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Effect of Intermediate Quenching and Tempering on the Mechanical Behaviour of Low Carbon Steel

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Abstract— *This research has assessed the impact of intercritical annealing using the intermediate quenching technique and tempering on the mechanical properties of low carbon steel. The procedure involved austenitizing at 850^{O}C for 1hour followed by quenching in water and thereafter annealed at 730^{O}C, 750^{O}C and 770^{O}C (i.e. $\alpha + \gamma$ region) for 30 minutes and then quenched in water again. Some of the as-quenched samples were tempered at 320^{O}C for 1hour and cooled in still air. Tensile, hardness and impact tests as well as microstructure characterization were conducted for samples from all the heat treatment schedules. It was observed from the results that martensite volume fraction increases almost linearly as a function of temperature. Ductility and impact decreased with increase in temperature. Tempering deteriorated the assessed mechanical properties. Hence, for a steel of this composition, intermediate quenching should not be followed by tempering.*

Keywords— intermediate quenching, intercritical annealing, mechanical properties, martensite volume fraction, dual phase steel.

I. INTRODUCTION

The microstructure of steels can be altered by changing processing parameters, which ultimately affects their mechanical properties. These process parameters which can be altered include the base steel composition, mode of manufacture, type of heat treatment and parameters of heat treatment such as temperature, soaking time, heating and cooling rates, cooling media etc.

Intermediate quenching is one of the various types of heat treatment for developing dual phase (DP) steels. Over the recent years, DP steels have been widely used in the automotive industries because of good compromise between its high strength and reasonable ductility which enhances performance and crash safety as well as high fuel economy due to weight reduction as a result of the improve strength. This weight reduction also impacts positively on the environment because of drastic reduction in emissions to the environment. Apart from the automotive industry, DP steels have found applications in oil and gas industries, building and structural industries, earth moving equipment (yellow goods) etc. Furthermore, very tall structures in the form of skyscrapers are becoming common these days due high demand on land. Hence, the urgent need to provide common materials with ultra-high strength at reasonable cost cannot be over emphasized. This will help to militate against the frequent occurrence of collapsed buildings in the country. Again, the high demand for large diameter and high strength pipes for the conveyance of crude oil and petroleum productions requires materials with excellent formability, high strength and good weldability.

Hence, this research is intended to investigate the influence of intermediate quenching and tempering on the mechanical behaviour of low carbon steel. Intermediate quenching uses martensite microstructure as the starting or initial microstructure for the intercritical annealing heat treatment process. Bag*etal* have worked on intermediate quenching, step quenching and direct quenching using high strength low alloy (HSLA) steel. Ikpeseni *etal* had worked on step quenching and direct quenching using low carbon steel [17, 18]. A good number of researchers have worked on the effect of processing parameters on the properties of dual phase steels with encouraging results [1 – 20]. [1 – 3] examined the effect of cooling rates; the effect of alloying element on mechanical properties was investigated by [4 – 12]; while [13, 16,17] worked on the effect of the temperature. Furthermore, [14, 15, 17] examined strain or deformation effect while [15, 18 - 21] examined the impact of microstructure on mechanical properties of the investigated steels.

II. MATERIALS AND METHOD

2.1 Materials

The carbon steel used for this research was supplied by universal steel Lagos, Nigeria. Its chemical composition shown in Table 1was determined as documented in [17].

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Table.1: Chemical Composition of the Investigated Steel

Element	C	Si	Mn	S	P	Cr	Ni	Cu	B	Ti	Fe
Weight (%)	0.23	0.20	0.73	0.03	0.03	0.12	0.10	0.27	0.001	0.001	98.28

2.2 Methods

2.2.1 Sample preparations

Standard samples for tensile test, impact test, hardness test and microscopic examination were prepared from the as-received 16mm diameter rod. All the samples were prepared following standard procedures.

2.2.2 Heat Treatments

All the samples were first of all normalized in a muffle furnace at 850^0C for 1hr in order to cancel the effects of previous mechanical, thermal or thermo-mechanical treatments. After normalizing some of the samples were left as control, while others were subjected to the intermediate quenching (an intercritical annealing) heat treatment. This involved austenitizing at 850^0C for 1hr and quenching in water to produce martensite which was used as the starting or initial microstructure for the intercritical annealing. Thereafter, all the samples were intercritically annealed at various temperatures of 730^0C, 750^0C and 770^0C (i.e. in $\alpha + \gamma$ region) for thirty minutes each, followed by quenching in water. Then some set of these sample were tempered at 320^0C for 1hr, while the others are left in their intermediate quenched state.

