EFFECT OF INTERCRITICAL HEAT TREATMENT ON THE MICROSTRUCTURES AND MECHANICAL PROPERTIES OF MARTENSITIC DUAL PHASE STEEL

BY

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DECLARATION

I declare that the work in the project thesis entitled “Effect of intercritical at treatment on the microstructures and mechanical properties of martensitic dual phase steel” has been performed by me in Department of Metallurgical Engineering under the supervision of Dr. S.B. Hassan and Dr. O. Aponbiede.

The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this project thesis was previously presented for another degree or diploma at any university.

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CERTIFICATION

This project thesis entitled “EFFECT OF INTERCRITICAL HEAT TREATMENT ON THE MICROSTRUCTURES AND MECHANICAL PROPERTIES OF MARTENSITIC DUAL PHASE STEEL” by Bello, Kamilu Adeyemi meets the regulation governing the award of the degree of Masters of Engineering (Metallurgical Engineering) of Ahmadu Bello University, Zaria, and is approved for its contribution to knowledge and literary presentation

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DEDICATION

This research work is dedicated to ALMIGTHY ALLAH; the supreme architect of all affairs.

"Verily my devotion, my sacrifice, my living and my dying are for Allah, the lord of the 'Alamin (mankind, Jin and all that exists)".

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ABSTRACT

The effects of variation of the intercritical annealing (ICA) temperature and time on the microstructural and mechanical property behaviours of high martensite dual phase (HMDP) steel have been investigated by intermediate quenching of microalloyed steel. The treatment consisted of initial austenitization and rapid quenching of the steel samples to achieve a fully martensitic state followed by annealing in ferrite and austenite ($\alpha+\gamma$) region between 730^{o}C and 810^{o}C and subsequent quenching to produce dual phase microstructure containing varying proportion of ferrite and martensite constituents. The annealed samples were further subjected to tempering treatment at different tempering temperatures. Microstructural characterization and mechanical properties of these steel samples were studied and compared to those of conventionally quenched and tempered steel specimens. The results show that HMDP steel specimens annealed in the intercritical temperature range of 770^{o}C to 790^{o}C revealed finer martensite and precipitate-free ferrite microstructure. The tensile, hardness and impact properties of the developed HMDP steels increase with increasing intercritical annealing temperatures, with optimum properties obtained at 790^{o}C. At all the ICA temperatures, an increase in the intercritical annealing time from 30 to 90 minutes was found to enhance the tensile, hardness as well as impact properties of the HMDP steels. The tempering of HMDP steel samples significantly increases their ductility and toughness with only slight loss to strength as compared to the normal quenched and tempered steel specimens. Hence, the developed HMDP and tempered HMDP steel samples exhibited enhanced mechanical properties as compared to the conventional quenched and tempered heat-treated ones.

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CHAPTER ONE

1.0 INTRODUCTION

The design of metal parts for industrial and commercial use requires precise knowledge of the mechanical and physical properties of the metal. One reason for the versatility of steel materials lies in the wide range of attractive mechanical properties they possess which are accessible to management by various means and enable them to be used for an enormous number of applications. The mechanical properties of steels are controlled by controlling its microstructure and the nature of the phases within that microstructure. The properties of engineering alloys are, therefore, optimized by a careful control of the microstructure. The final microstructure of steel depends upon its composition and its thermo-mechanical history. This history can involve heat-treatment alone, mechanical treatment alone or combination of the two (Callister, 1997).

In practice, the modification of microstructure to effect property changes in steel has over the years being achieved by a process known as heat treatment. This process consists of combination of controlled heating and cooling operations, applied to metal and its alloy in the solid state to obtain desirable properties (Metal handbook, 1990).

For quite sometime, a lot of research has been centred on microstructure-property improvement in steel through the use of heat treatments, because of the relative cost advantage it has over the use of alloying which involves the use of alloying elements that are very scarce and expensive for a developing country like Nigeria. If properly designed and implemented, the process of heat treatment results in optimum modification in the

composition and distribution of phases; and corresponding changes in physical, chemical and mechanical properties are thus achieved in substantial measures (Rajan et al, 1988).

After years of losing ground in automotive applications to aluminium, steel is winning interest back with lighter and higher-strength steel grades (Hulka, 2002). Throughout the last few decades, a good deal of effort has been put in the development of higher strength in low carbon steel with the formability greater than those obtained in conventional controlled rolled high strength microalloyed, and various quenched and tempered steel grades Evaluation of newer materials with improved combinations of strength, ductility and toughness has led to the development of a series of microstructure-strengthened steels, in which dual phase (DP) steels represent a distinguished class**.** Of the microstructure-strengthened grades such as transformation-induced plasticity (TRIP), dual phase, high strength low alloy (HSLA) and martensitic steels; martensitic steels have the highest strength often in excess of 1200Mpa, with dual phase steels having the highest ductility (Pritchard et al.2004).

Dual phase steels are one of the important new advanced high strength steels (AHSS) product developed to satisfy an increasing (primarily in the automobile industry) need for new high strength steels that permit weight reduction without sacrificing formability nor dramatically increasing costs. They have numerous applications in the automotive component such as bumpers, wheel discs, and body panels. The specific potentials that have been technologically exploited are their superior ductility and formability characteristic compared to high strength low alloy or conventional plain carbon steels of similar strength level (Sudhakar et al, 2000). Dual phase steel is characterized by composite microstructure consists typically of a dispersion of a hard phase islands in a

ductile matrix of ferrite. The second phase is usually martensite, but other low-temperature constituents, such as bainite, may be present (Waterschoot, 2002).

The amount of martensite present in ferrite-martensitic steel will depend on the intercritical annealing temperature in the ferrite plus austenite region. As martensite is the stronger phase, any further increase in strength of dual phase steel can only be obtained by an increase of the martensite content. Unfortunately, this might be done at the expense of ductility and toughness (Saikaly et al., 2001). Hulka (2002) and Bag et al (2001) have observed, in their individual research work, that the ductility and impact toughness of these materials degrade rapidly with increasing intercritical temperatures. The degradation has been attributed to the formation of coarse martensite structure usually observed as the intercritical temperature increases based on the processing technique. However, by adopting suitable treatment procedure, it is possible to overcome this limitation by developing finer grained microstructures with a uniform distribution of ferrite and martensite phases (Hulka, 2002). To this, Bag et al (2001) has suggested, on the basis of heat treatment alone, that high-martensite dual phase (HMDP) steel with a finer microstructure and good combination of strength, ductility, and impact toughness can be obtained by either intermediate-quench (IQ) or step-quench (SQ) heat treatment technique.

Unfortunately, due to the aforementioned limitations, little research effort has been done to ensure full structural integrity of HMDP steel. Also, based on the account of temper embrittlement and limitation of strength-elongation combinations associated with the conventional quench and temper steel grades, the need to develop and adopt dual phase steel with potential cost savings and greater scope for substitution is very essential to fulfil necessary requirement for some applications in the automotive industry.

The aims of this research work are therefore;

1) To develop a high martensite dual phase steel from microalloyed steel by intermediate quench heat treatment and to establish an optimum heat treatment condition to achieve a strength-elongation improvement.

2) To investigate the effect of intercritical annealing treatment on the microstructure and mechanical properties of high martensite dual phase steels by varying the intercritical annealing temperature and time using tensile properties, impact energy and hardness values as criteria.

3) To investigate the effect of tempering temperatures on the microstructure and mechanical properties of the developed HMDP and conventional quenched steels.

CHAPTER TWO

2.0 LITERATURE REVIEW

Steel is the world's most "advanced" material. It is very versatile engineering material with a wide range of attractive properties, which can be produced at a very competitive production cost. It has a diverse range of applications, and is second only to concrete in its annual production tonnage (Westwood, 1996). The properties of steels are related to its structural make up. The desired level of mechanical properties can be achieved by altering the size, shape and distribution of various phases micro-constituent by heat treatment. Thus, heat treatment plays an important role in such a wide acceptance of steel as engineering material (Rajan et al, 1988).

Heat treatments can change the microstructures of steels by processes such as recovery, recrystallization, grain growth, and phase transformation (Honeycombe, 1982). For a given composition of steel, the recent studies have shown that a numbers of microstructure strengthened steel with microduplex structure can be developed by heat treatment in order to meet special design requirements in engineering application (Callister, 1997).

2.1 CLASSIFICATION OF STEELS

The classification of steels takes a variety of forms. Steels can be classified into two broad groups; namely plain carbon steel and alloy steels. In plain carbon steel carbon is

the main strengthening agent that governs properties while alloy steel contains carbon and certain amounts of other elements that contribute towards the properties of the steel (Rajan et al, 1988).Generally, steels are classified on the basis of their composition, structure and applications.

2.1.1 Low Carbon Steels

Low carbon steel contains about 0.1% carbon. They are not heat treated, as the hardenability is too low to produce martensite. The microstructure consists essentially of ferrite, with a small amount of pearlite. Typical mechanical properties attained are (Raghavan, 1989): 200-300Mpa yield strength, 300-370Mpa tensile strength and 28-40% elongation. The excellent formability of these steels makes them suitable for cold-formed application such as stampings of automobile and refrigerator bodies, tin cans and corrugated sheets. They are ideally suited for cold forming operations such as deep drawing stretch forming. In the tensile test, low carbon steels show a pronounced yield point, followed by plastic deformation, at constant load. This plastic deformation is inhomogeneous and occurs by the passage of luder bands. This produces a rough surface called the orange peel effects, which mars the appearance of the finer product and hence not desirable

Mild steels

Mild steel used in large tonnages for structural purpose contains 0.15-0.25% carbon. The microstructure is about 25% of fine pearlite in a ferrite matrix. Typical mechanical properties attained are: 300-350Mpa yield strength, 400-450Mpa tensile strength and 26-30% elongation.

Due to higher carbon contents, the strengths are higher as compared to the low carbon steels, but the ductility is lower. Both mild steel and low carbon steels possess

excellent weldability. Structural components (that are often welded in situ) such as plates for ship hulls, boiler, oil pipeline, I-beams, H-beams, angles, channels and girders are some of the numerous applications of mild steel (Raghavan, 1989).

