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Effect of tempering on the microstructure and mechanical properties of austenitic dual phase steel

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This research is focused on the effect of tempering on the microstructure and mechanical properties of 0.23%C austenitic dual phase steel locally manufactured in Nigeria. The as-received steel was normalized in order to annul the thermo-mechanical history of the steel. Thereafter, some of the normalized samples were austenetized at 850°C for 30 min and then slowly cooled in the furnace to the (α+ϒ) region and soaked for 30 min each at 790, 770, 750 and 730°C; and then quenched in hot water at 50°C. Again, some of the as-step quenched samples were tempered at 320°C for 1 h and air cooled. Mechanical testing and microstructures studies were conducted on all the heat treated samples. Optimum combination of properties was observed with samples intercritically step quenched at 770°C and tempered at 320°C for 1 h. Its hardness, impact strength, total elongation and ultimate tensile strength improved from 241.45 to 327.15 Hv, 0.26 to 1.66 J/mm^2, 9.14 to 36.03% and 785.68 to 1569.28 N/mm^2 respectively over the as-step quenched steel; representing 35.49, 538.46, 294.20 and 99.74% respectively. Microstructure photographs revealed duplex microstructure essentially comprised of ferrite and martensite with dispersion of carbide or retained austenite which is a typical characteristic of conventional dual phase steels.

Key words: Tempering, austenitic dual phase steel, mechanical properties, microstructures.

INTRODUCTION

The development of new materials with improved properties has remained the hallmark of the progress and breaks through made in technological advancement. Over the past three decades research in micro alloy steel has been directed towards the development of a new class of High Strength Low-Alloy (HSLA) steels known as Advanced High-Strength Steels (AHSS). This class of steel have been used in the automotive industry as a solution for weight reduction, safety performance improvement and cost saving. Among them, the dual phase steels (DPS), whose microstructure consists of mainly ferrite and martensite, are an excellent choice for applications where low yield strength, high tensile strength, continuous yielding and good uniform elongation are required. Their potential as superior strength and formability substitutes for current automotive

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Table 1. Chemical composition analysis result.

Element	C	Si	Mn	S	P	Cr	Ni	Cu	Fe
Weight (%)	0.23	0.20	0.73	0.03	0.03	0.12	0.10	0.27	98.30

steels was recognized and has provided an incentive for their rapid development and acceptance in this role (Sang et al., 2006; Bello et al., 2007; Zhao et al., 2009).

Recently, several researches have been conducted to determine the effect of temperature on the size and volume of the hard phase (martensite) and properties (Zhao et al., 2009; Offor et al., 2010; Daramola et al., 2010; Yazici et al., 2009; Mohammad and Ekarami, 2008). Among other notable researchers, Smallman (1995) studied and reported that the flow stress and tensile strength of these steels increase with a corresponding decrease in ductility with about 20% volume fraction of the martensite producing the optimum properties. However, Bag and the associates has shown that dual phase steels containing approximately equal amounts of finely dispersed ferrite and martensite phases (50 to 60%), exhibit the optimum combinations of high strength and ductility with impact toughness (Bag et al., 1999). Alaneme and the group also reported that better combination of fatigue and tensile properties were obtained at 760 and 780°C compared to 740°C. They attributed the improved mechanical properties to increased volume fraction of martensite (Alaneme et al., 2010). In the same line of reasoning, Majid observed that dual phase steels with equal amount of ferrite and martensite have excellent mechanical properties. He also reported that volume fraction of martensite increases with temperature (Majid, 2010).

In general, literature review has reported that the optimum intercritical annealing temperature lies between 760 and 790°C depending on the composition of the steel. All attested to the fact that dual phase steels possess superior properties when compared with normalized steels and quenched and tempered steels.

In spite of this observations and unique properties exhibited by dual phase steel, little has however been experimented on the steel's reaction(s) to tempering after development. This thus serves as impetus to this research as it focused on examining the effect of tempering on the microstructure and mechanical properties of austenitic dual phase steel. Austenitic dual phase steels are those dual phase steels with austenite as the starting microstructure during development or production (that is, the product of step quenching intercritical annealing process).