2.2.3 Mechanical Properties Testing

Tensile test: an instron Universal tensile testing machine was used to conduct the tensile test. The sample were tested at a cross head speed of 20mm/min and were all tested to fracture at room temperature (25 -27^0C). All the tensile properties data were captured by the interfacing computer system.

Impact test: The charpy impact tester (Avery) was used to determine the absorbed energy and thereafter the impact strength (toughness) of the heat treated samples were evaluated. Again, all the samples were tested to fracture at room temperature. Thereafter the fractured surfaces were examined under the scanning electron microscope (SEM) in order to ascertain the mode of fracture.

Hardness test: The hardness property of samples from all the heat treatment schedules were examined using the Vickers hardness testing machine (LM 7 700AT Leco) with a dwell time of 10 – 15S. The hardness values are digitally displayed on the machine screen.

2.2.4 Microstructure Characterization

Nikon Eclipse (me 600) was used to examine the microstructures developed after the various heat treatment schedules. This was preceded by sample preparation using standard procedures. A combination of sylvert cloth and 0.2µ diamond paste was used to polish the samples while 2% NITAL was used as etchant. The standard grid point count technique was used to determine martensite volume fracture (MVF) as contained in Russ and Dehoff (1999) [22].

III. RESULTS AND DISCUSSION

3.1 Microstructural Evolution of Intermediately Quenched Samples

Figures 1a - g show the microstructures developed after intermediate quenching and subsequent tempering. The structures revealed predominantly features of ferrite (light) and martensite (grey) with dispersion of carbide or retained austenite (dark) in some of the microstructures.

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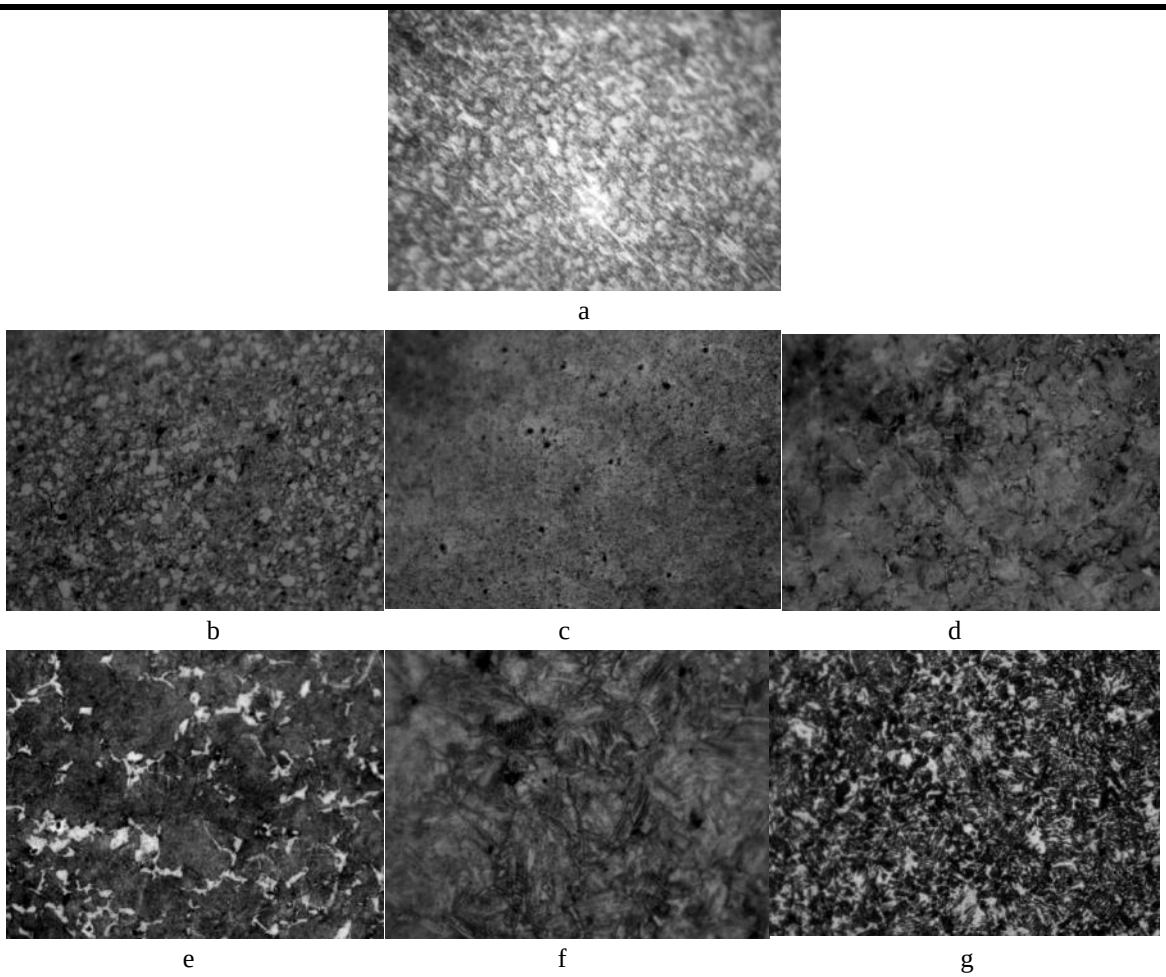