Microalloyed steels

In the last decade, attempts have been made to improve the strength of mild steel, without impairing its excellent weldability. This has led to a new class of steels knowing as microalloyed steels. To the basic composition of mild steel, small amount of strong carbide forming elements such as niobium, vanadium, titanium are added. The total concentration of microalloying elements is less than 0.2%.The microalloyed steels have a very fine ferrite grain. The yield strength increases with decreasing grain size. Typical mechanical properties of microalloyed steels are: 400-500Mpa yield strength, 600-650Mpa tensile strength and 20-22% Elongation. It may be noted that the increase in strength of microalloyed steels results in lower elongation values. Unlike dual phase steels, which have good formability in spite of an increase in the tensile strength, microalloyed steel has poorer formability than conventional low carbon steel (Raghavan, 1989).

Weathering steels

If copper and phosphorous are added to mild steel, the resistance to atmospheric corrosion is improved to an extent that no painting may be required. These are known as weathering steels .Cu^{2+} and $PO4^{3-}$ ions promotes the formation of a cohesive crack-free layer of FeOOH, thereby limiting the access of oxygen and water to the metal surface. Copper in conjunction with chromium is also used for improving the corrosion resistance of mild steel.

High strength low alloy (HSLA) steels

These are group of steels with chemical composition specially developed to impart higher mechanical properties and greater resistance to atmospheric corrosion than is obtainable from conventional carbon steels. These steels contain carbon in the same range as mild steels (0.15-0.25%). In addition they all contain about 1% manganese to produce solid solution strengthening of the ferrite, and they usually contain small concentrations (less than 0.5%) of other element for additional strengthening or other effects (Kenneth, 1999). The microstructure may consist of ferrite and bainite or ferrite and tempered martensite. Typical mechanical properties attained are (Raghavan, 1989): 300-350Mpa yield strength, 400-450Mpa tensile strength and 26-30% Elongation.

These steels have satisfactory weldability, as the carbon content of martensite (if present) is low. As a class of steels they should be used wherever their high strengths or their corrosion resistance is an advantage. For instance these steels have been widely used for structural applications (bridges, tower, railing, and stairs) and they left unpainted.

2.1.2 Medium Carbon Steel

The medium-carbon steels have carbon content between about 0.25-0.65wt%. The response of these steels to heat treatment is much better than that of low carbon steels. They are most often utilized in the tempered condition, having microstructure of tempered martensite. The plain medium carbon steels have low hardernability and can be successfully heat-treated only in very thin sections and with rapid quenching rates. Addition of chromium, nickel, and molybdenum improve the capacity of these alloys to be heat treated, giving rise to a variety of strength-ductility combination. These heat-treated alloys are stronger than the low-carbon steels, but at a sacrifice of ductility and toughness. (Callister, 1997)

Rail structural

Structural parts used by the railways as rails, wheels, wheel tyre and axles are either forged or hot rolled and have a carbon content of 0.5- 0.65%. The higher level of carbon combined with about 1% Mn shifts the eutectoid composition sufficiently to yield a mostly-pearlitic structure. The microstructure is hard, strong and has good wear resistance. (Raghavan, 1989)

Spring steels

Spring steels have carbon content in the range of 0.5-0.6%. They are quenched and tempered to high-level yield strength. A spring must possess a high resilience. It must be capable of storing a large amount of elastic energy, so that it can spring back without permanently deforming. The quantity $\delta y/Y$ should be maximized for this purpose. The Young's modulus Y being a structure-insensitive property, a large increase in the yield strength is the key to achieve a high resistance. Tempering is done to give yield strength of about 1500Mpa (Raghavan, 1989).

2.1.3 High Carbon Steels

The carbon content of high carbon steels generally varies from 0.65 percent to 1.5 percent. The higher the carbon, the more is the strength with attendant brittleness. Therefore, steels with more than 1.3 percent carbon are rarely used in practice. These steels have poor fabricability, formability, mach inability and weldability as compared to medium carbon steels. Machinability and formability can be improved to a great extent by spherodizing annealing. This treatment results in reduction in strength and hardness values. However, the tendency towards brittleness is reduced considerably with attainment of a

satisfactory level of ductility. High carbon steels are utilized for making gauges, machine knives, piston rings, saws, cutting tools, chisels and hand tools.

2.2 PHASE TRANSFORMATION IN STEEL

A variety of phase transformations are important in the processing of engineering alloy materials. The development of microstructure in both single and two-phase alloys ordinarily involves some type of phase transformation. These transformations are divided into three classifications. In one group is simple diffusion-dependent transformation, in which there is no change in either the number or composition of the phases present. These include solidification of a pure metal, allotropic transformation, and, recrystallization and grain growth.

In another type of diffusion-dependent transformation, there is some alteration in phase composition and often in the number of phases present; the final microstructure ordinarily consists of two phases. Eutectoid reaction is of this type. The third kind of transformation is diffusionless, wherein a metastable phase is produced. A martensitic transformation, which may be induced in some steel alloys, falls into this category (Callister, 1997).

2.3 Austenitic Solid-State Reactions in Steel

Austenitization converts steel into the fcc solid solution called austenite when the steel is heated into the austenitic phase field and held there long enough. In the case of alloy steels, austenite may also have other elements dissolved in it in addition to carbon (Raghavan, 1989). It is the first and foremost step in the majority of heat-treating processes and the nature of the austenite that is obtained is important because it has a direct effect

upon the microstructure produced during subsequent treatments in which the austenite is transformed into other phases. Thus, the array of microstructures (and thus mechanical properties) that are obtainable in steel by various heat-treatments depends on the manner in which austenite transforms when it is cooled to room temperatures (Sinha, 1989).

The process of austenite formation on heating proceeds by nucleation and growth reaction. Therefore, the factors, which can vary either the rate of nucleation or the rate of growth or both, will change the kinetic of austenite formation. Two such parameters are transformation temperature and holding time at transformation temperatures. The transformation of austenite on cooling is a complex process in the sense that compositional as well as configurational changes are involved and are discussed below.

2.4 EFFECTS OF ALLOYING ELEMENT ON IRON CARBON ALLOYS

A study of the effect of alloying elements on iron-carbon phase diagram (fig.2.1) is very helpful in understanding the various heat treatment processes of alloy steels. By knowing the behaviour of such elements in the Fe-Fe_3c diagram, it is possible to evolved suitable heat treatment cycles to obtain alloy steels with desire properties. Alloying elements affect the relative stabilities of alpha and gamma iron as also on critical points and carbon content. These elements produce a variety of changes in the behaviour and properties of steel (Rajan, 1988).

Honeycombe (1982) has pointed out that iron binary equilibrium systems falls into four categories (fig.2.2): Open and close γ-field systems, and expanded and contracted γ-field systems. This approach indicates that alloying elements can influence the equilibrium diagram in two ways.

a. By expanding the γ-field, and encouraging the formation of austenite over wider compositional limits. These elements are called γ -stabilizers

b. By contracting the γ-field, and encouraging the formation of ferrite over wider compositional limits. These elements are called α-stabilizers.

Class 1: Open γ-field: To this group belongs the important steel alloying elements nickel and manganese, as well as cobalt and inert metals ruthenium, rhodium. Both nickel and manganese, if added in sufficiently concentration, completely eliminate the bcc α-iron phase, down to room temperature with the γ-iron. So nickel and manganese depress the phase transformation from γ to α to lower temperatures (fig.2.2a) i.e. both Ae_1 and Ae_3 are lowered.

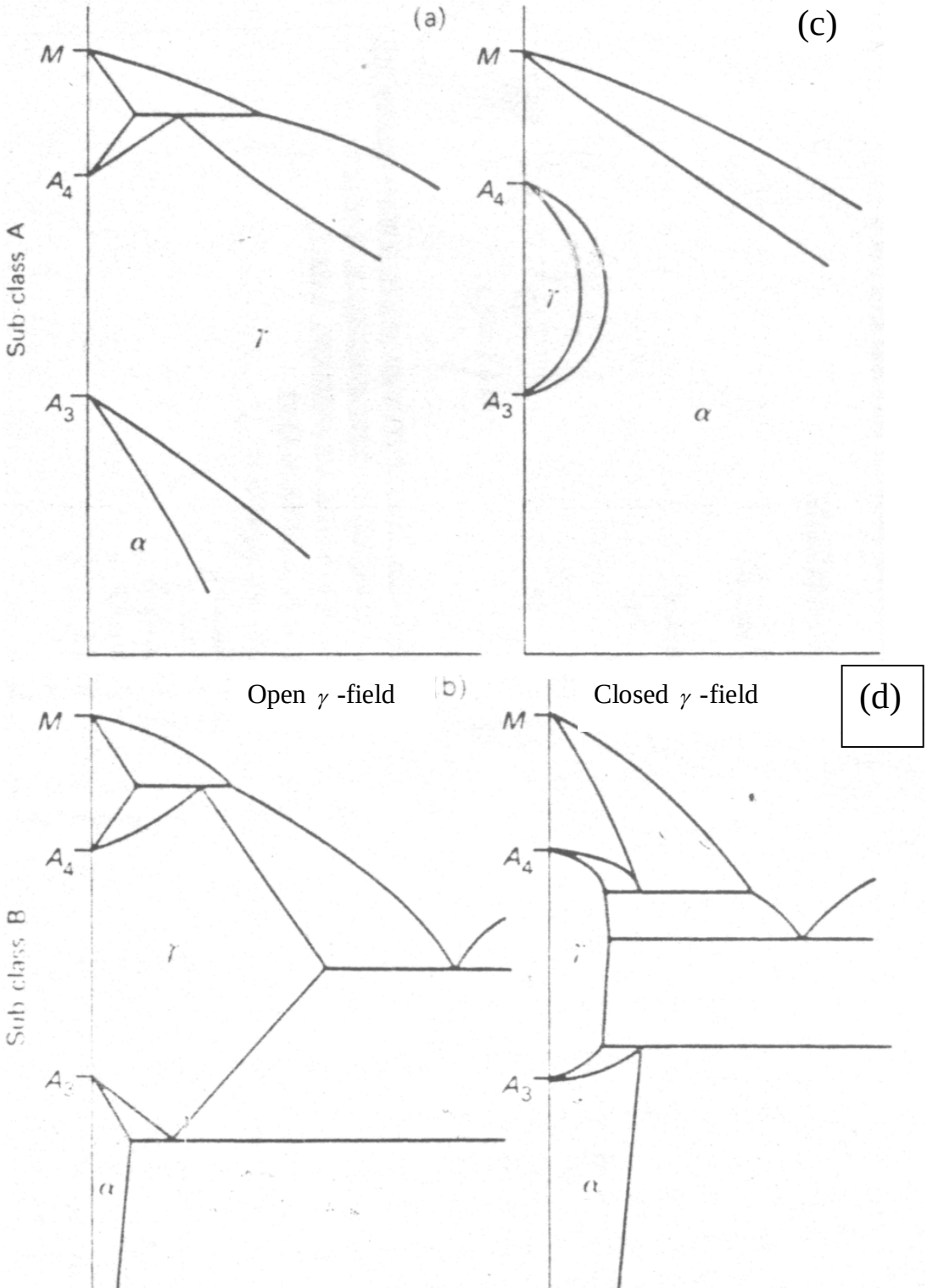
Class 2: Expanded γ-field Carbon and nitrogen are the most important elements in this group. The γ-phase is expanded, but its range of existence is cut short by compound formation (fig.2.2b). Copper, zinc and gold have similar influence. The expansion of γ-field by carbon, and nitrogen, underlies the whole of the heat treatment of steels by allowing formation of a homogeneous solid solution (austenite) containing up to 2.0wt% of carbon or 2.8wt% of nitrogen.

