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Microstructure and Corrosion Resistance of Intercritically Treated Duplex Medium Carbon Steels

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Abstract: This study examines the mechanical characteristics and corrosion resistance of medium carbon low alloy steel subjected to intercritical annealing to develop varying ferrite/martensite duplex microstructures. Intercritical treatments produced dual-phase steels with ferrite–martensite phase fractions ranging from 45% to 80%. Mechanical performance was evaluated through hardness, tensile strength, and fracture metrics, while electrochemical and gravimetric techniques were applied to assess corrosion behavior in acidic and alkaline media. Results show that steels annealed at 760°C and 800°C for 45 and 60 minutes exhibited enhanced ultimate strength, ductility, and impact resistance, attributed to the increased proportion of refined martensite. Corrosion studies revealed that dual-phase structures provided superior resistance in alkaline solutions compared to normalized steels, but acid attack rates were primarily influenced by the connectivity of martensitic islands. SEM images confirmed uniform phase distribution without significant segregation. Specimens treated at 760°C/60min and 800°C/45min demonstrated the most advantageous blend of mechanical toughness and corrosion resistance. The findings highlight the potential of intercritical processing to optimize both durability and operational life for structural applications in harsh environments.

1. INTRODUCTION

The versatile application of ferrous materials has been attributed to its amenability to alloying and heat-treatment which makes it possible to modify its microstructure to improve desired properties to suite specific service requirements [1,2]. One often explored structural modification is the development of dual phase steels which are used to a large extent in the production of automobile body parts [3]. Dual phase steels possesses a ferritic (soft and ductile) and martensitic (hard and brittle) microstructure; and the combination of both phases makes it possible to develop high strength-ductile microstructures in low carbon micro alloy steel [4,5].

Plain carbon and medium carbon low alloy steels are utilized locally for a number of applications particularly for structural use. For such applications, high strength, toughness and reasonable ductility are some of the crucial engineering properties that are required for excellence service performance [3,6]. In the quest for development of steel based materials for applications where high

strength and ductility are both required, a lot of researches have been carried out on dual phase steel production by treatment in the (α+γ) phase region. This has led to the development of 70% - 30% ferrite – martensite dual phase proportions for the development of automobile body parts from low carbon microalloy steel – high strength low alloy steels [7,8]. Sun and Pugh [9] investigated the properties of thermomechanically processed dual – phase steels containing fibrous martensite; Xu et al [10] researched on the mechanical properties of fine-grained dual phase low – carbon steels based on dynamic transformation. Dongsheng et al [11] studied the microstructural evolution for hot rolling of dual phase steel with lean C-Mn-Si chemistry. He discovered that the ferrite grain size is quantified as a function of transformation start temperature. Das et al [12] studied the mechanical properties of a low-carbon microalloy steel subjected to three different heat treatment schedules namely, intermediate quenching (I_Q), step quenching (S_Q) and intercritical annealing (I_A). They discovered that the intermediate quenching treatment forms very fine and fibrous martensite uniformly distributed in the ferrite matrix and also exhibited much higher impact toughness value compared to S_Q and I_A samples. Alaneme [3] investigated the phase transformation behaviour in low alloy steel during treatment in the intercritical phase region. He observed that well defined micro-duplex structures of ferritic and martensitic nature, possessing good combination of strength and ductility were obtained in normalized and 650^{0}C tempered martensite initial microstructures. He also observed that the 300°C and 500 °C tempered martensite initial microstructures were unsuitable for the production of dual phase microstructures with optimized strength characteristics.

The conventional medium carbon steel microstructures have been observed not to offer the broad property base highlighted above. Little finding have been offered on the development of dual phase steel with medium carbon steel, this is due to a great extent on the complex nature of the transformation processes occurring during treatment of steels of medium carbon composition in the (α+γ) phase region. This has made it difficult to optimize the process variables and potential engineering properties that can be harnessed from the intercritical treatment [11,14]. Alaneme and kamma [15] attributed this to the competing metallurgical phase reactions like solid solution decomposition, precipitation, recrystallization and coalesces of phases which occur concurrently with the $\alpha \rightarrow \gamma$ during intercritical treatment.