a b c d e f g

Fig.1: (a) Photomicrograph of A X400 i.e. Phase structure produced by normalizing at 850ºC for one hour. *The structure reveals ferrite (light phase) and pearlite (alternate layers of ferrite - light and cementite – dark).* ***(b) Photomicrograph of IQ730 X200. Sample intermediately quenched at 730^{O}C for 30 minutes.*** *The structure reveals distribution of regularly shaped globular ferrite (light) and martensite (grey) with little dispersed carbide (dark).*
(c) Photomicrograph of IQ730T X200. Sample intermediately quenched at 730^{O}C for 30 minutes and tempered at 320^{O}C for 1hour. *The structure reveals distribution of ferrite (light) matrix, tempered martensite (grey) and dispersed fine carbide (dark).* ***(d) Photomicrograph of IQ750 X200. Sample intermediately quenched at 750^{O}C for 30 minutes.*** *The structure reveals distribution of fine fibrous martensite (grey) in a ferrite (light) matrix.* ***(e) Photomicrograph of IQ750T X200. Sample intermediately quenched at 750^{O}C for 30 minutes and tempered at 320^{O}C for 1hour.*** *The structure reveals distribution of tempered martensite (gray) and nucleated ferrite (bright) mainly along grain boundaries in a network of ferrite (light) matrix.* ***(f) Photomicrograph of IQ770 X500. Sample intermediately quenched at 770^{O}C for 30 minutes.*** *The structure reveals lath martensite (grey), ferrite (light) and little dispersed carbide (dark).* ***(g) Photomicrograph of IQ770T X200. Sample intermediately quenched at 770^{O}C for 30 minutes and tempered at 320^{O}C for 1hour.*** *The structure reveals network of tempered martensite (gray), ferrite (light) and dispersion of plenty carbide (dark).*

Figures 1(b, d and f) are the microstructure photographs of the as-quenched samples subjected to intermediate quench treatment.

Martensite (probably with very small percentage of retained austenite) formed as a product of quenching from the austenitizing temperature (850ºC) was used as the initial microstructure for this treatment. Intercritical annealing at the various temperatures gradually transforms the formed martensite to austenite and ferrite. Austenite formation from martensite structure was observed to occur by classical heterogeneous nucleation at imperfect lattice sites like spheroids in ferrite, matrix /carbide interface, martensite lath boundaries, and prior austenite grain boundaries [20, 22]. The photomicrographs display tiny but numerous

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globular/fibrous martensites after soaking for 30 minutes, with some carbide particles precipitated along prior austenite boundary, in a ferrite matrix in the microstructure (see Fig. 1b, d). Martensite volume fraction is noticed to increase as a function of intercritical annealing temperature. This is so because austenite nucleation at different sites mentioned above continued and increases at higher temperatures. Hence the microstructures became greatly enriched with more martensite on quenching. It was clearly shown (qualitatively) that martensite grain size remained fairly the same. Pinning down of the grain boundaries by precipitated carbide particles in prior boundaries of austenite must have been responsible for this. Honeycombe and Bhadeshia[23] stated that these are usually present in grain boundaries; as such an interaction occurs between the grain boundary and the particles. They explained that when there is replacement of a short length grain boundaries by particles, the interfacial energy help to maintain a stable configuration such that any attempt for the grain boundary to move away or separate from the particles, there is an increase in local energy; as a result the particle exerts a drag on the boundary.