Class 3: Closed γ-field many elements restrict the formation of iron causing the γ-area of the diagram to contract to a small area referred to as the gamma loop (fig.2.2c). This mean that the relevant elements are encouraging the formation of the bcc iron (ferrite); and one result is that the δ- and α-phase fields become continuous. Alloys in which this has taken place are, therefore, not amenable to the normal heat treatments involving cooling through the γ/α phase transformation. Silicon, aluminium, beryllium and phosphorous fall into this

category, together with the strong carbide forming elements, titanium, vanadium, molybdenum and chromium.

Class 4: Contracted α-field Boron is the most significant element of this group, together with the carbide forming elements tantalum, niobium and zirconium. The γ-loop is strongly contracted, but is accompanied by compound formation (Fig.2.2d).

Figure 2.1: The Iron-Iron-Carbide Equilibrium diagram (Westwood, 1996)



Expanded γ -field Contracted γ -field

Figure 2.2: Classification of iron alloy, phase diagrams: a. open γ -field;b. expanded γ -field; c. closed γ -field ; d. contracted γ - field (Honeycombe 1982)

2.5 HEAT TREATMENT OF STEELS

Phase transformation may be wrought in metal alloy systems by vary temperature and composition; however, temperature changes by means of heat treatments are most conveniently utilized to induce phase transformations (Callister, 1997). The microstructure of steel depends upon its prior thermo-mechanical history. For example, a hot-rolled steel can consist of equiaxed ferrite grains and pearlite nodules. If the steel does not have the microstructure desired for its application or for the next process that is to be performed on it, then the microstructure is changed by heat treating. A number of standard processes exist (Honeycombe, 1982). These are briefly discussed in the subsequent sections.

2.5.1 Annealing Heat Treatment

This process is usually employed to make the steel relatively soft and to endow it with good machinability (Hassan, 2001). The microstructure is large-grained ferrite and coarse pearlite. Austenitization is conducted at a high temperature to produce large-grained austenite and to dissolve all the carbides. The steel is then cooled slowly enough to allow the desired microstructure to form by the transformation of the austenite at temperatures near to the Ac line. Alloys steels, especially those containing strong carbides forming

elements need higher annealing temperature than plain steel and other alloy steels (Callister, 1997).

A variety of annealing heat treatments are possible; they are characterized by the changes that are induced which many times are Microstructural and are responsible for the alteration of the mechanical properties.

Spheroidizing annealing: This involves heating to just below lower critical temperature. The cementite lamellae in pearlite colonies tend to become sphere. With prolonged annealing, the spheroidized particles coarsen, becoming fewer in numbers and more widely spaced. For carbon and low alloy structural steels, the optimum machinability corresponds to 50% spheroidized-50% lamellar carbide in the microstructure. For high alloys tool steels, the fully spheroidized condition is preferred.

Processing and Stress- relieving anneal: These terms normally mean some form of treatment in which the steel is heated to and held at a temperature below the Ac_1 line. Subsequent cooling is air cooling (for thick sections) and furnace cooling for thin sections Recovery process occurs, which removes or diminishes residual stresses that might be present. Recrystallization can also be produced in cold-worked parts.

Intercritical annealing: This is also referred to as partial annealing. In this process, steel is heated between the lower critical and upper critical temperature and followed by slow cooling. Both hypoeuctectoid and hypereutectoid steels are subjected to this treatment. A considerable amount of ferrite remains unformed in this process (Rajan et al, 1988).

2.5.2 Normalizing Heat Treatment

Generally, normalizing consists of homogenizing steel for a short time at a temperature corresponding roughly to its hardening temperature and then allowing to

cooling in air (Hassan, 2001). This heat treatment is performed to refine grain size and to obtain distribution that provides a favourable starting point for subsequent heat treatment. (Sinha, 1989).

Normalizing treatment is effective in eliminating precipitation of carbide network at the austenitic grain boundaries of cast steels and formation of fine fully pearlitic matrix. This considerably enhances the mechanical properties of the steels (Thelning, 1984). It is important to recognize that the microstructure produced by normalizing heat treatment will depend on the size of the part; a thin part cools more rapidly in still air than does a thick part.

2.5.3 Hardening

Hardening by quenching involves heating steel to a temperature within or above the critical range, held at that from 15 to 30 minutes per 25mm of cross section, in order to covert it partially or completely to austenite. The cooling is usually done rapidly at a faster rate than the critical rate for complete transformation of austenite present at the quenching temperature to 100% martensite. The basic purpose of hardening in carbon-and alloy steel is to produce a fully martensitic structure with high hardness and strength. High level of surface hardness developed by this process also improves wear resistance. (Avner, 1984). Cooling of steel from the austenitizing temperature is carried out by oil or water quenching. (Kempster, 1984).Anderson et al. (1977) reported that many of the low-alloy steel might be fully hardened with a slower rate of cooling than obtained in a water quench. Also some of the high-alloy steels have a very slow rate of transformation from austenite and can be fully hardened in air. In general, alloy steels are easier to harden than plain carbon steels. Alloy s may be quenched in oil to produces the hardened state (Callister, 1997).

2.6 MICROSTRUCTURE OF FERROUS MARTENSITE

Of the various microstructures that may be produce for a given steel alloy, martensite is the hardest, strong and brittle; it has in fact negligible ductility. These properties are attributed to the effectiveness of the interstitial carbon atom in hindering dislocation motion and to the relatively few slip systems (along which dislocation moves) for body centred tetragonal (BCT) structure (Callister, 1997)

The successful heat-treating of steels to produce a predominantly martensitic microstructure throughout the cross-section depends largely on the grade of steel, the conditions of austenitizing treatment, and the rate of cooling on quenching for hardening. Based on the appropriateness of application manipulation of these parameters, one can come up with the following possible martensitic microstructures (Kenneth, 2002)

(a) 100% martensitic structure (M): ideal parameter selection

(b) Martensite + intermediate phase: inadequate quenching rate

(c) Martensite + retained austenite: existence of alloying elements such as chromium.

(d) Martensite + segregates of carbon atoms: martensitic transformation atom with subsequent natural ageing.

(e) Martensite + peartile structures (p): incomplete austenitization before quenching.

(f) Martensite + pearlite + iron carbide: by hypereutectoid steels and insufficient austenitization time.

Marder (1971) have studied the structure of martensite and found two different types of martensite depending on the composition and temperature at which they are formed; lath and acicular plate martensite. The lath martensite consists of packets of parallel lath with carbon contents up to 0.6% in carbon steel. While plate martensite consists of plates in a

matrix of austenite and is found only in steels containing 0.6%C or more. In the range of 0.6%-1% both types of martensite may occur simultaneously (Thelning, 1984).

Raghavan (1989) wrote that lath martensite is formed at low concentration of carbon and alloying elements and at high temperatures. At higher concentrations of carbon and alloying elements and at lower transformation temperatures, plate martensite formed.

2.7 TEMPERING PROCESS

The as-quench hardened steel consists of martensite and retained austenite. Martensite formed is exceedingly brittle, hard and highly stressed, and retained austenite is also unstable phase and as it changes with time, dimensions may alter. It is therefore necessary to modify the mechanical properties by tempering heat treatment processes before the steel can be put into practical use (Khanna, 2002).

Generally, tempering treatment consists of heating hardened steel in the range of 150-700^{o}C followed by cooling in air or at any desired rate to room temperature. The transformation that occurs and the property changes that are produced, depend upon tempering time and temperature. The general aim of tempering heat-treatment is to raise the toughness, ductility and relieving internal stresses (Kempster, 1984). Because this is achieved at the cost of a reduction in hardness and strength, the process is always a compromise and is selected according to the intended applications. The higher the tempering temperature, the more is the restored ductility and the tougher the steel. (Ause, 2000). The tempering time must be sufficient to allow the desired degree of change to take place. The cooling rate after tempering is not important in the case of plain carbon steels,

but water-cooling is often used. The resulting structures are referred to as tempered martensite (Davies et al, 1983).

2.7.1 Structure of Tempered Martensite

A number of structural changes take place during tempering treatment. These changes include ejection of carbon atom from body centred tetragonal lattice of martensite, isothermal transformation of retained austenite, growth and spheroidization of carbide particles and formation of ferrite carbide mixture (Rajan, 1988). On reheating as-quenched martensite, the tempering takes place in four distinct but overlapping stages, depending on the range of tempering temperature and the type of steel.

Stage 1

The maximum temperature, to which steel is heated, is restricted to about 250^{o}C at this stage. This results in the decomposition of high carbon martensite to low carbon martensite and epsilon (ε) iron carbide.

As far as mechanical properties are concerned, marginal decrease in hardness value takes place at this stage. Strength and toughness improve considerably. Excellent wear resistance and reduced internal stress are the characteristics of hardened steel in this range (Rajan, 1988). But in the higher carbon steels, an increase in the hardness has been observed on tempering in the range 50 – 100^{o}C, which is attributed to precipitation hardening of the martensite by ε-carbide. Low carbon and alloy steels are unlikely to precipitate ε - carbide due to their low carbon content. At the end of stage 1, there is a partial loss of tetragonality in martensite (Honeycombe, 1982).

Stage 2

The second stage of tempering consists of heating steels usually in the temperature range 200- 300^{o}C. During this stage, austenite retained during quenching is decomposed to bainitic ferrite and inter-lath cementite (Honeycombe, 1982).

The transformation of austenite in this stage is accompanied by volumetric expansion. It is likely that the formation of inter-lath cementite and the associated volumetric expansion is responsible for tempered martensite embrittlement, frequently encountered with minimum toughness the 250-350^{o}C ranges. Also, there is further loss of tetragonality in the martensite structures due to further precipitation of more coherent ε-carbide in the original martensite. Transformation in this temperature range will depend on the composition of the alloy steel (Rajan et al, 1988).