This research work is an effort to optimize the mechanical properties of the medium carbon low alloy steels without compromising its ductility by adoption of intercritical treatment conventionally applied to low carbon micro-alloy steels for the development of dual phase steels.

2. MATERIALS AND METHODS

2.1. Materials

The steel material for the investigation was medium carbon low alloy steel as-supplied as cylindrical rod of 10.5 mm. The chemical composition of the steel rod was determined using a spark spectrometric analyser; and the result is presented in Table 1.

Table 1: Chemical Composition of the MCLA Steel (in wt %)

Elements	C	Si	S	P	Mn	Ni	Cr
Composition	0.3300	0.1740	0.0499	0.0341	0.8225	0.0911	0.0585
Elements	Mo	V	Cu	W	As	Sn	Co
Composition	0.00180	0.0029	0.3031	0.0003	0.0060	0.0230	0.0094
Elements	Al	Pb	Ca	Zn	Fe		

Composition	0.0019	-0.0006	0.0002	0.0037	Bal.		

2.2. Design of Dual Phase Microstructures

Intercritical heat-treatment process was adopted for the design of dual phase microstructures in the medium carbon low alloy steel samples. The steel rod was initially normalized by heat-treating at 860°C for one hour followed by air cooling in order to eliminate the previous thermal and mechanical history of the steel. Thereafter, the lower critical temperature (AC_1) and the upper critical temperature (AC_3) for the medium carbon low alloy steel composition was determined in accordance with Alaneme (2011). Intercritical treatment was performed at 760°C for holding periods ranging between 5 and 90 minutes followed by quenching in water maintained at 40°C (in order to avoid the development of quench cracks).

Table 2: Sample Designations with Percentage volume fractions of developed DP Steels

Specimens designation	Treatment		Volume of ferrite (%)	Volume of martensite (%)
	Temperature (°C)	Holding time (mins)		
Cont.	Normalizing		55.55	44.45 (Pearlite)
DP1	790	15	20.39	79.61
DP2	750	15	29.79	70.21
DP3	750	60	38.05	61.95
DP4	790	60	41.59	58.41
DP5	790	30	45.40	54.60

2.3. Hardness Measurement

The hardness values of all treated specimens were evaluated using a Digital Rockwell Hardness Tester. Prior to the test, the specimens were polished to obtain a smooth surface. A direct load of 584.9MN (60kg) was applied on the specimens and the hardness values were recorded. Multiple hardness tests were performed on each specimen and the average value was taken as the hardness of the specimen.

2.4. Tensile Testing

Room temperature uniaxial tension tests were performed on round tensile samples machined from the prepared composites with dimensions of 6 mm diameter and 30 mm gauge length. The testing was performed using an Instron universal testing machine operated at a constant cross head speed of 1mm/s. The specimen dimension specifications and the test procedure adopted were in conformity with ASTM E8M - 91 standards [16]. Three repeat tests were performed for composite composition to guarantee reliability of the data generated. The tensile properties evaluated from the stress-strain curves developed from the tension test are - the ultimate tensile strength (σ_u), the 0.2% offset yield strength (σ_y), and the strain to fracture (ε_f).