3.1.1 Effect of Intercritical Annealing Temperature and Starting Microstructure on the Volume Fraction of Martensite (MVF)

Martensite was used as starting microstructure for the intermediate quenching intercritical annealing heat treatment to produce the dual phase (ferrite-martensite) structures. The intercritical annealing heat treatment was conducted at various temperatures in order to produce dual phase microstructures with varied proportions of martensite in a ferrite matrix. The effect of intercritical annealing temperature on MVF is presented in Fig. 2. As indicated, it is clear that MVF increases almost linearly with intercritical annealing temperature. This is in agreement with the observation of earlier published research findings [19, 20,24] As intercritical annealing temperature increases, the amount of austenite increased which transformed to martensite upon quenching. The simulated (fitted) linear curve for the treatment schedule is shown in equation (1) as obtained from Fig. 2.

$V_M = 0.325T - 203.4$ (1)

$R^2 = 0.953$

where T = intercritical annealing temperature

V_M = martensite volume fraction

The high value of R^2 indicates a high correlation between the simulated and experimental data, which also indicates the validity and reliability of equation (1).

The high martensite volume fraction of intermediate quenched samples could be attributed to numerous sites for nucleation of austenite which transforms to martensite upon quenching. Some of these sites as suggested by Bag et al [20] include:

a. Prior grain boundaries of austenite
b. Carbide particles precipitated on prior grain boundaries of austenite
c. Spheroids in ferrite
d. Fine carbide arrays formed on the prior martensitic plate/lath boundaries.

The shape, size and distribution of martensite in the microstructures of the samples given the intermediate quenching treatment stems from the process of reversion of austenite from the tempered martensite. Austenite nucleation from the prior martensite could take place at various locations or sites as listed from a – d above.

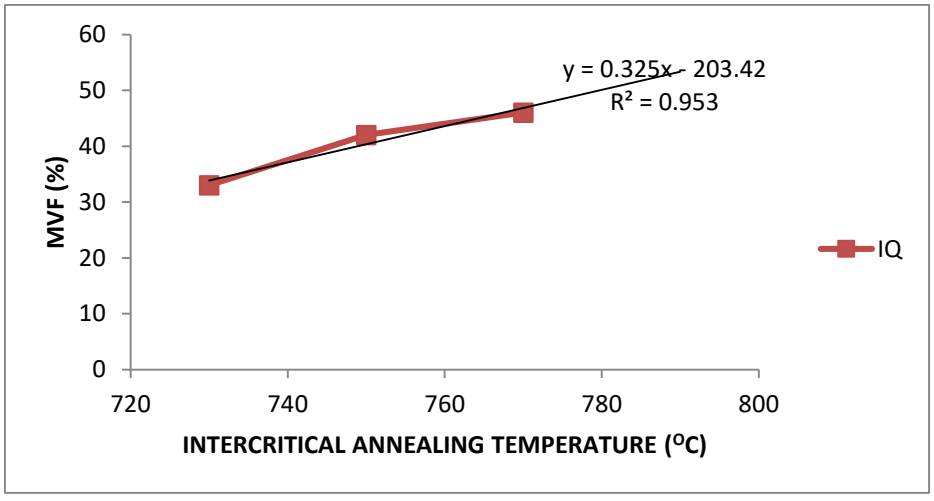

Fig.2: Martensite Volume Fraction vs Temperature

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3.1.2 Effect of Tempering on the Microstructure of Intermediate Quenched Samples

On tempering, the as-quenched intermediate quenched samples at 320^{O}C for 1hr, the microstructure consisted of fine grained tempered martensite and ferrite for IQ730T, with dispersion of fine carbide – Fig. 1c, while for IQ750T and IQ770T, the ferrite phase became well defined and coarse with plenty of carbide especially IQ770T as shown in Fig. 1e and g.

3.2. Effect of Process Parameters on the Mechanical Properties

Figures 3 – 6 present the results of the mechanical properties which show the relationships between the mechanical properties and intercritical annealing temperatures.