MATERIALS AND METHODS

The material for this investigation is a carbon steel, as-supplied in cylindrical form of 16 mm diameter. The chemical composition of the steel shown in Table 1 was performed using spectrometric analyzer (NCS Labspark 750B).

Heat treatment

All initially machined samples were normalized at 850°C (30°C above AC_3) and held for 60 min in a muffle furnace. A group of these normalized samples were labelled as “A” and used as control. The other group of normalized samples were subjected to step-quenching intercritical annealing treatment in order to produce the austenitic dual phase steel. This involved heating to austenitic temperature of 850°C and soaking for 1 h, followed by slow cooling in the furnace to the dual phase (α + γ) region, soaked for 30 min at different temperatures and then quenched in water. This is followed by subjecting some of the step-quenched samples to tempering operation at 320°C for 1 h and air cooled. All samples were designated as shown in Table 2.

Sample preparation and testing

After the heat treatment operations, all samples at their respective test configurations were separated and properly designated prior to tests and analysis. The impact test was conducted by means of a pendulum charpy impact tester. All the samples were tested to fracture at room temperature. The hardness of the samples was determined using a microhardness tester with inbuilt display unit. The tensile properties of the samples were determined using the Instron 3369 universal tester fitted with computer interface. The specimens were prepared to ASTM standard (ASTM E8M-91, 1992).

Small samples cut from the heat treated samples were metallographically prepared for microstructural examination following standard procedures. The fractured surfaces of some fractured impact samples were examined using the scanning electron microscope (SEM).

RESULTS AND DISCUSSION

Intercritical annealing temperature and the mechanical properties of intercritically annealed step quenched samples

With austenite as the starting microstructure for this intercritical annealing, Figure 1 reveals that the hardness of the as-step-quenched samples (series G) is rising and falling with samples annealed at 790°C having the highest hardness value while minimum value is observed at 770°C. The high hardness recorded at 790°C could be attributed to high volume martensite content as a result of more austenite which transforms to martensite on quenching from that temperature.

Figure 2 shows that there is a decrease in the UTS above 750°C after an initial increase. The initial rise in

Table 2. Identification code of samples.

Type of heat treatment	Temperature (°C)	Identification code
Normalizing	850	A
Step quenching	730	G730
	750	G750
	770	G770
	790	G790
Tempered step quenched samples	730	G730T
	750	G750T
	770	G770T

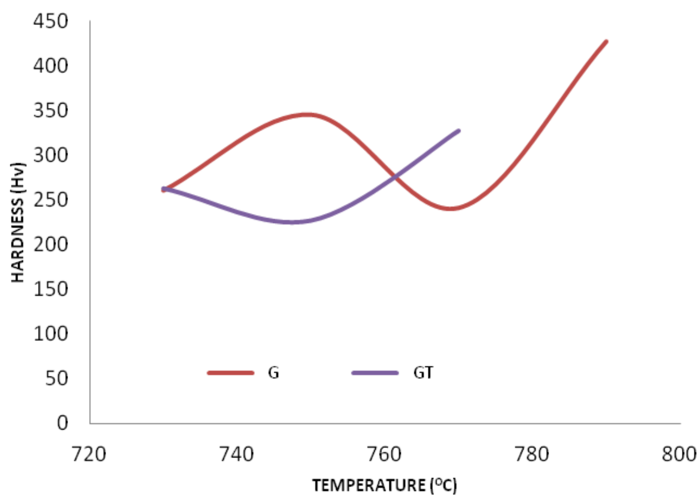

Figure 1. Hardness value versus temperature.