Stage 3

During stage-3, there is formation and growth of lath like cementite at the expense of ε-carbide from 300-475^{o}C (Khanna, 2002). Also, the tetragonality of the matrix disappears and it is then, essentially, ferrite, not supersaturated with respect to carbon (Honeycombe, 1982). Ductility and toughness increase by this treatment with a corresponding decrease in hardness and strength.

Stage 4

During the fourth stage of tempering, the cementite particles undergo a coarsening process and essentially lose their crystallographic morphology, becoming spheroidized. The coarsening commences between 300-400^{o}C, while spheroidization takes place increasingly up to 700^{o}C.

Tempering in this stage in the range 350-600^{o}C leads to a mark reduction in the dislocation density and to lath shape ferrite grains. This process, which is essentially one of recovery, is replaced between 600-700^{o}C by recrystallization that result in formation of equi-axed ferrite grains with spheroidal Fe_3C particle in the boundaries and within the grains. The final process is the continued coarsening of the Fe_3C particles and gradual ferrite grain growth, which result in an appreciable loss in strength and hardness with improved ductility and toughness.

2.8 DUAL PHASE STEELS

In recent years, good deal of effort has been put in the development of higher strengths in low carbon steels, without significant loss of formability. Dual phase steels have thus been developed. Low carbon steel sheets are annealed in the intercritical ($\alpha + \gamma$) region of the Fe-C phase diagram and then cool rapidly enough to convert the austenite to lath martensite. The microstructure consists of martensite islands in a ferrite matrix. Typical mechanical properties attained are (Raghavan, 1989): 350Mpa yield strength, 650Mpa tensile strength and 30% Elongation.

Dual-phase (DP) steel can most conveniently be defined as low carbon low alloy ferritic steels with an aggregate microstructure of ferrite and martensite (Bhadeshia, 1980). The attention towards high strength dual-phase (DP) steels has increased significantly in recent years. The automotive industry in particular has shown a lot of interest in DP steels due to the possibility of reducing weight of vehicle and enhancement passenger safety at a very competitive cost. For example, compared to other materials, such as high metal

aluminium and magnesium or plastics and composites, the higher strength steels have, besides weight reduction, the additional advantages, that the processing is similar to that of conventional mild steel. Therefore the overall fabrication costs will be reduced in addition to the benefit of weight saving, which is exactly opposite for all other competitive materials (Hulka, 2002).

2.8.1 Metallurgy and Characteristic of Dual Phase Steel

Dual phase steels are characterized by distribution of a second phase in a ferrite matrix. The second phase, which is often hard martensite particles provide substantial strengthening while the ductile ferrite matrix gives, good formability (Hulka, 2002). The ferrite and martensite dual phase mixture acts like a particle reinforced composite. The tensile strength of the composite is given by a simple rule of mixtures:

$$\delta_{DP} = V_F \delta_F + V_M \delta_M \quad \text{..... (1)}$$

Where

V is the volume fraction of the phases and δ is the tensile strength. The subscripts DP, F, M represent composite dual phase structure and the ferrite and martensite phases respectively (Saikaly et al, 2001)

As martensite is the stronger phase, increase in the volume fraction increases the strength of the dual phase material. Unfortunately increasing the martensite content reduces ductility and for this reason the volume fraction is usually restricted in the range 10-20%, where the martensite remains as discrete isolated islands in an interconnected ferrite matrix.

Further, to maintain high ductility in these steels, it is desirable not only to limit the martensite fraction but also to restrict the carbon content of this phase. At lower carbon

contents (<0.4wt%) lath martensite forms, rather than high carbon twinned martensite. Lath martensite is desirable as it deform to a limited degree and this help to delay void formation at martensite/ferrite interfaces when the material is strained (Pritchard et al, 2004).

According to Hulka (2002), the microstructural configuration of this steels influence the stress-strain curve. The yield strength is determined by the onset of plastic flow in the soft phase, i.e. the ferrite. At this stage, the hard phase is still in the elastic strain region. With higher applied stress the material exhibit high work hardening behaviour according to the rule of mixtures.

2.8.2 Production Routes for DP steel.

To produce a dual phase microstructure, the equilibrium pearlite phase needs to be eliminated, with austenite being encouraged to form martensite by rapid cooling. The simplest method for producing dual phase microstructure is to anneal ferrite/ pearlite steel in the intercritical temperature range. The annealing temperature is controlled within the ferrite plus austenite two-phase region, such that much of the room temperature ferrite phase remains. When the steel is then quenched from the annealing temperature the austenite proportion is sufficiently hardenable to transform to martensite. Controlling the steel chemistry and annealing temperature can adjust both the phase fractions and the properties of martensite (Pritchard, 2004).

Arvedi et al (2004) has also reported that a DP microstructure can be developed directly by controlling both hot rolling process and cooling on the run-out table (ROT). This has been achieved through a combination of process control and new alloy design. The new chemistries are low in carbon and rich in hardenability enhancing elements. The low carbon content encourages ferrite to form at temperatures above that necessary for pearlite

transformation and therefore increase the potential to form martensite (Pritchard, 2004). In this processing route, ferrite is formed from austenite on the run- out table of a hot mill and the post cooling process controlled to allow desired volume fraction of ferrite to form before rapid cooling. The later stage-cooling rate must be sufficient to ″quench″ the remaining austenite to martensite (Militzer et al, 2004). But for hot rolled DP material such an additional post rolling heat treatment would add considerably to the costs, therefore the alloy design and the cooling conditions on the run-out table have been optimized in order to obtain the desired microstructure in the rolled condition (Beynon, 2004).

2.8.3 Mechanical Properties of DP Steels.

There is currently a large interest in producing steels with superior mechanical behaviours provided by dual- phase (Ferrite/martensite) steels. Overall, the mechanical behaviours of intercritically annealed dual phase steels are function of composite morphology, the volume fraction, size, and distribution of constituent phase. These are determined by the composition of the steels, intercritical annealing temperature and soaking time among other factors (Pichler, 2000). Thus DP steels are characterized by superior combination of strength, ductility, continuous yielding, and high working hardening rate. The high formability of dual phase steels is due to low yield to tensile strength ratio with high working hardening rates, promoting uniform elongation. (Pritchard, 2004).

Beynon et al. (2004) has pointed out that during the austenite to martensite transformation, a hard deformation resistance phase is introduced into microstructure and due to the volume expansion that takes place during the transformation of austenite to martensite, mobile dislocation are introduced into the surrounding ferritic constituent

matrix. The mobility of these dislocations is responsible for the continuous yielding behaviour and high initial work hardening rate attributed to dual phase grades.

2.8.4 General Applications of DP Steels

Due to exceptional strength, formability and associated properties of dual phase grades, they are quickly becoming one of most popular and versatile material in today′s automotive industry. Currently they are mostly used in structural applications where they have replaced more conventional materials. The improved formability, capacity to absorb crash energy, and ability to resist fatigue has driven this substitution. They offer a great opportunity where weight material cost saving can be realized by reducing part thickness without lowering strength and formability (Kenneth, 1999).

Recent applications include front and rear rails, crush can, rocker reinforcement, back panels, cross members, bumpers, door intrusion beams, wheel disc, wheel rims. The adoption of these steels in the high strength strip market sector is also of relevant to wire rod customers (Sudhakar, 2000).

2.9 Austenite Formation/Decomposition during Intercritical Annealing.

The formation of austenite has been widely investigated over the last century albeit not as extensively as austenite decomposition. What appears to be the first account that austenite forms as a nucleation and growth process from ferrite-cementite aggregates upon heating can be traced to Arnold and McWilliams (Huang et al, 2004).Huang et al, investigated austenite formation in 1.5wt % Mn steels containing 0.06 to 0.20wt % carbon with ferrite-pearlite microstructure. They concluded that austenite formation can be

separated into three stages: (1) Very rapid transformation of austenite into pearlite until complete pearlite formation. (2) Slower growth of austenite into ferrite primarily controlled by carbon diffusion in austenite, and (3) Very slow equilibration of austenite and ferrite controlled by manganese diffusion in austenite.

Again, alloying additions play a critical role in the process of austenite formation and decomposition. For dual-phase steels, cooling to room temperature produces a microstructure consisting of ferrite and martensite. The cooling path and composition of these steels must be carefully optimized to minimize transformation of austenite back to ferrite or the pearlite or bainite reactions. This is usually facilitated by alloy addition such as Si, Mn, Mo, or Cr, which increases the hardenability of the austenite to form martensite. The mechanical properties of the dual- phase steels are primarily dependent on the volume fraction, carbon concentration, and distribution of the final transformation products, which inherit the composition and distribution of the parent austenite phase. Therefore, the austenite condition at the intercritical temperature is important for the evolution of the final microstructure of the steel and consequently its mechanical properties (Huang, 2004).

CHAPTER THREE

3.0 EXPERIMENTAL PROCEDURE

3.1.0 Materials and Equipment

3.1.1 Materials

Commercial microalloyed steel (in the hot-rolled condition) supplied by SCC Pipe Manufacturing Ltd, Abuja was used for this investigation. The as-received steel blank was in the form of 600×500×15mm in size. The chemical composition of which is given in table 3.1.

Table 3.1: Chemical Compositions of the Steel Used.

%C	%Mn	%Si	%P	%S	%Mo	%V	%Ti	%Al
0.22	1.20	0.25	0.025	0.025	0.001	0.004	0.002	0.051

3.1.2 Equipment

The equipment used for this research are; Muffle furnace with a maximum temperature of 1600^{o}C, Brine (quechant), SAE 40 Engine Oil (quechant), Grinding and Polishing Machine, Optical Metallurgical Microscope with an in-built camera, Lathe turning machine, Vickers hardness tester, Monsanto type W-tensiometer , Izod impact testing machine.

3.2.0 Methodology

In this work, an intermediate-quench (IQ) heat treatment technique was applied to develop high martensite dual phase (HMDP) steel from the microalloyed steel. The steel specimens were initially hardened in iced brine (-8^{o}C), after normalizing treatment. This was followed by intercritical treatments at various intercritical annealing temperatures between 730^{o}C and 810^{o}C at interval of 20^{o}C and holding time from 30-90minutes for each of the temperature regimes to obtain a series of high martensite dual phase in austenite plus ferrite region. Some of the specimens in the as-quenched and the HMDP conditions were subsequently tempered at various tempering temperatures between 250 and 550^{o}C for 1hour. Mechanical properties tests were carried out on the heat-treated samples to examine the followings:

a) The effects of variation of the intercritical annealing temperature and time on the microstructure and mechanical properties of the developed HMDP steels.

b) The mechanical properties of conventionally quenched and tempered specimens and IQ-treated steels.

c) The effect of tempering heat treatments on the microstructure and mechanical properties of HMDP specimens.