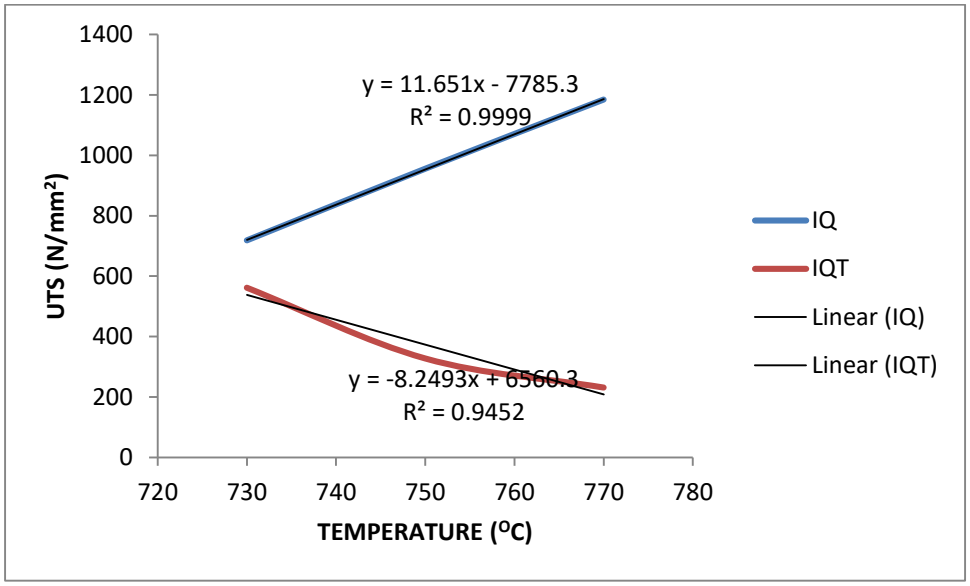

Fig. 3: Ultimate Tensile Strength vs Temperature

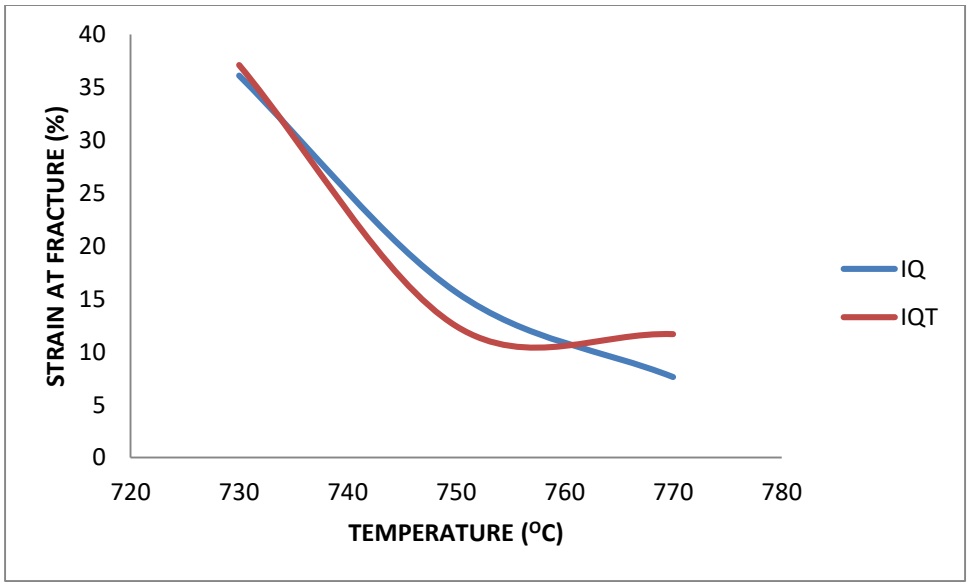

Fig. 4: Strain at Fracture vs Temperature

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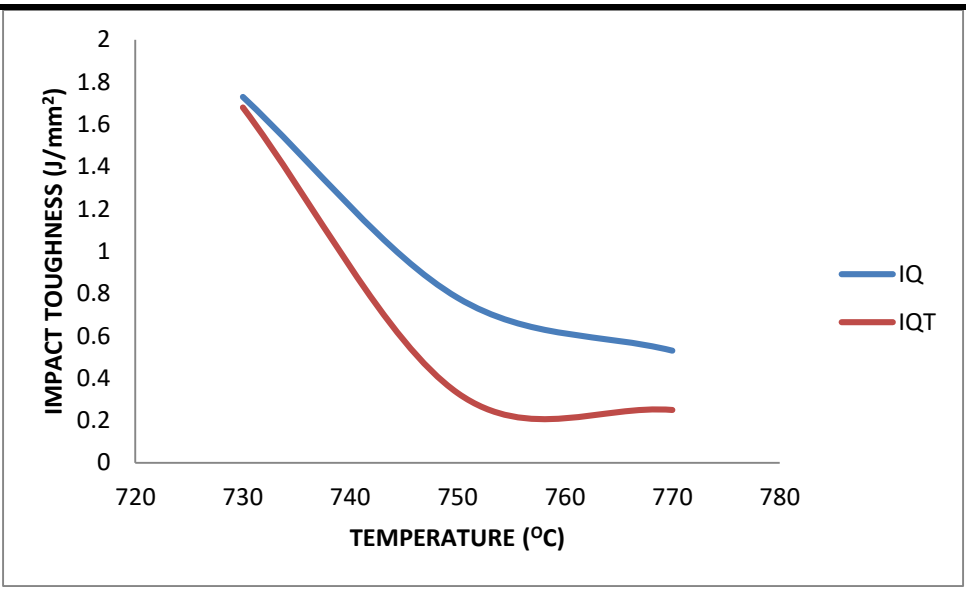

