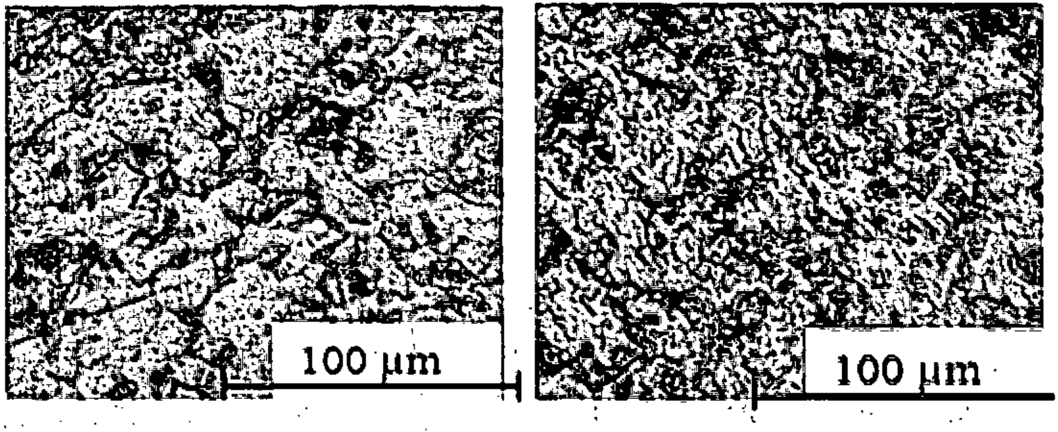


Fig. 2: Microstructure of normalised sample before intercritical treatment



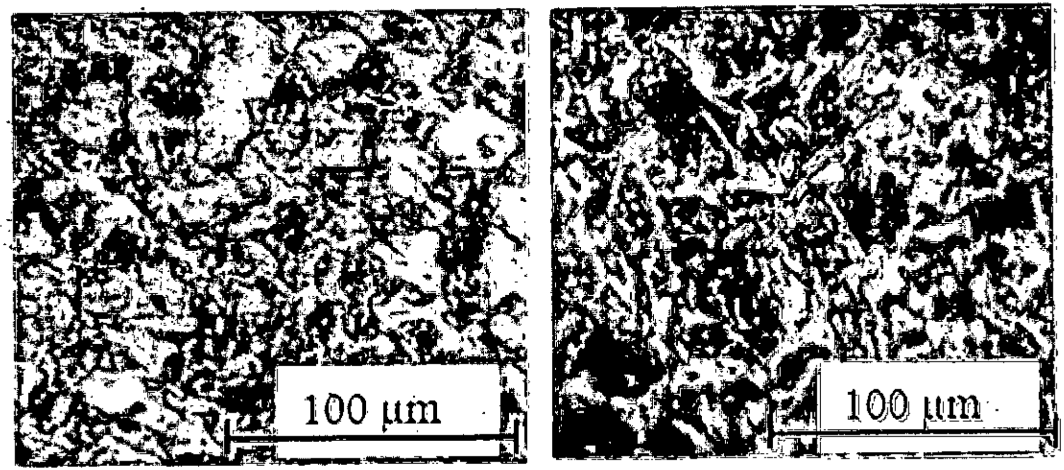
(A) (B)

Fig. 3: Microstructures of samples treated at 730°C at different holding times: (A) 30 and (B) 60 minutes