Metallographic analysis was carried out for each of the heat-treated conditions.

3.3.0 Heat Treatment Operations

The heat treatment operations were carried out at the heat treatment laboratory of Obafemi Awolowo University, Ile-Ife. The workshop is equipped with heat treatment facilities, which were found very adequate for this investigation. All test pieces heat-treated were packed in a container with cast iron chips mixed with pulverized charcoal to prevent decarburisation and oxidation of the samples.

3.3.1 Normalizing.

125 test specimens were initially normalized by heating to austenitizing temperature of 950^{o}C, soaked for one hour and then cooled in air. Normalizing heat treatments were carried out as pre-hardening treatments so as to restore their original condition and to induce homogeneity in their structure (after machining operation). The normalized microstructures served as the starting point phases for this investigation.

3.3.2 Intercritical annealing treatments

Ferrite-martensite dual phase microstructures were developed by intermediate quench heat treatment, the schedule of which is schematically shown in figure 3.1. The heat treatment consisted of a double quench operation:

(a) 120 specimens were first soaked at austenitizing temperature of 920^{o}C in a muffle furnace for 30minutes and then quenched in 9% iced brine solution (-8^{o}C) to get a fully martensitic structure.

(b) 95 brine-quenched specimens were then held individually at different intercritical temperatures of 730^{o}C, 750^{o}C, 770^{o}C, 790^{o}C and 810^{o}C for 30, 60 and 90 minutes and were finally quenched in engine oil at room temperature to obtain dual phase structures of ferrite and martensite.

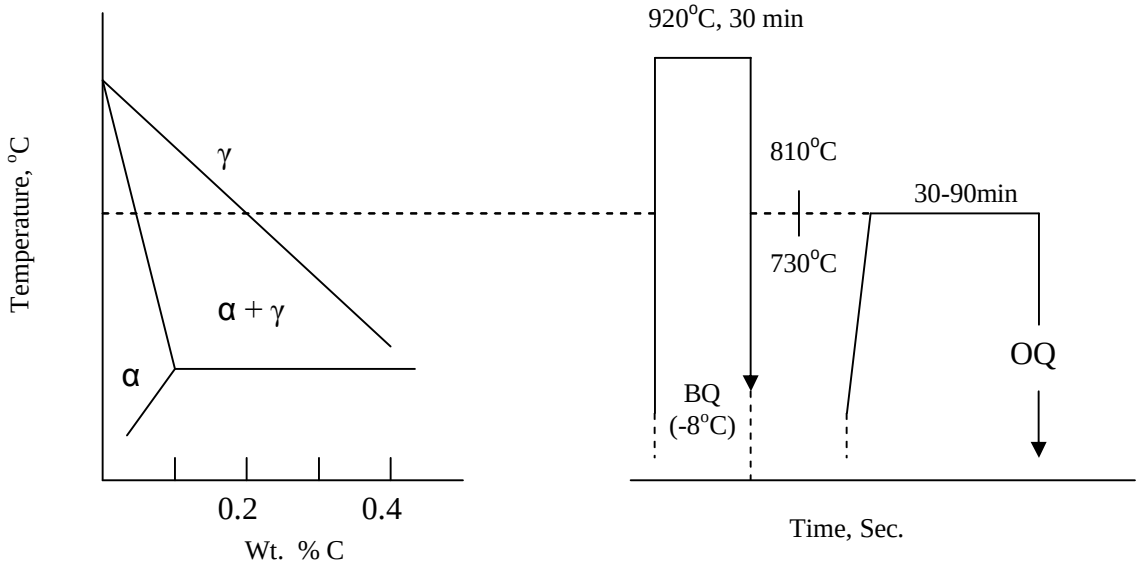


Figure 3.1: Schematic representation of heat-treatment schedules for intermediate quench treatment

3.3.3 Tempering

For the purpose of comparison, 40 test specimens for brine-quenched and HMDP-treated steel specimens were tempered at different temperatures ranging from 250-550^{o}C at interval of 100^{o}C for 60minutes followed by air-cooling.

3.4.0 Mechanical Tests

3.4.1 Tensile properties test

The effect of various heat treatment conditions on the tensile properties was studied. The yield and maximum loads were recorded directly from the machine. After fracture, the two broken end of the specimens were fitted together and final gauge length as well as the smallest diameter of the local neck were measured. The readings thus obtained were used in the calculation of the following properties: Yield strength, ultimate tensile strength, and percentage elongation.

3.4.2 Hardness Test Measurement

The hardness values of the sample subjected to various heat treatments were obtained using an Avery Vickers hardness tester. The samples were prepared with flat and smooth surfaces. A diamond indenter was used to indent the surface of the test specimen by the application of static load of 30kgf, which was maintained for fifteen minutes. Both diagonals of the impression were measured using a lower power graduated microscope. The mean value of the diagonal length was used in the determination of the hardness number as shown in the formula below.

$$H_D = \frac{1.8544F}{D^2} \quad \text{.....(2)}$$

Where F is the applied load in kg, and D is the mean diagonal length in mm.

The procedure was repeated for 3 different points of the test piece and the average of these was recorded.

3.4.3 The Izod Impact Test

The impact test was carried out according to standard of 11.4mm diameter circular section with a 2mm deep V-notch as specified in BSS.131. Before the test, the pendulum was set to a potential energy position of 162.75J. The test specimen was then gripped vertically in a vice, the trigger was released and the registering pointer of the quadrant scale indicated the energy absorbed in joule by the specimen. The energy absorbed in breaking the samples were recorded and repeated for other test pieces.

3.5.0 Metallography

All the heat-treated specimens were prepared for optical microscopic investigation using the standard metallographic techniques. The specimens were ground on a water-lubricated grinding machine with silicon carbide abrasive papers of grades 240, 320,400, and 600 grit sizes. Polishing was carried out on two 15cm rotating discs of a METASERV universal polishing machine. Rough polishing was done with synthetic velvet polishing clothes impregnated with 1-micron Alumina paste. Final polishing was carried out with 0.5-micron chromic oxide polishing powder. The specimens were then etched with 2% Nital solution.

The microscopic examinations were carried out on a Wild M50 optical metallurgical microscope and the microstructures obtained were recorded with the aid of in-built camera.

CHAPTER FOUR

4.0 RESULTS

4.1.0 Microstructure of Heat-treated HMDP Steel Specimens

The results of microstructures obtained for the as-received and heat-treated HMDP steel specimens from this research work are shown in micrographs A1-A26.

4.1.1 As-received structures.

The as-received structure of the microalloyed steel (as-rolled condition) was ferritic-pearlitic as expected and is shown in micrograph A1.

4.1.2 Normalized structures

The normalized structure of the as-received specimen is pearlitic. According to Hassan 2002, the pre-hardening normalizing treatment is normally carried out to ensure conditioning and homogenization of austenite matrix prior to quenching. Hence, normalizing of the microalloyed steel at 950^0C for 30 minutes prior to hardening ensure fine pearlite structure as seen in micrograph A2. All the as-received samples were initially normalized.

4.1.3 As-quenched structures

The microstructure of the steel specimens quenched in iced brine was found to be fully martensitic as expected and is shown in micrograph A3. The structure is mainly martensite.

4.1.4 Intermediate Quenched Structures.

Micrographs A4 to A18 showed the structures of high martensite dual phase (HMDP) steel specimens produced by intermediate quenching process. The microstructures of the specimen were found to contain uniform distribution of lath martensite and ferrite structures. The distribution of constituent phases is similar to those reported for conventional DP steels (Raghavan, 1997).

4.1.5 Tempered structures

As shown in Micrographs A19-A22, the microstructures of as quenched and tempered specimens between 250-550^{o}C consist of tempered martensite as expected. Micrographs A23-A26 reveal the microstructure of HMDP steel specimens intercritically annealed at 770^{o}C for 60minutes and tempered at different tempering temperatures range of 250-550^{o}C. The structure developed consists of ferrite and tempered martensite. This tempered martensite with associated ferrite

structure is responsible for the unique combination of strength, ductility, and toughness that occurred in the tempered dual phase steel.

4.2.0 Comparative Mechanical Properties.

The mechanical properties of both as-received and heat-treated conditions are tabulated in tables 1-10 (appendix B). The various graphs of the mechanical properties obtained for different heat-treated conditions are shown in figures 4.1-4.10.

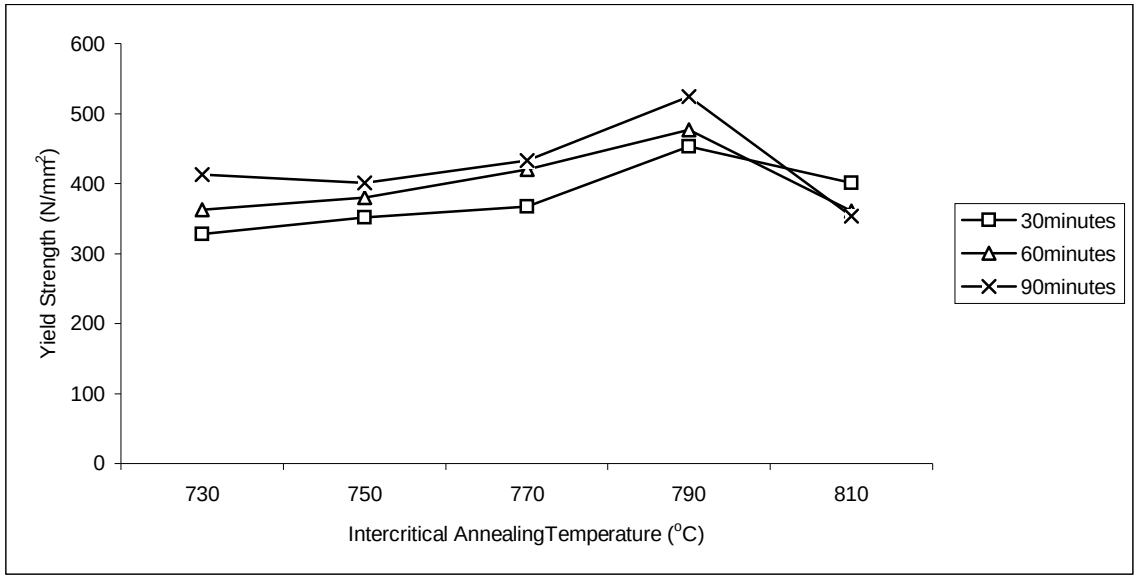


Figure 4.1: Yield strength of intercritically annealed HMDP steel at various intercritical temperatures and times.