Fig. 5: Impact Toughness vs Temperature

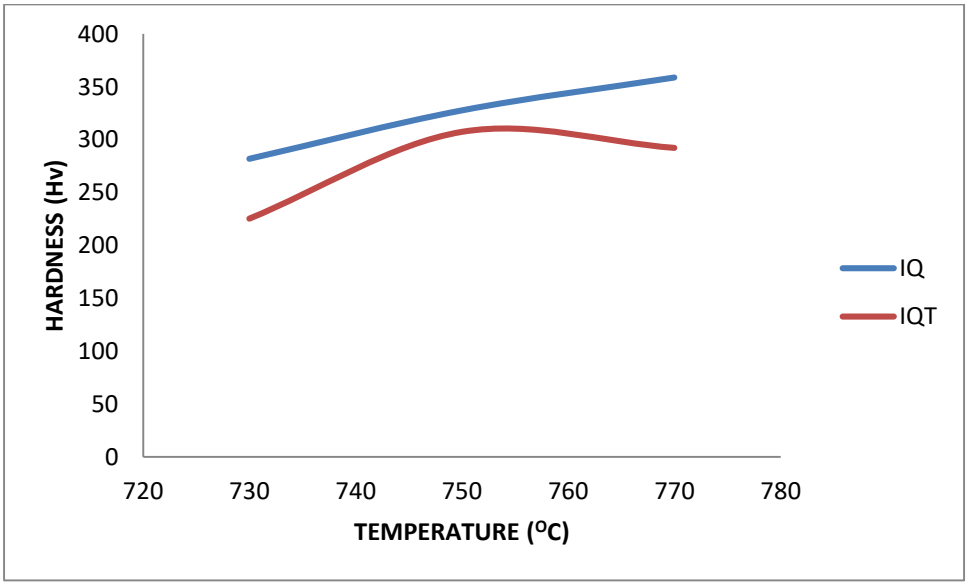

Fig. 6: Hardness vs Temperature

3.2.1 Effect of Annealing Temperature on the Mechanical Properties of Intermediate Quenched Samples

Variation of hardness value temperature for all the samples is presented in Fig. 6. The hardness of intermediate quenched samples increases steadily with rise in temperature within the investigation limit. The rise in hardness value is attributed to the increasing martensite volume fraction as intercritical annealing temperature increases (Fig. 1).

The ultimate tensile strength of samples given the intermediate quenching intercritical heat treatment, increased steadily with increasing temperatureas shown in Fig. 3 (IQ series). The decrease in strain at fracture (i.e. ductility or total elongation) and impact strength could be traced to nucleation, growth and recrystallization of ferrite and austenite from the initial martensite structure, which upon quenching the nucleated austenite transforms to martensite. Consequently, strain at fracture and impact toughness of samples given this same treatment decreased as temperature increased (Fig. 4 and 5). Continuous yielding was observed for all the samples given this particular treatment, which is a common characteristic of regular dual phase steels. Austenite to martensite transformation involves volume expansion which introduces

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residual stress on the surrounding ferrite as a result of the strain produced during the transformation [25,26, 27]. Davis (1979) [28] and Rigsbee*et al* (1979) [29] explained that this change in volume causes neighbouring ferrite grains to be plastically deformed, thus generates high density of mobile dislocations in the surrounding ferrite. This ultimately results to the generation of mobile dislocations. This could have increased the hardness and ultimate tensile strength, while ductility was decreased as a function of temperature. The fall in impact strength and ductility could also be attributed to decreased carbon content of the martensite at higher martensite volume fraction, because MVF increases with increase in temperature and at higher MVF carbon content of martensite decreases.