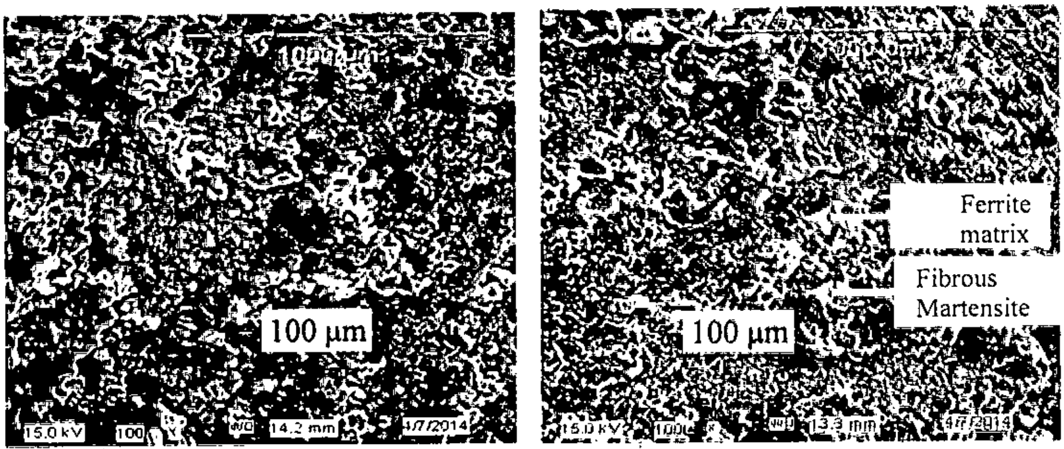
(A) (B)

Fig. 4: Microstructures of samples treated at 770°C at different holding times: (A) 30 and (B) 60 minutes

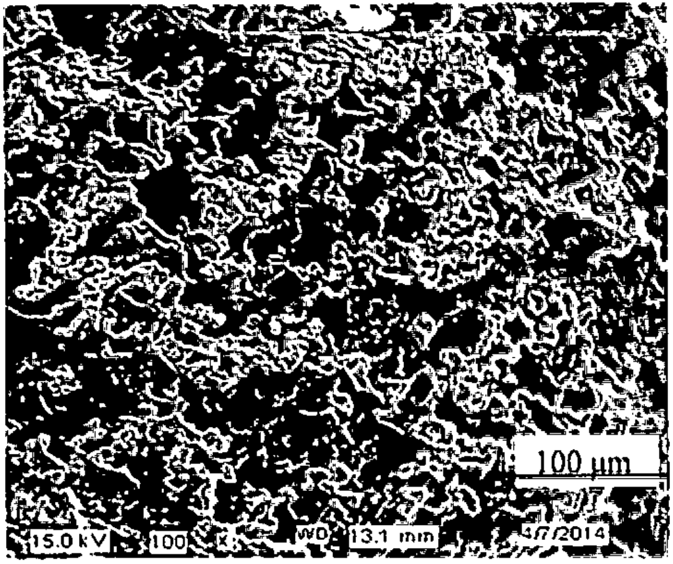


(A) (B)

Fig. 5: Microstructures of samples treated at 810°C at different holding times: (A) 30 and (B) 60 minutes



(A) (B)



(C)

Fig. 6: SEM morphology of samples intercritically treated at different temperatures at a holding time of 60 minutes: (A) 730, (B) 770 and (C) 810°C

