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# INFLUENCE OF DIFFERENT CYCLIC INTERCRITICAL HEATTREATMENT SCHEDULES ON THE MICROSTRUCTURE AND MECHANICAL BEHAVIOUR OF A DUAL PHASE MEDIUM CARBON LOW ALLOY STEEL

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### Abstract

In the present research, the prospect of optimizing high strength and formability in medium carbon low alloy steel via different cyclic intercritical treatment procedures was investigated. Three intercritical treatment schedules (Route A - cyclic intercritical annealing only, Route B - cold rolling followed by cyclic intercritical annealing, and Route C - cyclic cold rolling and intercritical annealing operations) performed at 770°C were utilized in this research work. For each treatment route, a maximum of three heating cycles was used. Microstructural examination, hardness measurement and tensile properties evaluation were used as basis to assess the mechanical behaviour of the dual phase structures produced. The results show that for Route A and B, grain refinement and homogeneous distribution of ferrite and martensite was obtained for specimens subjected to two cycles of intercritical annealing. This resulted in peak strength, toughness and hardness in comparison to specimens subjected to one or three cycles of intercritical annealing. For Route C, the same properties were impoverished with increase in intercritical annealing cycles. The best combination of hardness, strength, toughness and strain to fracture was achieved with the use of an initial cold rolling and two cycles of intercritical annealing at 770°C.

*Keywords: dual phase steels; cyclic intercritical annealing; mechanical behaviour; cold rolling; strength* 

### Introduction

The science and applications of dual phase steels has continued to attract a lot of interest among researchers in the physical metallurgy of steel [1-4]. This is because the advancement in automobile design and manufacturing technologies frequently demands

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the use of dual phase steels having improved combination of high strength, toughness and excellent formability [5-6]. This property combination is required for thinner steel gauges having significant high strength to weight ratio, ductility and improved crash performance [7-8]. The production of machines, auto and truck body parts; vehicle chasis, engine seats, automobile rims and structural parts are a few of the applications requiring this set of properties [9-10]. Dual phase medium carbon low alloy steel grades have been successfully utilized for these applications by virtue of its peculiar combination of ferrite and martensite ( $\sim 50 - 60\%$ ) in proportions different from that of conventional low carbon microalloyed dual phase steels [11]. The design of dual phase structures with well defined ferrite - martensite phases in steels with medium carbon composition require a thorough understanding of the transformation mechanisms that occur in the  $(\alpha + \gamma)$  phase region during intercritical heat-treatment operations [12]. The proportion, size, dispersion, and morphology of ferrite and martensite phases in steels with medium carbon composition are reported to require proper selection of the intercritical heat-treatment processing parameters [13]. This is necessary so as to control the kinetics of competing phase reactions such as phase decomposition, precipitation and recrystallization which often retard the  $\alpha \rightarrow \gamma$  transformation rates during intercritical heat-treatment [14-17]. The influence of initial microstructural conditions, cold deformation ratio, intercritical temperatures and holding times on the chronological order of the transformation mechanisms that occur in medium carbon low alloy steels has also been reported [17-18]. These studies have served as background to the design of ferrite - martensite microstructures with high strength and ductility, improved fatigue and fracture properties in low alloy steels with medium carbon composition [7, 19-21]. However, there is still prospect of optimizing the strength – plasticity combinations in this grade of steel to meet its growing industrial applications if the ferrite -martensite phases can be processed to finer length scales.

The use of cyclic heat-treatment operation to produce dual phase microstructures in medium carbon low alloy steels has not received much research attention judging from available literatures. Cyclic heat-treatment performed at optimum conditions has been reported to have the benefit of refining grain structures increasing significantly the potentials of enhancing high strength, work hardening capacity and plasticity in metallic alloys [22]. Cyclic heat-treatment has been applied to refine the grain structure in a variety of steel grades with marked improvement in strength levels [23-24]. But its application in the intercitical phase region with a view to enhancing both strength and plasticity has not received much attention for medium carbon low alloy steels. Cyclic heat-treatment in two phase metallic systems can resulth in the production of microduplex structures with the potentials of higher strength levels and extended plasticity [10, 25]. In the represent study, the influence of different cyclic intercritical heattreatment routes on the microstructure and transformation behaviour of a medium carbon low alloy steel is investigated.

# Materials and methods

### Materials and Sample Preparation

Commercial medium carbon low alloy steel as supplied as cylindrical rib rod of 12mm was used as test material for the investigation. The chemical composition of the steel rod was determined using a spark spectrometric analyzer; and the result is presented in Table 1. Normalizing heat-treatment was carried out on the rods at 860°C

for one hour followed by cooling in air in order to eliminate the previous thermal and mechanical history of the steel.