2.5. Fracture Toughness Evaluation

Circumferential notch tensile (CNT) specimens were also prepared for the evaluation of fracture toughness in accordance with Alaneme [6] and Ahmed et al [8]. The CNT specimens were machined with gauge length of 30mm, specimen diameter of 6mm (D), notch diameter of 4.5mm (d) and notch angle of 60°. The specimens were then subjected to tensile loading to fracture using an Instron universal testing machine. The fracture load (P_f) obtained from the CNT specimens' load – extension plots were used to evaluate the fracture toughness using the empirical relations by Dieter [17]:

$$K_{1C} = \frac{P_f}{D^{3/2}[\alpha_1 + \alpha_2(d/D) + \alpha_3(d/D)^2]} \quad (2.1)$$

where, D and d are respectively the specimen diameter and the diameter of the notched section. The validity of the fracture toughness values was evaluated using the relations in accordance with Nath and Das [2]:

$$D \geq (K_{1C}/\sigma_y)^2 \quad (2.2)$$

A minimum of two repeat tests were performed for each composite composition and the results obtained were taken to be highly consistent if the difference between measured values for a given composite composition is not more than 2%.

2.6. Microstructural Examination

Microstructural examination of specimens was performed using a ZEISS Axiovert 200MAT optical microscope with accessories for image analysis. The specimens for the optical microscopy were metallographically prepared by grinding using a series of emery papers of grit sizes ranging from 60 – 2400µm; while fine polishing was performed using polycrystalline diamond suspension of particle sizes ranging from 10 – 0.5µm with ethanol solvent. The specimens were etched with 2%Nital solution by swabbing for 5 – 10 seconds before observation with the optical microscope. **2.7. Corrosion Testing**

The corrosion test was investigated in 0.3M H_2SO_4 and 0.3M NaOH solutions which were prepared following standard procedures. Prior to immersion, the samples surface were mechanically polished with emery papers of up to 400 grits on a rotary buehler grinding machine. It was then accurately weighed, immersed in 0.3M H_2SO_4 and 0.3M NaOH solution for the desired exposure time. After each exposure time the samples were removed from its beakers, dried and reweighed to a constant weight using an analytical balance with accuracy of ±0.0001g. The experimental results were thereafter evaluated in accordance to ASTM G – 31 standard procedures.

3. RESULTS AND DISCUSSION

3.1. Hardness

Figure 1 shows the variation of hardness with the intercritical treatment, observation of the graph show a significant rise in hardness values of the intercritically treated samples (DP3 – DP5). This increment signifies the formation of increased amount of martensite, as a function of holding time, in the specimens. However, the very slight increase in hardness values of specimen DP2 and the slight reduction in DP1 could be as a result of multiple phase reactions occurring simultaneously, for example, decomposition of cementite [6,14], spheroidization and primary recrystallizaton which are dominant at this stage [11]. These reactions occur in situ and compete with (α + γ) phase transformations taking place. In the over all, the specimen treated at 750°C and held for 60min was observed to exhibit the highest hardness value of 219.2HV.

3.2. Mechanical Behaviour

The tensile properties of the dual phase structures are summarized in Table 3. The yield strength (Y.S) and ultimate tensile strength (U.T.S) increased with the volume percent martensite formed while the percent elongation for all duplex phase structures ranged between 24.0% - 25.32% with a negligible difference of less than 1.32 in comparison with the conventional normalized structure which had percent elongation of 25.35%. The increase in tensile strength and yield properties of developed DPS strength with increase in the volume percent martensite (especially for DP3 specimens) is in harmony with the observations of Kumar et al [18]. The fracture toughness (K_{1C}) values presented in Table 3 revealed higher toughness values for all the dual phase structures intercritically treated at 750^{0}C and 790^{0}C which is consistent with the large volume fractions of martensite. The conventionally normalized specimen is observed to possess the least toughness of 56 $MPa\sqrt{m}$. The generally improved toughness observed in the dual phase structures is attributed to the composite structure of ferrite and martensite which creates a synergy of the soft ferritic phase and the hard martensitic phase, which helps in increasing the materials resistance to crack propagation and fracture [19].