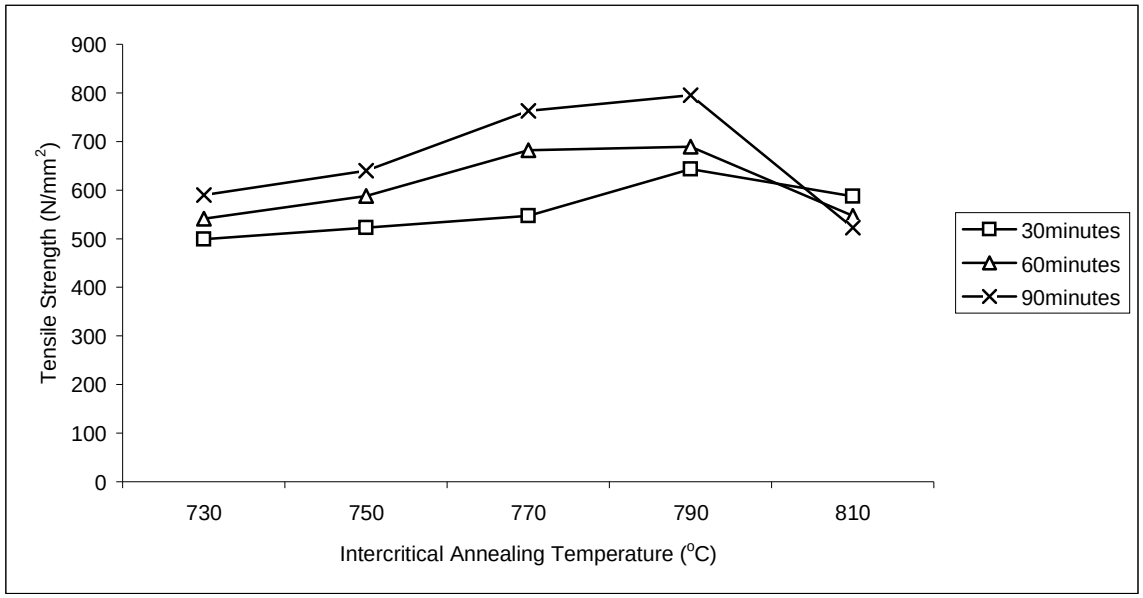


Figure 4.2: Tensile strength of intercritically annealed HMDP steel at various intercritical temperatures and times.

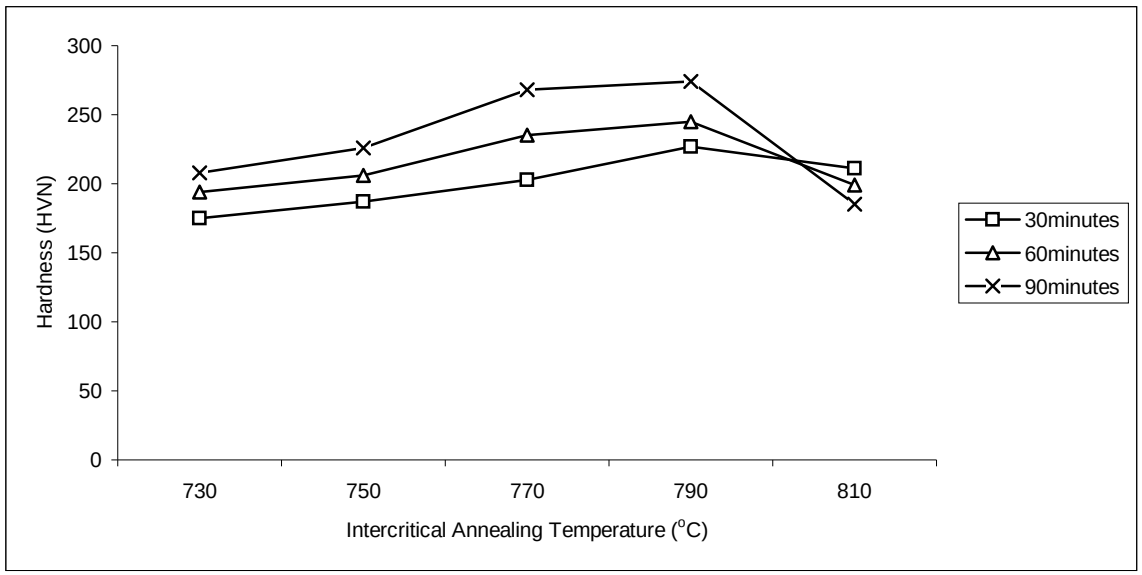


Figure 4.3: Hardness of intercritically annealed HMDP steel at various intercritical temperatures and times.

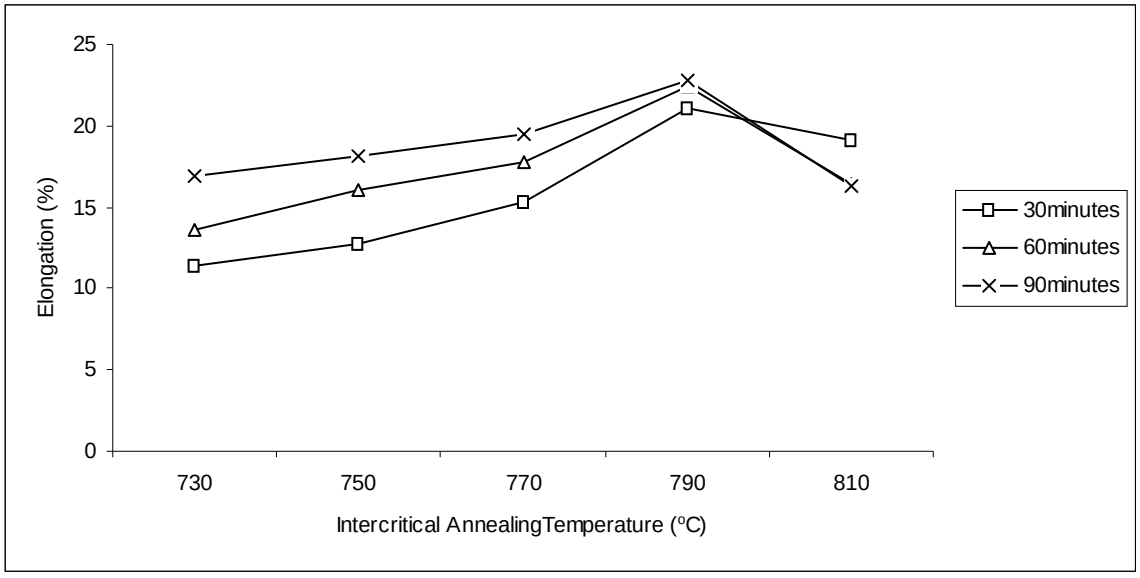


Figure 4.4: Percentage elongation of intercritically annealed HMDP steel at various intercritical temperatures and times.

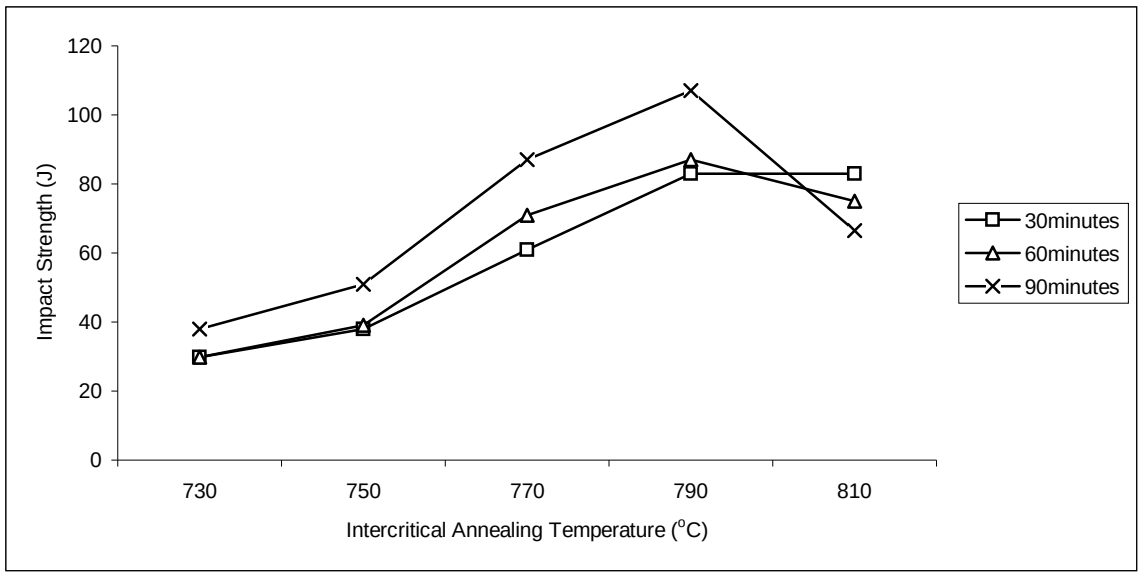


Figure 4.5: Impact strength of intercritically annealed HMDP steel at various intercritical temperatures and times.