Table 1: Elemental composition of the Medium carbon low alloy steel

element	С	Si	Mn	Р	S	Cr	Ni	Мо	Со	Fe
wt %	0.31	0.13	0.88	0.086	0.029	0.11	0.095	0.033	0.01	98.35

### Cyclic Intercritical Treatment Operations

Before commencing with the intercritical heat-treatment operations, the reference lower critical temperature  $(AC_1)$  and upper critical temperature  $(AC_3)$  for the steel composition was determined in accordance with Alaneme [12]. The test materials were then divided into three groups for the cyclic intercritical heat-treatment operations.

All the test specimens for Route A processing were subjected to cyclic intercritical annealing only. This was achieved by initially subjecting the samples to normalizing heat treatment at 860°C for 60 minutes followed by cooling in air. The specimens were then subjected to intercritical heat-treatment at 770°C holding for 45 minutes followed by quenching in water maintained at 40°C (in order to avoid the development of quench cracks). The intercritical heat-treatment was repeated for a maximum of three (3) cycles.

The specimens for Route B processing were subjected to cold rolling and cyclic intercritical annealing treatment. This was achieved by initially subjecting the samples to normalizing treatment followed by 20% deformation by cold rolling. The specimens were then isothermally treated at 770°C, holding for 45 minutes followed by rapid quenching in water maintained at 40°C. The intercritical heat-treatment was repeated for a maximum of three (3) cycles.

The specimens for Route C processing were subjected to repeated cold rolling and intercritical annealing treatment. This was achieved by initially subjecting the samples to normalizing treatment followed by 20% deformation by cold rolling. The specimens were then heat-treated at 770°C for 45 minutes followed by quenching in water maintained at 40°C. The specimens were further subjected to 10% deformation by cold rolling operation and then heat-treated at 770°C, holding for 45 minutes followed by quenching in water maintained at 40°C. The cold rolling - intercritical heat-treatment operation was repeated for a maximum of three (3) cycles. The specimen treatment and corresponding designations are presented in Table 2.

Treatment	Sample
	Designation
770°C, 45mins/H <sub>2</sub> O	AI
770°C, 45mins/H <sub>2</sub> O+ 770°C, 45mins/H <sub>2</sub> O	AII
770°C, 45mins/H <sub>2</sub> O + 770°C, 45mins/H <sub>2</sub> O + 770°C, 45mins/H <sub>2</sub> O	AIII
$20\% \text{ D} + 770^{\circ}\text{C}, 45 \text{mins/H}_2\text{O}$	BI
$20\% \text{ D} + 770^{\circ}\text{C}, 45 \text{mins/H}_{2}\text{O} + 770^{\circ}\text{C}, 45 \text{mins/H}_{2}\text{O}$	BII
$20\% \text{ D} + 770^{\circ}\text{C}, 45\text{mins/H}_{2}\text{O} + 770^{\circ}\text{C}, 45\text{mins/H}_{2}\text{O} + 770^{\circ}\text{C},$	BIII
45mins/H <sub>2</sub> O	
20% D + 770°C, 45mins/H <sub>2</sub> O + 10% D + 770°C, 45mins/H <sub>2</sub> O	CI

Table 2: Sample Designations for the Different Cyclic Intercritical Heat-treatment Procedures

20% D + 770°C, 45mins/H <sub>2</sub> O + 10% D + 770°C, 45mins/H <sub>2</sub> O + 10% D +	CII
770°C, 45mins/H <sub>2</sub> O	
20% D + 770°C, 45mins/H <sub>2</sub> O + 10% D + 770°C, 45mins/H <sub>2</sub> O + 10% D +	CIII
$770^{\circ}$ C, $45$ mins/H <sub>2</sub> O + $10\%$ D + $770^{\circ}$ C, $45$ mins/H <sub>2</sub> O	

### 2.3 Mechanical Testing

Hardness test was utilized to analysis the hardening behaviour of the test specimens for each cyclic intercritical processing condition. The hardness values of all test specimens were evaluated using a Digital Rockwell Hardness Tester. Prior to testing, the steel specimens were polished to obtain a smooth surface finish. A direct load of 584.9MN (60kg) was thereafter 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.

Tensile testing of all specimens was conducted at room temperature using a Table-Top tensile testing machine. The sample preparation and testing procedures was performed in accordance with the ASTM E8M-91 standard [26]. The tensile properties evaluated are: the ultimate tensile strength ( $\sigma_u$ ), the yield strength ( $\sigma_y$ ), toughness (the area under the stress-strain plot) and the strain to fracture ( $\epsilon_f$ ).