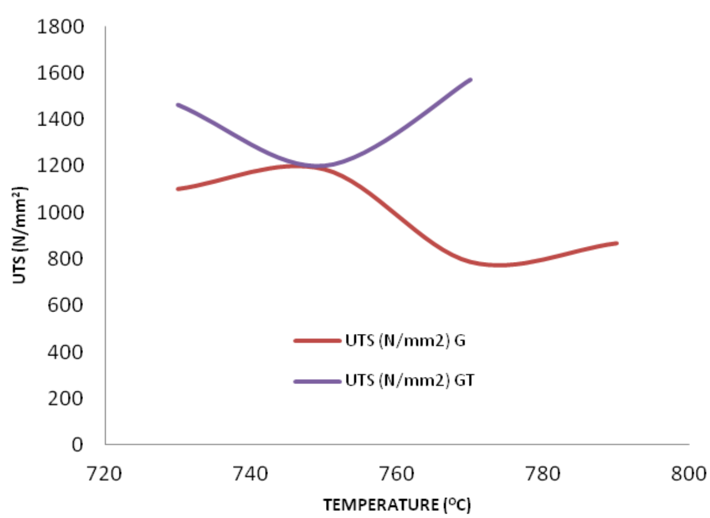

Figure 2. Ultimate tensile strength versus temperature.

UTS could be attributed to grain refinement after 730°C as evident in Figure 5 (a) and (c). The fall in UTS at higher temperatures could be attributed to very high martensite volume fraction, because at higher

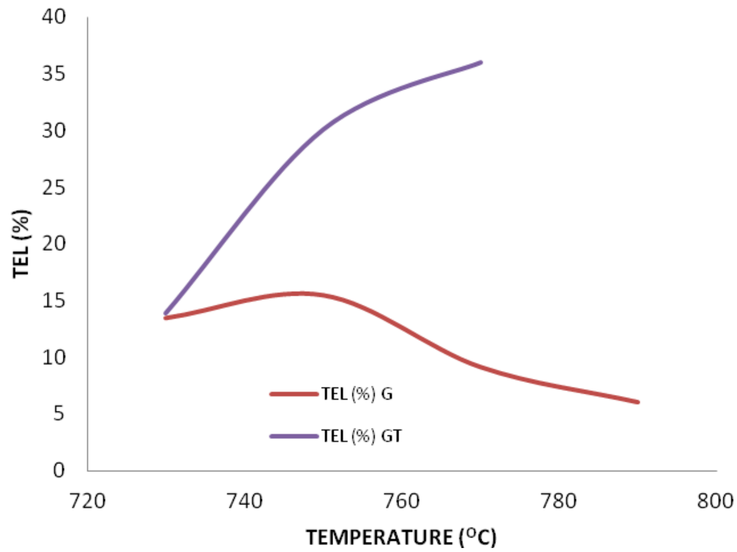

Figure 3. Total elongation versus temperature.

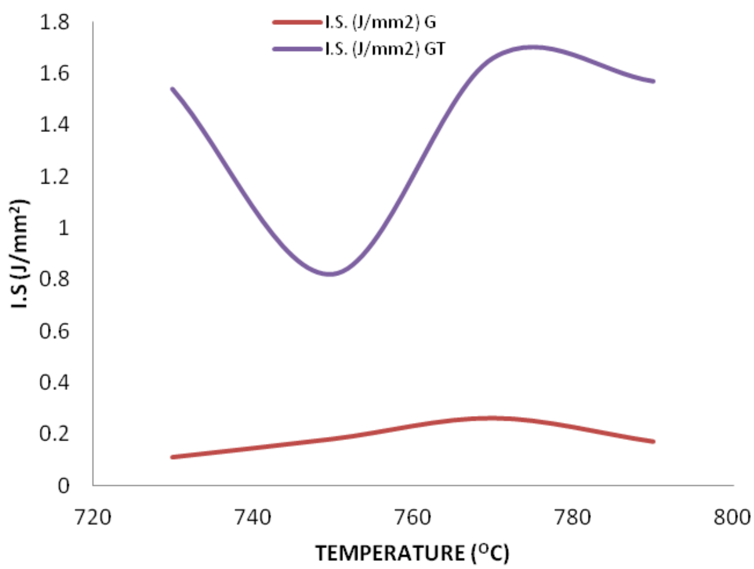

Figure 4. Impact strength versus temperature.