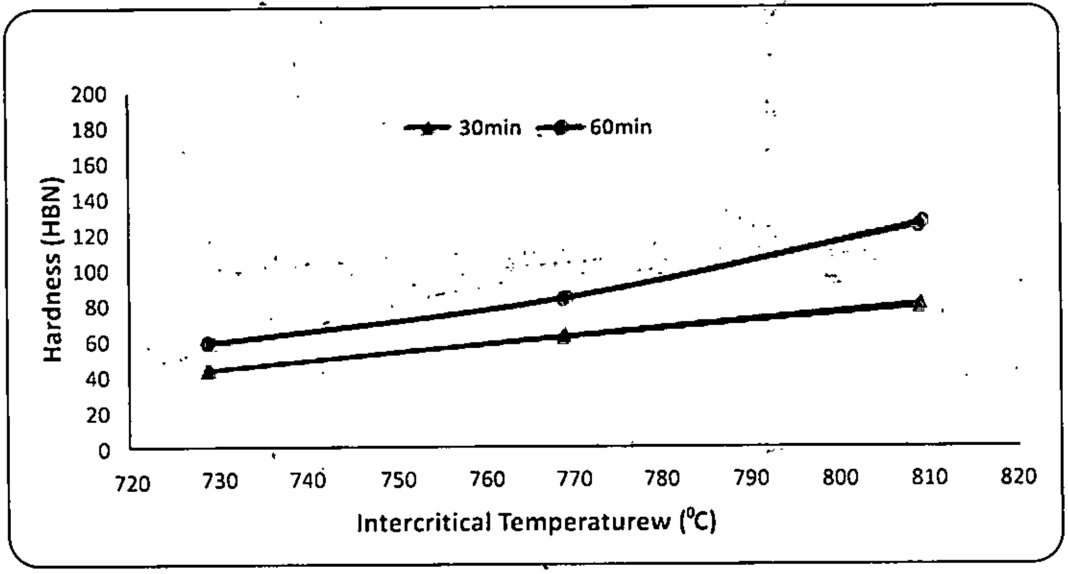


Fig. 7: Hardness variations with intercritical treatment conditions

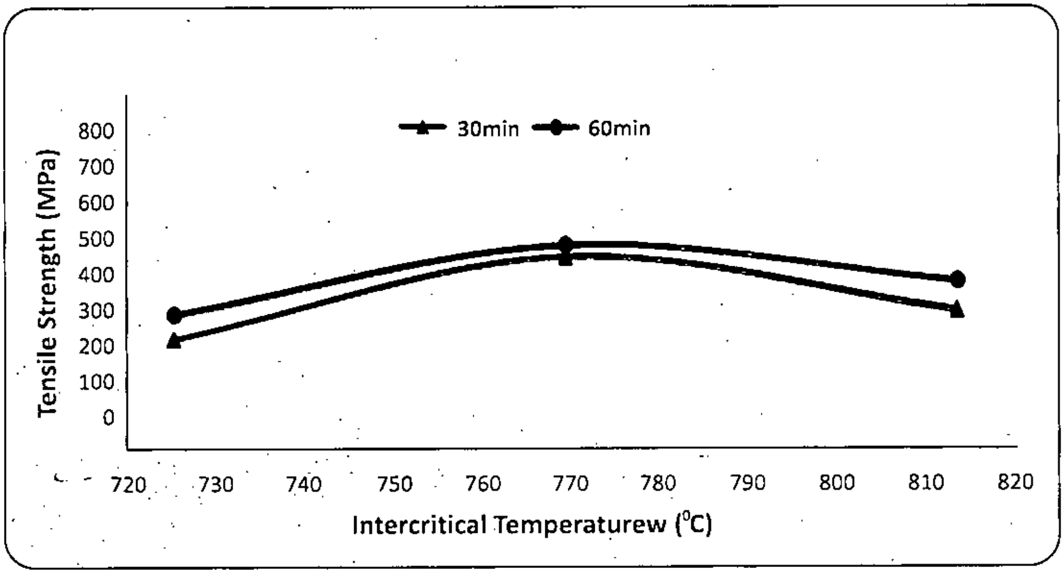


Fig. 8: Tensile strength of dual phase steel at different intercritical temperatures and holding times