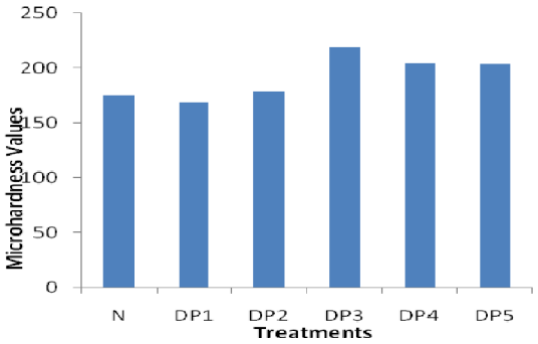

Fig. 1: showing the plots of variation of hardness

Table 3: Summary of Mechanical Properties of the Dual Phase Steels Produced.

Sample	HV	Y.S (MPa)	UTS (MPa)	Strain to fracture (%)	Load to fracture (N)	K_{1C} ($MPa\sqrt{m}$)
N	175.2	335.61	561.02	25.35	1230.780	56.58775
DP1	168.7	348.75	564.86	24.97	1357.993	62.43648
DP2	179.1	417.05	603.68	25.20	1242.354	57.11974
DP3	219.2	495.77	646.10	25.32	1513.511	69.58673
DP4	204.7	358.66	568.17	24.88	1495.255	68.74734
DP5	203.7	428.64	595.32	24.00	1370.501	63.01156

3.3. Micrographs

Figure 2(a) shows the micrographs of the normalized structure treated at 870^{0}C and gradually cooled in air. An equilibrium structure (pearlite) is formed, which is subsequently intercritically heat – treated in order to enhance the coalescence of cementite in the initial structure [20], thus resulting in the commencement of ($\alpha + \gamma$) transformation.

Figures 2(b) – (f) are micrographs of specimens DP1 – DP5 treated at 750^{0}C and 790^{0}C respectively over varied holding times formed from pearlite as initial microstructure. It is observed that the individual microstructures consist essentially of a homogenized ferrite (gray phase) and martensite (dark – formerly austenite). The grain sizes and volume fraction of martensite is observed to be dependent on the soaking time and intercritical temperatures [3].

The developed dual phase structures shown in Figure 2(b) – (f) confirms that medium carbon low alloy steel, when intercritical treated develops microstructures that exhibited micro-duplex features consisting of soft and ductile ferritic phase combined with hard and brittle martensitic phase.