temperatures the amount of austenite is quite high which upon quenching transforms to martensite. Carbon content increases with increased temperature and this affects mechanical properties adversely. While at lower temperatures ferrite would have precipitated out from the prior austenite which most likely reduces the amount of martensite to or near the optimum amount for high strength. Also the grain refinement observed at 750°C creates obstacles to movement of dislocation which in turn requires more stress. Total elongation (TEL) also follows the same trend with UTS (Figure 3). It has also been reported that the carbon content of martensite decreases with increased volume fraction of martensite; this has a detrimental effect on ductility above 50%. It is pertinent to note that TEL at all temperature fell below the normalized sample. There is a steady increase in impact strength of sample given the same treatment with increased temperature as depicted in Figure 4 (series G). Though, all the impact strength values obtained are very poor compared to the normalized steel sample. Results shown in Table 3 proved that intercritical step quenching does not favour the improvement of ductility and impact strength at all the investigated temperatures. However, the optimum temperature for properties is at 750°C for

Table 3. Summary of the mechanical properties of the developed DPS.

Sample	UTS (MPa)	Y.S (MPa)	Y.S/U.T.S	TEL (%)	I.S (J/mm^2)	Hd (Hv)
A	670.55	385.96	0.575	21.97	1.30	161.7
G730	1099.6	351.87	0.32	13.43	0.11	260.75
G750	1184.64	-	-	15.43	0.18	345.1
G770	785.68	-	-	9.14	0.26	241.45
G790	866.1	-	-	6.06	0.17	426.5
G730T	1462.32	921.26	0.63	13.85	1.54	262.75
G750T	1202.68	817.82	0.68	30.1	0.82	226.85
G770T	1569.28	816.03	0.52	36.03	1.66	327.15