Optimum mechanical properties for intermediate quenched samples are observed at 730^{O}C (i.e. 33% martensite volume fraction). A comparison of improvement of properties showed that hardness, ultimate tensile strength, total elongation and impact strength are 74.4%, 7.2%, 64.7% and 33.1% respectively over the normalized samples. Summary of all the assessed mechanical properties is presented in Table 2.

Table.2: Summary of Mechanical Properties Results

SAMPLE	**UTS(N/mm^2)**	**YS (N/mm^2)**	**YS/UTS**	**TEL (%)**	**IS (J/mm^2)**	**HARDNESS**
A	670.55	385.96	0.56	21.97	1.30	161.7
IQ730	718.56	416.76	0.58	36.12	1.73	282.0
IQ750	955.70	583.00	0.61	15.65	0.78	327.8
IQ770	1184.64	805.55	0.68	7.63	0.53	358.9
IQ730T	561.28	325.54	0.58	37.11	1.68	225.3
IQ750T	327.50	-	-	12.43	0.33	307.4
IQ770T	231.31	120.28	0.52	11.67	0.25	292.2

3.2.2 Effect of Tempering on the Properties of Intermediate Quenched Sample

Fig.6 shows that tempering the as-quenched intermediately quenched sample at 320^{O}C for 1hr decreases hardness (IQT series). However, the hardness increased with temperature and reached a peak at 750^{O}C. The increased hardness with increase in martensite volume fraction can be attributed to the precipitation of carbide on tempering — see Fig. 1e and g. The decrease in value of hardness observed with tempered intermediate quenched (IQT) samples could be as a result of the coarsening of soft ferrite phase as shown in Fig. 1e and g respectively. This emanated from precipitation of more ferrite from martensite on tempering.

All the other properties equally deteriorated on tempering at 320^{O}C for 1hour.

Conventional stress – strain curve with discontinuous yielding was observed for IQ730T (i.e. sample intermediately quenched with 33% martensite volume fraction and tempered at 320^{O}C for an hour), while the others exhibited continuous yielding which is typical of conventional dual phase steel.

3.3 Fractography

Figure 7 shows the fractured surfaces of the failed impact test samples upon testing. Figures 7b, c and d present the fractured surfaces of IQ730, IQ730T and IQ750 samples respectively. They revealed predominantly dimple fibrous surface which is typical of materials with good combination of high strength, ductility and impact toughness. It showed that IQ730T has majorly dimple fibrous fractured surface with dislocations cutting across circular obstacles. On the other hand, Fig. 7e, f and g display the fractured surfaces of IQ750T, IQ770 and IQ770T respectively, which revealed majorly pure or quasi cleavage fracture, no wonder the low impact strength exhibited by these samples (Table 2).