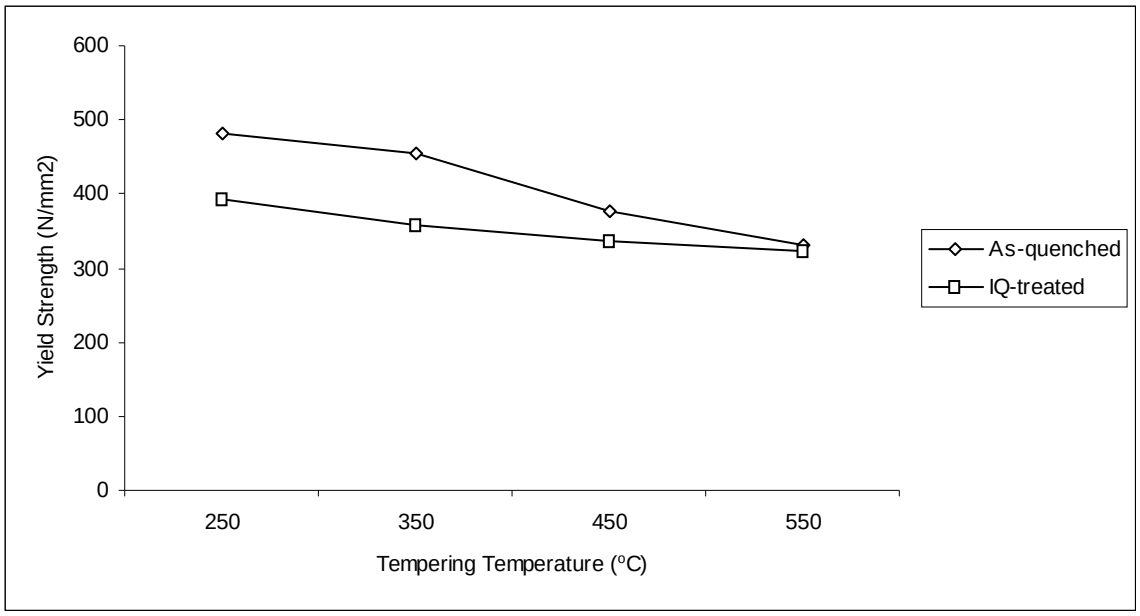


Figure 4.6: Effect of variation of tempering temperatures on the yield strength of as-quenched and intercritically annealed IQ-treated HMDP steels.

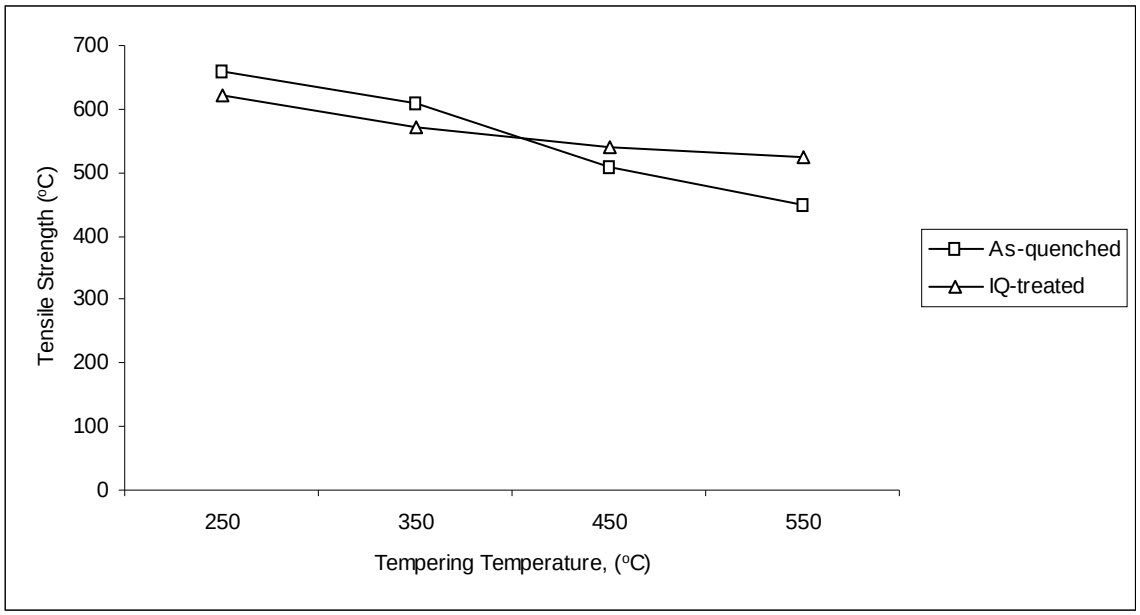


Figure 4.7: Effect of variation of tempering temperatures on the tensile strength of as-quenched and intercritically annealed HMDP steels.

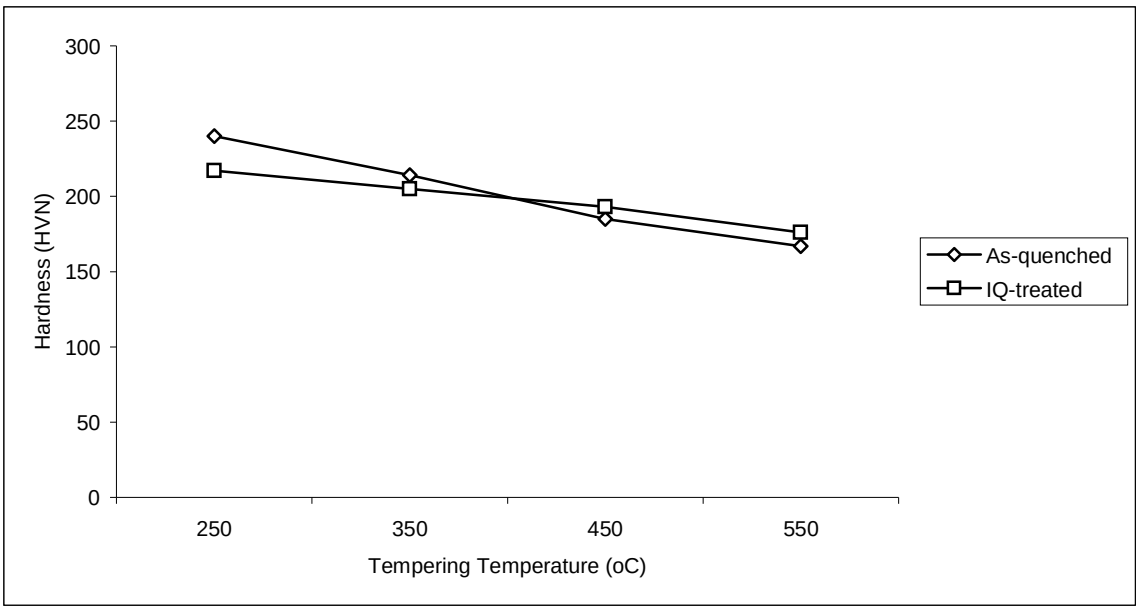


Figure 4.8: Effect of variation of tempering temperatures on the hardness of as-quenched and intercritically annealed IQ-treated HMDP steels.

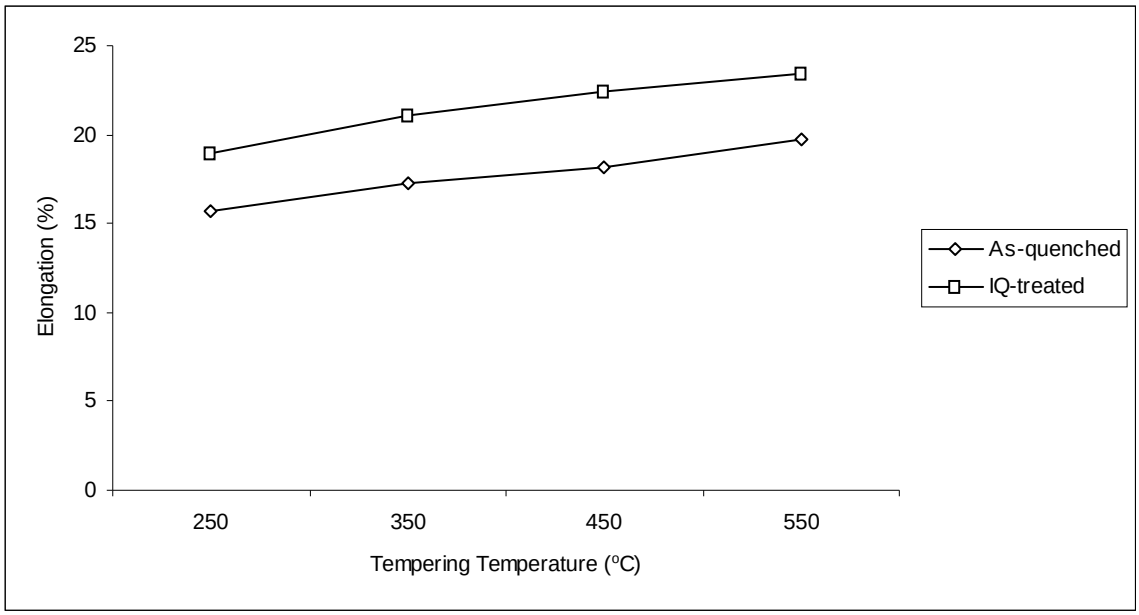


Figure 4.9: Effect of variation of tempering temperatures on the percentage elongation of as-quenched and intercritically annealed IQ-treated HMDP steels.

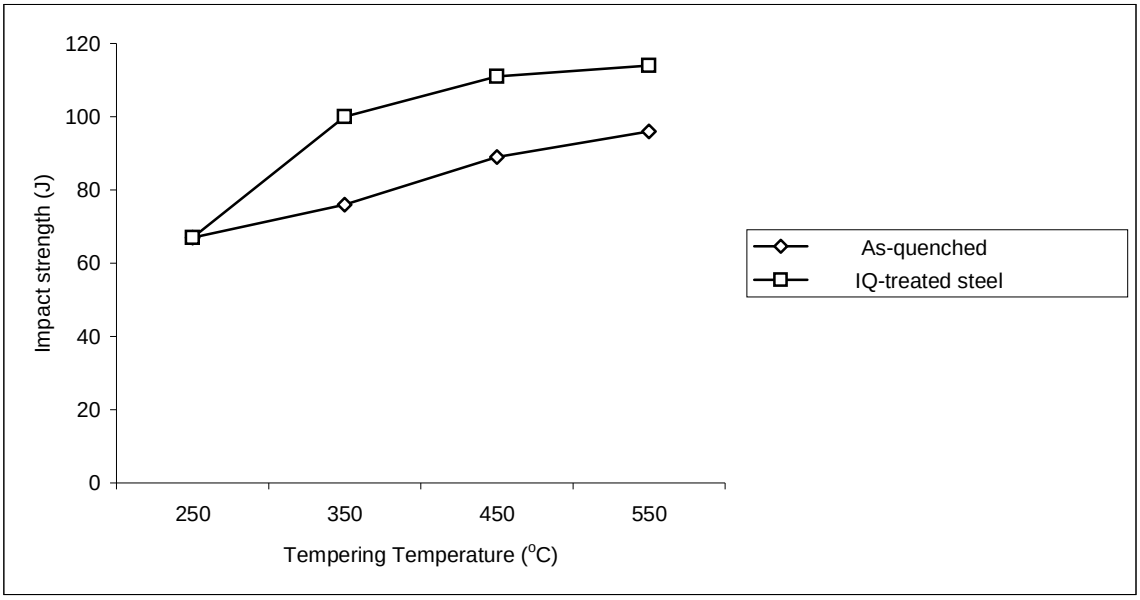


Figure 4.10: Effect of variation of tempering temperatures on the impact strength of as-quenched and intercritically annealed IQ-treated HMDP steels.