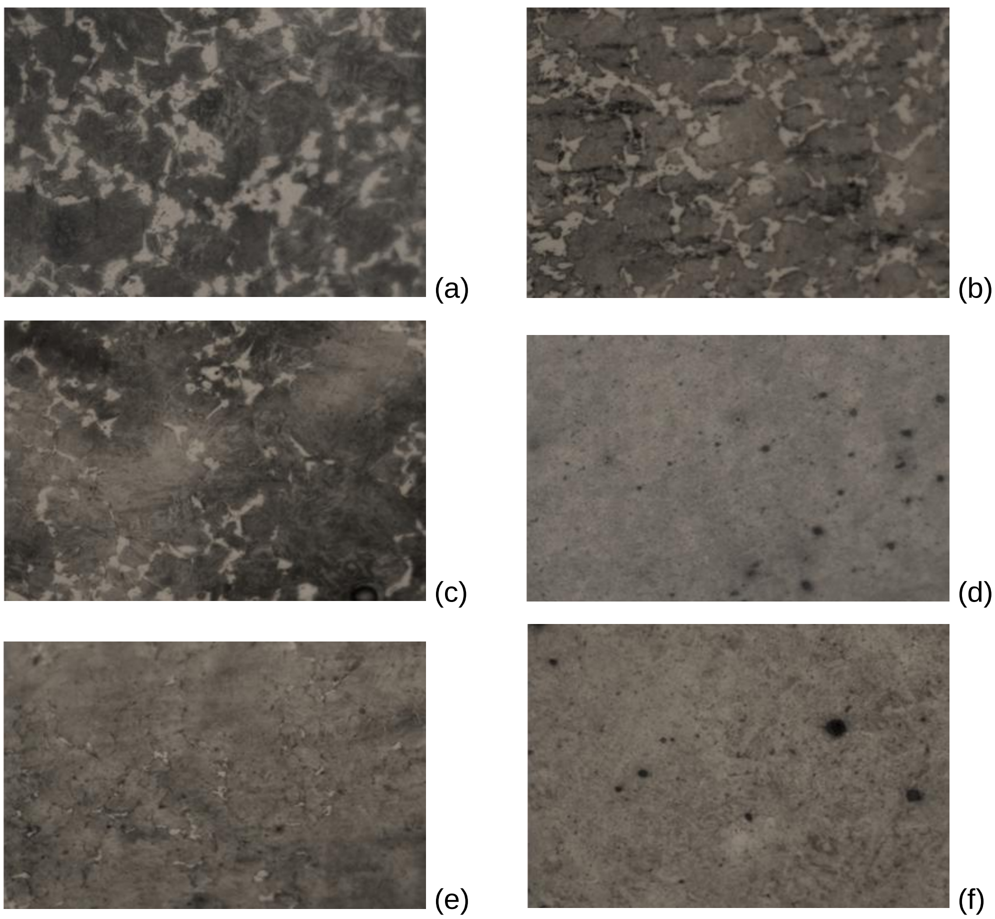

Figure 5. (a) G730 X200. Sample step quenched at 730°C for 30 min. (b) G730T X200. Sample step quenched at 730°C for 30 min and tempered for 1 h at 320°C. (c) G750 X200. Sample step quenched at 750°C for 30 min. (d) G750T X200. Sample step quenched at 750°C for 30 minand tempered for 1 hour at 320°C. (e) G770 X200. Sample step quenched at 770°C for 30 min. (f) G770T X200. Sample step quenched at 770°C for 30 min and tempered for 1 hour at 320°C.

the step quenching intercritical annealing heat treatment. The performance of UTS, hardness, impact strength and total elongation relative to the normalized sample at 750°C are 76.67, 113.42, -86.15 and -29.77% respectively. The negative signs indicate deterioration of property.

Microstructure evolution with temperature for samples intercritically step quenched

Figure 5(a), (c) and (e) are the photomicrographs of the samples that were subjected to “Step Quench” operation with austenite as the initial microstructure. The rather

Table 4. comparison of properties' improvement of sample intercritically step quenched at 770°C and tempered at 320°C for 1 h (G770T) with some selected samples (A, G750 and G770).

Property	Sample				% Improvement of G770T Over		
	A	G750	G770	G770T	A	G770	G750
Hardness (Hv)	161.7	345.1	241.45	327.15	102.32	35.49	-5.20
I.S. (J/mm^2)	1.30	0.18	0.26	1.66	27.69	538.46	822.22
TEL (%)	21.97	15.43	9.14	36.03	64.0	294.20	133.51
UTS (N/mm^2)	670.55	1184.64	785.68	1569.28	134.03	99.74	32.47

slow (furnace) cooling of austenite from 850°C to intercritical annealing temperatures of 790, 770, 750 and 730°C, took 7, 10, 14, and 19 min respectively, resulting in the formation of pro-eutectoid ferrite (light areas) and austenite, which transformed to large martensite laths (grey areas) on quenching from the intercritical temperature region (Figure 5(a) and (c)) respectively. Smallman stated that the morphology of this ferrite depends on the usual precipitation variables such as temperature, time, carbon content and grain size, and that growth occurs preferentially at grain boundaries and on certain crystallization Figures (Smallman, 1995). Honeycombe and partner further postulated that grain boundary allotriomorphs are the first morphology of ferrite to appear over the whole range of composition and temperature (Honeycombe and Bhadeshia, 1995). These they claim to predominate above 800°C, and grow along the grain boundaries and also into the austenite grains to give a well-defined grain structure. These features are clearly revealed in Figure 5(b). Precipitation of carbide particles is also noticed along the grain boundaries as temperature is increased, (Figure 5(b) and (c)). These carbide particles pin down the ferrite grains at locations where they are precipitated and therefore hinder the rather regular growth of the ferrite along the grain boundaries. The result of this, as seen in Figure 5(c) and (e), is that the ferrite grains are of irregular shapes mixed with equally irregularly curved globular martensite constituting a continuous network along prior austenite grain boundaries.

Figure 5(a) reveals irregularly shaped recrystallized ferrite (bright) and lath martensite (gray) precipitated along prior austenite grain boundaries in a net work of ferrite (light) with little retained austenite (dark). Figure 5(b) also reveals nucleated ferrite (bright) precipitated along prior austenite grain boundaries, tempered martensite (gray) in a ferrite (light) matrix, with dispersion of fine carbide (dark) precipitated from tempered martensite and retained austenite. More irregularly shaped ferrite (bright) and lath martensite (gray) precipitated along prior austenite grain boundaries in a network of ferrite (light) was observed in Figure 5(c) with very little retained austenite (dark). In Figure 5(d), fine grained tempered martensite (gray) in a network of ferrite (light) with dispersion of fine carbide (dark) precipitated from tempered martensite and retained austenite was observed. Figure 5(e) reveals lath martensite (gray) and recrystalized ferrite (bright) along prior austenite grain boundaries in a net work of ferrite (light) with some retained austenite (dark). Also, in Figure 5(f) fine grained tempered martensite (gray) in a net work of ferrite (light) matrix with dispersion of carbide (dark) precipitated from tempered martensite and retained austenite was observed.