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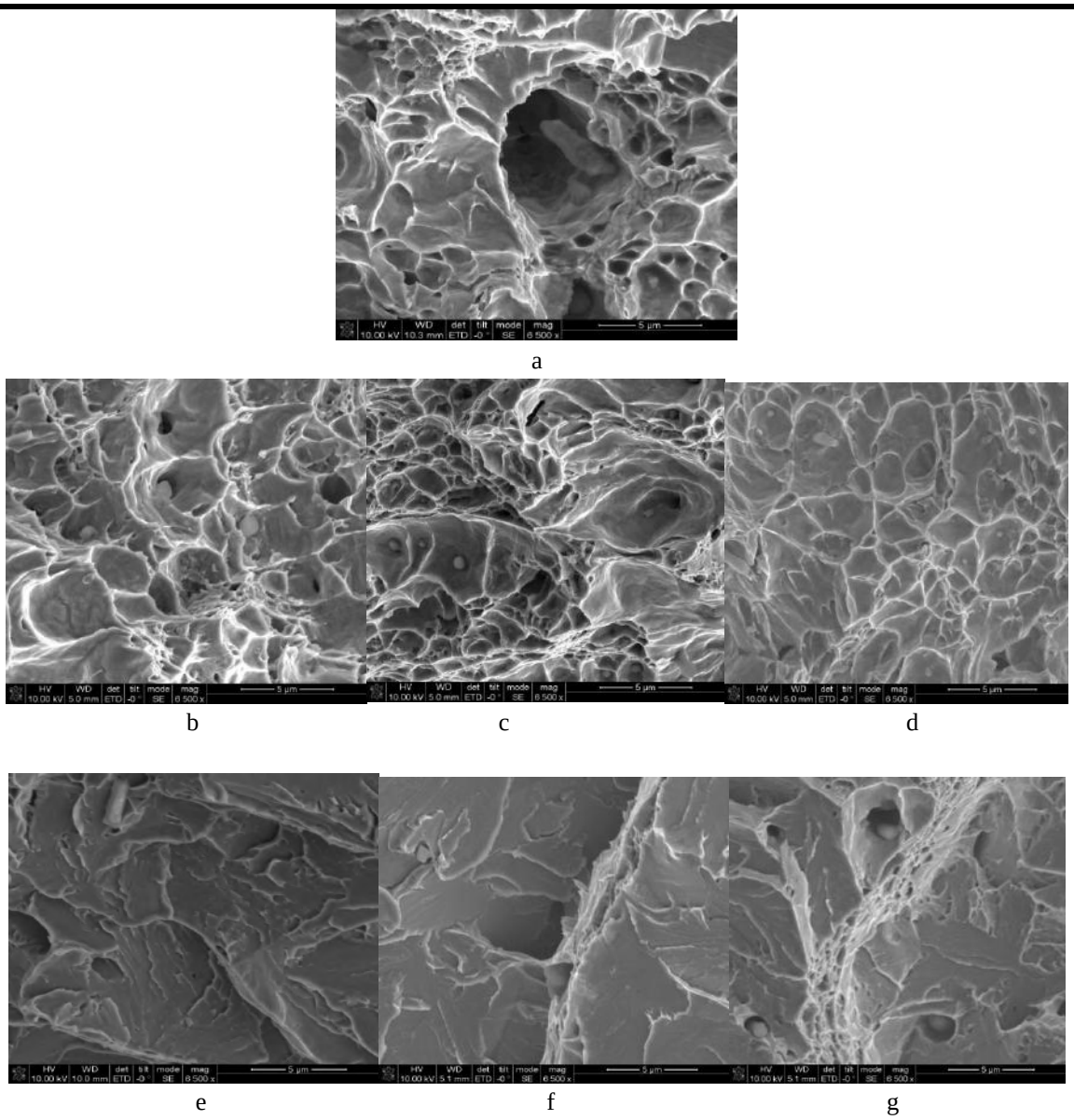

a b c d e f g

Fig. 7: (a)Impact fractured surface ofA (i.e. Sample normalised at 850^{o}C for one hour). *Structure reveals fibrous surfaces with cavity.* ***(b)Impact fractured surface ofIQ730(i.e.Sample intermediately quenched at 730^{O}C for 30 minutes).*** *Structure reveals fibrous surface.*

(c) Impact fractured surface of IQ730T (i.e. Sample intermediately quenched at 730^{O}C for 30 minutes and tempered at 320^{O}C for 1hour). *Structure reveals majorly dimple fibrous fractured surface with dislocations climbing on circular obstacles.* ***(d) Impact fractured surface of IQ750 (i.e. Sample intermediately quenched at 750^{O}C for 30 minutes).*** *Structure reveals fibrous fractured surface.* ***(e)Impact fractured surface ofIQ750T(i.e.Sample intermediately quenched at 750^{O}C for 30 minutes and tempered at 320^{O}C for 1hour).*** *Structure reveals pure cleavage surface.* ***(f) Impact fractured surface of IQ770 (i.e. Sample intermediately quenched at 770^{O}C for 30 minutes).*** *Structure reveals quasi cleavage fractured surface.* ***(g) Impact fractured surface of IQ770T (i.e.Sample intermediately quenched at 770^{O}C for 30 minutes and tempered at 320^{O}C for 1hour).*** *Structure reveals quasi cleavage fractured surface. Presence of some cracks across grain boundaries was also observed.*

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IV. CONCLUSION

From the analysis conducted in this research, it can be concluded that for intermediate quenching heat treatment:

i. Increase in intermediate quenching temperature leads to increased MVF.
ii. Tensile strength and hardness value of the as-quenched steel increased with increase in temperature and MVF while impact toughness and ductility decreases with increase in temperature and MVF.
iii. Tempering the as-quenched samples deteriorated all the properties assessed.
iv. Ferrite/martensite microstructures associated with conventional DP steels was developed from the investigated low carbon steel using intermediate quenching intercritical annealing heat treatment.

It is therefore recommended that unlike the step quenching technique, for a carbon steel of this composition, it should not be tempered after intermediate quenching heat treatment.

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