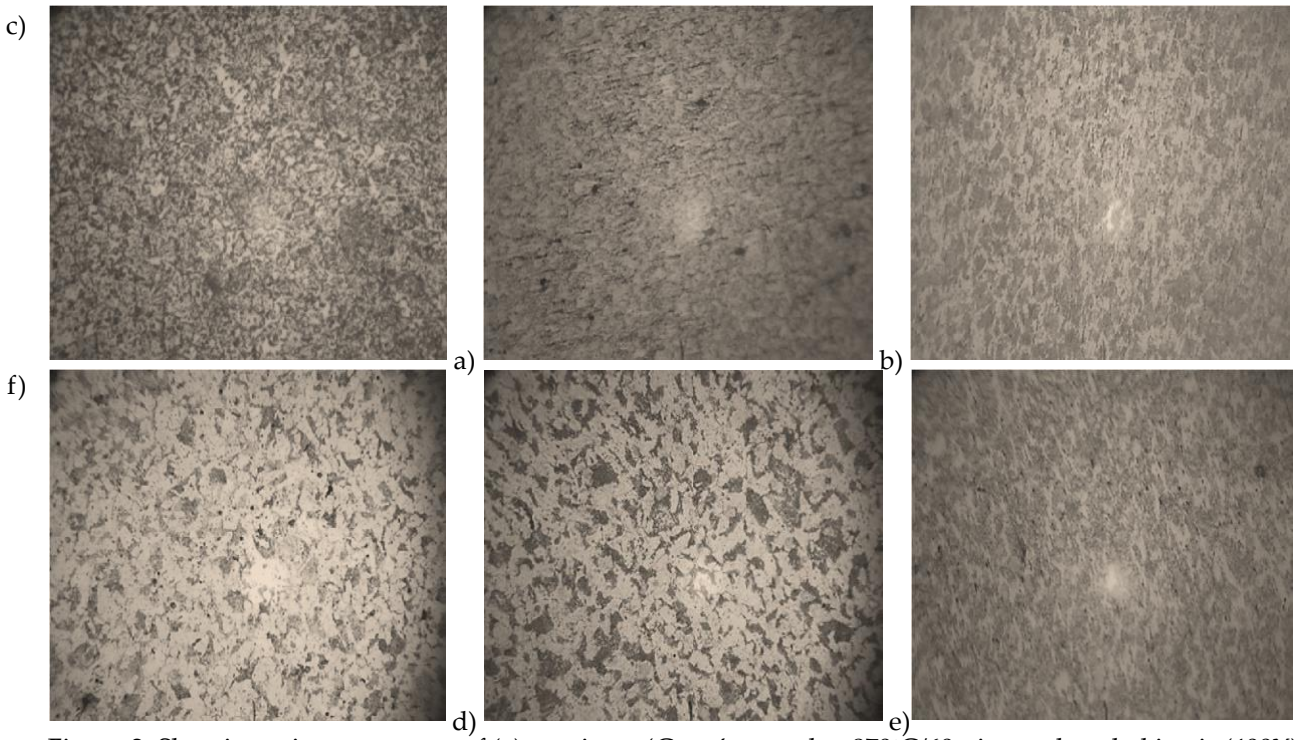

Figure 2: Showing microstructures of (a) specimen 'Cont.' treated at 870^{0}C/60mins and cooled in air (400X), (b) specimen DP1 treated at 870^{0}C/air cool/790^{0}C/15mins/Quenched in warm water (400X), (c) specimen DP2 treated at 870^{0}C/air cool/750^{0}C/15mins/Quenched in warm water (400X), (d) specimen DP3 treated at 870^{0}C/air cool/750^{0}C/60mins/Quenched in warm water (400X), (e) specimen DP4 treated at 870^{0}C/air cool/790^{0}C/60mins/Quenched in warm water (400X), and (f) specimen DP5 treated at 870^{0}C/air cool/790^{0}C/30mins/Quenched in warm water (400X)

3.4. Corrosion Behaviours

Table 1 shows the Chemical composition (in weight percent), it indicates a low sulphur and phosphor content, which provides high purity and quality for the selected medium carbon low alloy steel, because it minimizes the possibility of sulphides formation and other inclusions.

The corrosion rate and mass loss of the developed dual phase structures in 0.3M H_2SO_4 and 0.3M NaOH solutions are represented in Figure 3 – 6. The plots explain the corrosion behaviours of the various heat treatment induced microstructures. Figure 3 shows the variation of mass loss plots to exposure time in 0.3M H_2SO_4 solution. It was observed that the dual phase structures produced in specimens treated at 750^{0}C and 790^{0}C for 60mins (i.e DP3 and DP4) displayed a better level of corrosion resistance in 0.3M H_2SO_4 solution. Figure 4 shows the corrosion rate plots, it is observed that the corrosion rates for all specimens immersed in 0.3M H_2SO_4 medium attain their peak corrosion rate value within the first six (6) days and sinusoidally decreased as a function of exposure time. General observations on the medium profiles indicate that specimens DP4 (i.e specimen treated at 790^{o}C and held for 60 minute) display the highest corrosion resistance in the medium. Figure 5 shows the variations of mass loss to exposure time of that specimen DP4, among others, also displays the highest corrosion resistance as it loses the least mass in the 0.3M NaOH medium. Figure 6 as well; show the superimposition of the corrosion-rate plots of the specimens in 0.3M NaOH medium. An interwoven alternating trend is observed to be displayed by all the specimens immersed in the medium with majority of the specimens attaining their peak values within the first six (6) days. The sinusoidal trend of the mass loss and corrosion behaviour of the specimens in 0.3M NaOH solution shows the formation of passive film over the surface of the specimen

which prevent it from the attack of the solution. The curve in most of the developed structures begins with gain in mass (indicating formation of passive film), mass loss (indicating breakage of the passive film) on the third day and reform on the sixth day and continues in the same manner for the period of exposure time studied. This phenomenon is as a result of the presence of the lean corrosion resistance elements present in the selected material.