## **Microstructural Examination**

Microstructural examination of the test 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  $60 - 2400\mu m$ ; while fine polishing was performed using polycrystalline diamond suspension of particle sizes ranging from  $10 - 0.5\mu m$  with ethanol solvent. The specimens were etched with 2%Nital solution by swabbing for between 5 - 10 seconds before observation with the optical microscope.

# **Results and discussion**

### **Microstructures**

The photomicrographs presented in Figures 1 - 3 show dual phases of ferrite (white/grayish white phase) and martensite (dark phase) with varied sizes, distribution and morphology; indicating that the cyclic intercritical heat-treatment processes adopted had significant influence on the microstructure. Figure 1 shows the photomicrograph of the specimens subjected to only cyclic intercritical annealing treatment. It can be observed that more refined and uniformly dispersed ferrite (white phase) and martensite (dark phase) structures is obtained for the specimens that were subjected to two cycles of intercritical annealing treatment (Figure 1b) in comparison with specimens subjected to one cycle intercritical annealing (Figure 1a) and three cycle intercritical annealing (Figure 1a) shows ferrite-martensite structure which are not as dispersed as observed in specimens subjected to two cycles of intercritical annealing (Figure 1b). The photomicrograph of the specimens subjected to three cycles of intercritical annealing (Figure 1c), shows more coarse grains of ferrite annealing above two with Figure 1(a) and 1(b). This indicates that repeated intercritical annealing above two

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cycles may not be beneficial for the purpose of grain refinement and obtaining a homogeneous ferrite/martensite distribution.





(c)

Figure 1: Photomicrograph showing (a) dual phase microstructure of ferrite (white phase) and martensite (dark phase) for specimens subjected to one cycle of intercritical annealing at 770°C, (b) fine distribution of ferrite (white phase) and martensite (dark phase) of specimens subjected to two cycles of intercritical annealing at 770°C, and (c) fairly large grain size distribution of specimens subjected to three cycles of intercritical annealing at 770°C.

For the cold rolled – cyclic intercritical annealed specimens (Figure 2) it is observed that there is a significant change in the size and morphology of the dual phase ferrite-martensite structures with increase in the number of cycles. The specimens subjected to two cycles of intercritical annealing after cold rolling (Figure 2b), is observed to have the best distribution of martensite and a more refined grain structure in comparison with specimens cold rolled and subjected to one cycle intercritical annealing (Figure 2a) and the samples cold rolled and subjected to three cycles of intercritical annealing (Figure 2c). The photomicrograph of the specimens cold rolled and subjected to one cycle intercritical annealing shows a dual phase structure of ferrite (white phase) and martensite (dark phase). The specimens subjected to cold rolling and three cycles of intercritical annealing, also consist of ferrite and martensite but with relatively coarse grain structure. This indicates that optimum grain refinement is achieved by the adoption the cold rolling and two cycles of intercritical annealing treatment.



Figure 2: Photomicrograph showing (a) recrystallized ferrite (grayish white phase) dispersed with martensite (dark phase) for specimens subjected to cold rolling and one cycle of intercritical annealing at 770°C, (b) fine distribution of ferrite (white phase) and martensite (dark phase) for specimens subjected to cold rolling and two cycles of intercritical annealing at 770°C, and (c) coarse dual phase structure of ferrite and martensite for specimens subjected to cold rolling and three cycles of intercritical annealing at 770°C.

For the specimens subjected to repeated cold rolling – intercritical annealing heat-treatment (Figure 3), it is observed that the average grain size of the specimens increases with increase in the number of cold rolling – intercritical annealing cycles. This is in contrast with the observations made from Figure 1 and Figure 2. For the specimens subjected to one cycle of cold rolling – intercritical annealing (Figure 3a), it is observed that the microstructure consists of seemingly dominant martensitic structure. The specimens subjected to two cycles of cold rolling – intercritical annealing treatment is observed to consist of ferrite and martensite structures. For the specimens subjected to three cycles of cold rolling – intercritical annealing treatment it is observed that the

structure consists of ferrite and martensite with average grain size larger than observed in Figure 3(a) and 3(b). The microstructural observations indicate that cyclic intercritical annealing processing of medium carbon low alloy steel can be used as a reliable method for grain refinement as a means of enhancing strength and plasticity of the steel grade.





(c)

Figure 3: Photomicrograph showing (a) microstructure revealing a dominant martensitic phase for specimens subjected to one cycle of repeated cold rolling and intercritical annealing, (b) ferrite (white phase) and martensite (dark phase) for specimens subjected to two cycles of repeated cold rolling and intercritical annealing, and (c) more conspicuous signs of recrystallized ferrite (white phase) and martensite (dark phase) for specimens subjected to three cycles of repeated cold rolling and three cycles of intercritical annealing.