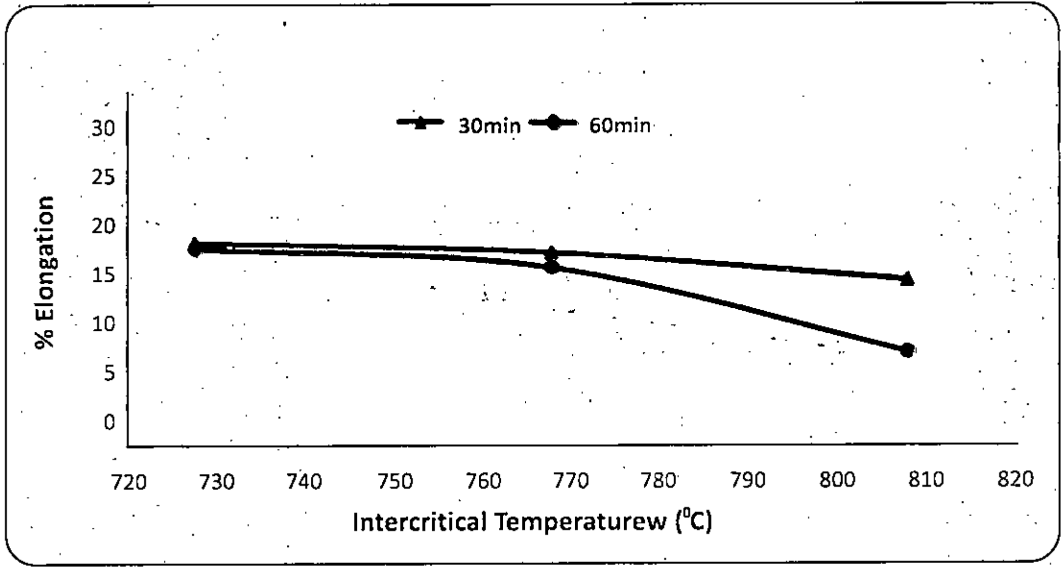


Fig. 9: Percent elongation of dual phase steel at different intercritical temperatures and holding times

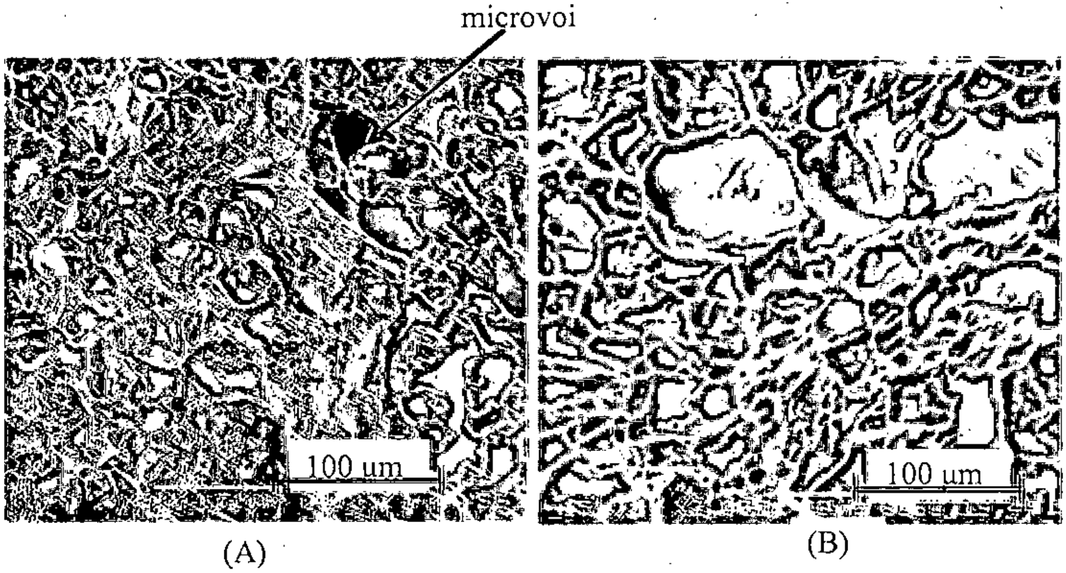


Fig. 10: Tensile surface fractographs in dual phase steel intercritically treated at: (A) 730°C and (B) 810°C at holding time of 60 minutes

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

Table 2: Mechanical properties of the normalised medium carbon steel

Sample Designation	**Tensile Strength (MPa)**	**Elongation (%)**	**Hardness (HBN)**
Normalised Sample	486.90	17.06	95