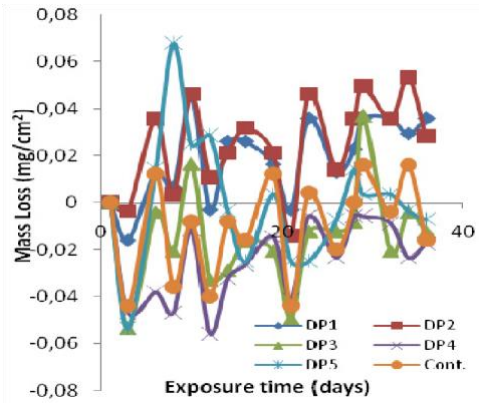

Fig. 5: Graph showing the plots of mass-loss versus exposure time of DP1 – DP5 in 0.3M NaOH

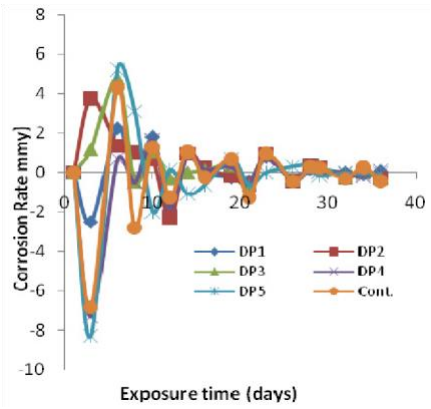

Fig. 6: Graph showing the plots of corrosion rate versus exposure time of DP1 – DP5 in 0.3M NaOH

General observations in both media's profiles shows that specimen DP4 treated at 790^{0}C and held for 60 minutes have the lowest corrosion rates value and thus displays the best corrosion resistance in 0.3M H_2SO_4 and 0.3M NaOH solutions. The reason for the observed corrosion resistance in the developed dual phase structures could be attributed to the presence of ferrite in an amount (Table 2) that could reduce the susceptibility of the structure to galvanic corrosion. The entrapment of most of the carbon atoms in the martensite structure [16] could serve as another reason for the noticeable corrosion resistance of the intercritically developed DPS in the media.

4. CONCLUSION

In the present study, the microstructures, mechanical properties and corrosion behavior of dual phase medium carbon low alloy (MCLA) steel was studied. From the result, it was observed that medium carbon low alloy steel when subjected to intercritical treatment at 750^{0}C and 790^{0}C resulted in a significant increase in the tensile and hardness properties of the developed dual phase steel in comparison to the normalized specimen while maintaining its ductility. The micrographs as well showed a uniformly distributed dual phase structure of ferrite and martensite at various volume fractions. It was observed from the corrosion rate and mass loss plots that there was a significant improvement in the corrosion resistance of the developed Dual Phase structures in the selected media (0.3M H_2SO_4 and 0.3M NaOH). Overall, specimen DP3 and DP4 were observed to possess the best combination of tensile properties, hardness, fracture toughness.

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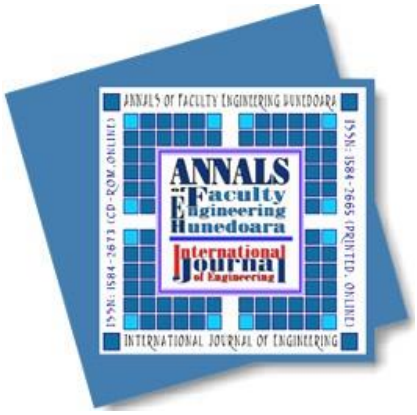

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