### Hardness Behaviour

Figure 4 shows the variation of hardness with number of cyclic intercritical annealing for the different processing routes adopted. It is observed that the hardness is markedly affected by the intercritical heat-treatment route and the number of intercritical annealing cycles. For specimens subjected to cyclic intercritical annealing treatment following Route A and B (cyclic intercritical annealing only and cold rolling

– cyclic intercritical annealing respectively), it is observed that peak hardness is obtained after two cycles of intercritical annealing at 770°C, after which there is a reduction in hardness with further increase in the number of annealing cycles. For specimens subjected to cyclic intercritical annealing through Route C (cyclic cold rolling and intercritical annealing), it is observed that the cyclic heat-treatment procedure did not improve the hardening capacity of the specimens as the hardness decreased with increase in the number of annealing cycles. These observations are supported by the photomicrographs (Figures 1 – 3) which show that more refined and evenly dispersed ferrite - martensite phases are obtained for specimens  $A_{II}$ ,  $B_{II}$ , and  $C_{I}$ , which are representative microstructures of specimens subjected to two annealing cycles via Route A and B, and one annealing cycle via Route C. The more refined grain structure obtained at these processing conditions result in enhanced strength due to the synergy of the strengthening mechanisms of grain size reduction, solid solution, dispersion and phase hardening [27-28].



Figure 4: Hardness Behaviour of the Cyclic Intercritical Heat-treated Specimens

### Tensile Properties

The tensile properties derived from the intercritical heat-treatment procedures are presented in Figure 5 - 7. Figure 5 shows the variation of yield and tensile strength for all specimens subjected to the varied cyclic intercritical heat-treatment routines. It is observed from Figure 5 that specimens subjected to 2 cycles of intercritical annealing through Route A (cyclic intercritical annealing only, AII) and Route B (cold rolling and cyclic intercritical annealing, BII) had peak strength levels compared to specimens subjected to one and three cycles of intercritical annealing. The specimens processed through Route C on the other hand exhibited reduction in yield and tensile strength with increase in the number of intercritical annealing cycles (CI had the highest strength levels for this processing route). These observations are consistent with the hardness trend observed in Figure 4. The finer grain structure (consisting of ferrite and martensite) and the improved dispersion of the phases as discussed in section 3.2 is responsible for the improved strength observed at AII, BII, and CI. It is noted that the specimen BII processed through cold rolling and 2 cycles of intercritical annealing had the highest tensile strength value (904.5MPa) for all processed specimens. Figure 6 shows the toughness of all specimens subjected to the varied cyclic intercritical heattreatment processes. It is observed that the specimens processed through Route C (repeated cold rolling – intercritical annealing) had lower toughness values in comparison with those subjected to Route A and Route B processing. Peak toughness of  $135J/m^3$  was obtained from the specimen subjected to cold rolling and 2 cycles of intercritical annealing treatment (BII). For the strain to fracture results (Figure 7), it is also observed that specimens processed through Route C had the least strain to fracture values (15-17 %) in comparison with specimens processed through Route A (17.5-19.5 %) and Route B (21.5-25%). This indicates that Route C processing may not be suitable for the purpose of improving strength and plasticity for the medium carbon low alloy steel composition worked on. The best combination of tensile properties is observed from the specimen (BII) subjected cold rolling and 2 cycles of intercritical annealing. The property combination obtained from this processing condition gives an improvement over results reported in literature for similar medium carbon low alloy steel compositions [3, 7].



Figure 5: Yield and Ultimate Tensile Strength of the Cyclic Intercritical Heat-treated Specimens



Figure 6: Toughness of the Cyclic Intercritical Heat-treated Specimens



Figure 7: Percent Elongation of the Cyclic Intercritical Heat-treated Specimens

### Conclusions

In this paper, the influence of different cyclic intercritical treatment on the microstructure and mechanical behaviour of a selected medium carbon low alloy steel is reported. The results show that when only cyclic intercritical annealing (Route A) or cold rolling followed by cyclic intercritical annealing (Route B) is adopted, grain refinement and homogeneous distribution of ferrite and martensite was obtained from specimens subjected to two cycles of intercritical annealing. This resulted in peak strength, toughness and hardness in comparison to one or three cycles of intercritical annealing. For repeated cold rolling and intercritical annealing (Route C), the same properties were impoverished with increase in intercritical annealing cycles. The best combination of hardness, strength, toughness and strain to fracture was achieved with the use of an initial cold rolling and two cycles of intercritical annealing at 770°C.

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