Effect of tempering on the properties of step quenched samples

All the accessed properties improved upon tempering (Figures 1 to 4 and Table 4). High strength and ductility observed with the tempered samples could be attributed to factors such as: grain refinement on tempering (Figure 5(c), (d) and (e), (f)) and recrystallization of ferrite from martensite on tempering at 320°C, because at higher intercritical annealing temperatures of the step quench process there are numerous prior austenite grain boundaries present which favour nucleation and recrystallization of ferrite on tempering than at lower temperatures. This enhances the synergy between plasticity and elasticity of martensite and ferrite respectively during deformation.

For hardness, after tempering for 1 h at 320°C, a minimum value was reached at 750°C (Figure 2). The decrease in hardness could be attributed to softening effect of the hard martensite and recrystallization of more ferrite on tempering. While the increased hardness of the G770T over G770 could be as a result of refinement of the coarse grains on tempering (Figure 5(e) and (f)). Table 4 shows that all the properties have positive improvement after tempering. Hardness value improved by 102.32% over the normalized sample while it improved by 35.49% over the intercritically step quenched sample. The high level of improvement observed could be attributed to the redistribution of carbon on tempering. Grain refinement on tempering the sample step quenched at 770°C could also account for the improvement of properties observed (Figure 5(f)). The high improvement observed for impact strength and ductility (TEL) will make this particular sample, G770T

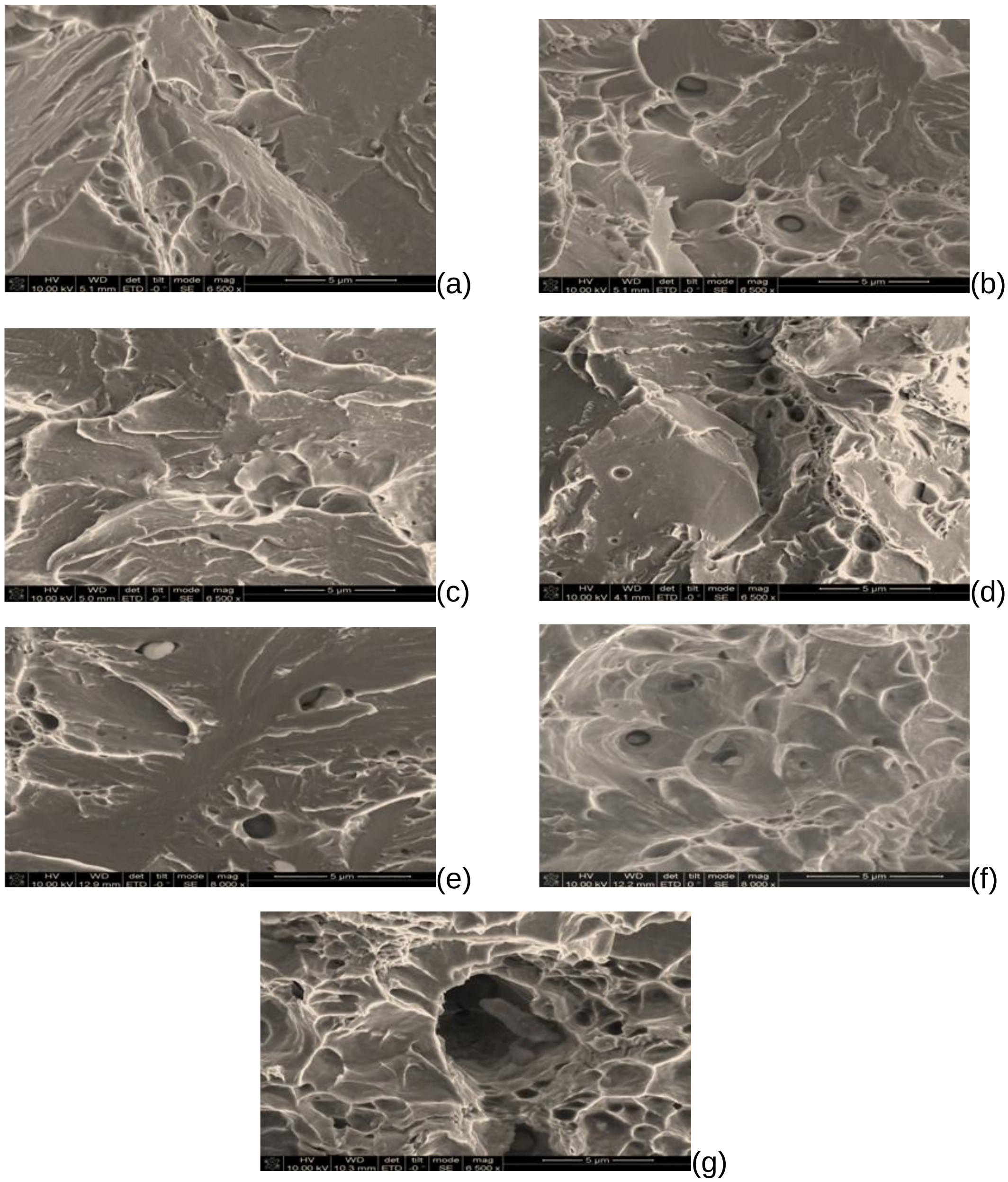

Figure 6. (a) SEM of G730: cleavage ridge-like surface is revealed. (b) SEM of G730T: fractured surface is blocky with dendrites crack lines at the crack tips. (The presence of precipitated carbides hinders the movement of dislocations). (c) SEM of G770: Structure reveals large blocky pure cleavage fracture with crack propagation along the grain boundaries. (d) SEM of G770T: structure reveals mixture of fibrous and cleavage fracture surface with more of the fibrous features at the centre. (e) SEM of G750T: structure reveals mixture of cleavage and fibrous surface. (f) SEM of G750: structure reveals almost pure cleavage fractured surface. (g) SEM of A: structure reveals mixture of fibrous and cleavage surfaces.

unique in terms of formability or workability and ability to withstand both high static and sudden loading.

Effects of process parameters on the fractography

Figure 6 shows the scanning electron micrographs of the fractured surfaces of some developed dual phase samples after subjecting it to impact test. Figure 6(a) and (b) revealed pure cleavage fracture for G730 and G770 respectively, while Figure 6(c) and (d) revealed a blocky fractured surface with dendrites crack lines at the crack tips and the presence of precipitated carbides for G730T. A mixture of fibrous and cleavage fracture surface with more of the fibrous at the centre for G770T was observed in Figure 6(e). All of this could account for the tremendous improvement in impact strength observed after tempering of the steels. This phenomenon could be attributed to the dispersion of hard martensite in soft and ductile ferrite matrix naturally exhibited by conventional dual phase steel. Figure 6(g) shows the fractured surface of the normalized sample with mixture of fibrous and cleavage surfaces. A predominantly cup and cone structure was exhibited by the normalized sample thus indicating its failure to be ductile. This justifies the relatively increased strain-to-fracture (TEL) observed (as earlier discussed).

Generally, sample G770T was found to develop a unique higher mechanical properties (impact, hardness, tensile strength) with a surprising improvement in the ductility. These exhibited properties make it suitable for structural applications.

Conclusion

From the foregoing, it can be concluded that tempering intercritically step quenched 0.234wt%C steel at 320°C improved all the investigated properties, both on the normalized and as-step quenched steels. The tremendous improvement recorded for UTS, TEL and IS will make the tempered steel put up outstanding performance in the automotive industry where high strength, light weight and ability to maintain high crash safety are required. Also, a combination of high hardness, high UTS and good toughness will promote its application in the oil and gas sector for the construction of pipelines used for conveying high pressured petroleum products and crude. It will also perform maximally in structural designs such as sky scrapers, bridges, towers etc. Hence, these steels are strongly recommended for use in the above mentioned industries.

Conflict of Interest

The authors have not declared any conflict of interest.

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