CHAPTER FIVE

5.0 DISCUSSION

5.1 Microstructure of Intermediate Quenched-treated Steel

Microstructures of the IQ-treated HMDP steel are shown in micrographs A4-A18. The microstructures of HMDP steel specimens obtained at low intercritical annealing (ICA) temperatures (micrographs A4-A8) showed fine particles of carbide precipitates (black dots). These precipitates were reported to form during the reheating process to the ICA temperature, wherein the quenched martensite get tempered and partly dissociated into ferrite plus carbide, and then revert to the ferrite, austenite, and undissolved carbide upon reaching the intercritical

temperature (Bag et al, 2001). Upon rapid quenching of the steel from the intercritical temperature, at various holding time, austenite transforms to martensite producing ferrite-martensite dual phase steel microstructure. The amount of these carbides decreases as the intercritical temperature increases and such carbides are not present in the HMDP steel specimens heat-treated between 770 and 810^{o}C, as shown in micrographs A10-A18. At ICA temperature from 770 to790^{o}C, the microstructure developed revealed finer ferrite and martensite (see micrographs A10-A14). For all the IQ-treated HMDP steel specimens, the micrographs show martensite layers thickening on the ferrite grain boundary as the intercritical time increases from 30 through 90 minutes for each of the intercritical temperatures. According to Barros et al. (2003), this is a result of the increase in the martensite content with intercritical treatment time.

5.2 Microstructure of Tempered Steel

Microstructures of brine-quenched and tempered steel specimens are shown in micrographs A19-A22. As expected, the microstructures obtained were found to consist of tempered martensite when the as-quenched specimens were tempered at four different temperatures, 250, 350, 450, 550^{o}C for 1 hour. Micrographs A23-A26 revealed typical microstructure of HMDP steel specimen intercritically annealed at 770^{o}C for 60 minutes and subsequently subjected to tempering treatment in the tempering temperature range of 250-550^{o}C. Result of microstructural details for the tempered HMDP samples indicates that the dual phase sample has undergone microstructural changes on tempering that are akin to those of fully martensite low carbon steel. The microstructure of the IQ-treated HMDP steel specimen tempered

at 250^{o}C was obviously different from the HMDP specimen (micrograph A11) without tempering. Micrograph A23 shows that specimen tempered at 250^{o}C consists of ferrite and tempered martensite. A further increase in the tempering temperature beyond 350^{o}C does not show any significant variation in the microstructure using the optical metallurgical microscope.

5.3 Mechanical Properties of Intermediate Quench-treated Steel

Tables B2, B3 and B4 summarized the influence of intercritical annealing treatment temperatures on the tensile properties of IQ-treated HMDP steel specimens. The yield strength, tensile strength and % elongation readily change with increase in the intercritical annealing temperatures. It was found that the HMDP steel showed continuous yield behaviour and hence its 0.2 % proof strength was taken as the yield strength. This behaviour can be attributed to introduction of mobile dislocations into ferritic matrix during transformation of austenite to martensite after intercritical annealing treatment in the dual phase steel (Beynon et al, 2004). The presence of these mobile dislocation are responsible for the absence of luder bands phenomenon in HMDP steel as usually observed in the low carbon steels. The yield strength, tensile strength and percentage elongation were plotted against intercritical temperatures shown in figures 4.1, 4.2 and 4.4 respectively. As seen from the figures, the yield strength, tensile strength and elongation increased with increasing intercritical annealing temperature peaking at 790^{o}C and then decreased with a further increase in the intercritical temperature. Sudhakar et al. (2000) has reported that the increase in strength is mainly attributed to the load bearing capability of martensite in the HMDP structures and that the deformation in

the ferrite phase is constrained by martensite. It is further reported by Speich (1981) that the yield strength and ultimate tensile strength of DP steels are linearly related to the martensite content, which increases with intercritical annealing temperatures. Also, Bag et al. (2001) have observed from their previous work that HMDP steel with finer microstructure exhibits higher ductility. On comparison, the result of the % elongation obtained in this investigation appears to agree with this finding.

The average values of hardness and impact energy recorded for IQ-treated HMDP steel specimens (Tables B5&B6) were also plotted against the intercritical annealing temperatures as shown in figures 4.3 and 4.5. Both values significantly increase with increasing intercritical temperatures and then decreases. The trend is similar to the one obtained for the tensile properties of the HMDP steels.

Previous observation has pointed out that the lower strength, ductility and toughness encountered at lower intercritical temperature is attributed to the relatively coarser ferrite with carbide precipitates whereas for lower strength at higher intercritical temperature is due to coarser martensite (Bag et al, 2001). Hence the optimum value obtained for all the mechanical properties investigated occurred at intercritical temperature of 790^{o}C, which corresponds to the microstructure comprised mostly of finer ferrite and martensite. It therefore implied that good combination of higher strength, ductility and toughness are associated with finer martensite and precipitate-free ferrite that permit ease of dislocation flow owing to the absence of barriers such as the undissolved carbides.

Generally, the mechanical properties of IQ-treated HMDP specimens increase with the intercritical time from 30 to 90minutes for each of the intercritical temperatures (Fig. 4.1-4.5) except at higher intercritical temperature where further

increase to about 810^{o}C gave decrease in the properties of the steel as time increases. This is probably due to the continued coarsening of martensite phase (Barros et al., 2003).

5.4 Mechanical Properties of Tempered Steel

The yield strength, tensile strength, hardness, elongation and impact strength of as-quenched and tempered steel specimens are plotted against tempering temperature as shown in figures 4.6-4.10 respectively. The results show that the yield strength, tensile strength and hardness significantly decrease with an increase in tempering temperature. On the other hand, both the elongation and impact strength increases with an increase in tempering temperature from 250-550^{o}C.

The values of yield strength, tensile strength and hardness of IQ-treated HMDP steel specimens are plotted against the tempering temperatures as shown in figures 4.6-4.8 respectively. Unlike IQ-treated sample, the samples show yield point behaviour when they were tempered. The yield strength of tempered HMDP specimens are slightly lower than that of the IQ-treated one but the yield strength does not show significant variation with tempering temperature (Fig.4.6). The tensile strength and hardness values consistently decrease with an increase in the tempering temperature. Furthermore, the graphs of percentage elongation and impact strength as a function of tempering temperature are shown figures 4.8 and 4.9. Both the elongation and impact strength increases with an increase in tempering temperature and slightly greater than that of the IQ-treated condition.

The absence of discontinuous yielding in the ferrite-martensite DP steels has been explained in terms of both the residual stresses and high dislocation density

surrounding the martensite Islands. As expected, when the HMDP steel sample was tempered at 250^{o}C, the residual stresses and high dislocation density surrounding the martensite islands is reduced as the ε-iron carbides start to precipitate and the martensite becomes less tetragonal. At a tempering of about 350^{o}C the ε-iron carbides is replaced by cementite and the martensite further loses its tetragonality (Honeycombe, 1995). The matrix structure of the tempered martensite transforms to body centre cubic (b.c.c) and the carbon concentration of the matrix approaches that of ferrite. Furthermore, the strength difference between the ferrite and the tempered martensite is reduced (Fang 2002). Meanwhile, ultimate tensile strength and hardness decreases and the elongation increases.

When the tempering temperature is higher than 350^{o}C, the cementite in the tempered martensite coarsens and spheroidises. With further increase in the tempering temperature the carbon concentration of the matrix in the tempered martensite decreases due to diffusion of carbon atoms to the cementite. Hence the ultimate tensile strength of the tempered ferrite-martensite decreases and its ductility increases.

CHAPTER SIX

6.0 CONCLUSIONS AND RECOMMENDATIONS

6.1.0 CONCLUSIONS

From the results of this research work, the following conclusions can be drawn.

1) The as-received steel specimens respond well to heat treatment, both during intercritical annealing and tempering treatments.
2) The yield strength, tensile strength, % elongation and impact toughness of the developed high martensite dual phase steel increased to a maximum value of 524N/mm^2, 795N/mm^2, 22.8%, and 107joules respectively when intercritically

annealed at temperature of 790^{o}C. This is attributed to the finer distribution of ferrite and martensite constituents as well as absence of carbide precipitate in this region. The investigation therefore shows that intercrititial annealing treatment accompanied with grain refinement is strikingly successful in improving the mechanical properties of the developed HMDP steels.

3) Continuous yielding was found to occur for the steel samples intercritically annealed in the range730^{o}C-810^{o}C. Whereas sharp yield points were obtained for the quenched and tempered steel specimens.

4) Combination of higher toughness, strength and ductility is obtained for HMDP steel. The as-quenched and tempered steels with relatively similar toughness and ductility levels are characterized with lower strength.

5) The mechanical properties of the developed HMDP steel were substantially raised with intercritical annealing time from 30 to 90minutes.

6) A further improvement in ductility and impact toughness with slight drop in strength was obtained for HMDP steel intercritically annealed at 770^{0}C for 60 minutes and tempered at different tempering temperatures range of 250 to 550^{0}C. Whereas a significant drop in strength was obtained for as-quenched and tempered with relatively high toughness and ductility. This clearly indicates that tempered HMDP steel significantly exhibited superior mechanical properties as compared to that of conventionally quenched and tempered specimens. Hence, both HMDP and tempered HMDP steel grades can be successfully employed and proposed to substitute, at a competitive cost, most of the quenched and tempered steel grades in automotive applications where combination of high strength, toughness and ductility is the priority.

7) The strength of tempered HMDP steel does not show remarkable change with tempering temperatures; hence HMDP steels do not necessarily require tempering treatment.

6.2.0 RECOMMENDATIONS

For better understanding and fuller exploitation of the potential of high martensite dual phase steel, as an advanced structural materials for automotive applications, the followings are recommended for further studies;

1) Further research effort should be conducted to investigate other properties such as fatigue, hole expansion, creep and corrosion behaviour of the developed HMDP steel.
2) A comparative study of the effect of other heat-treatment techniques such as step quenching (SQ) towards producing HMDP steel with finer structures should be carried out.

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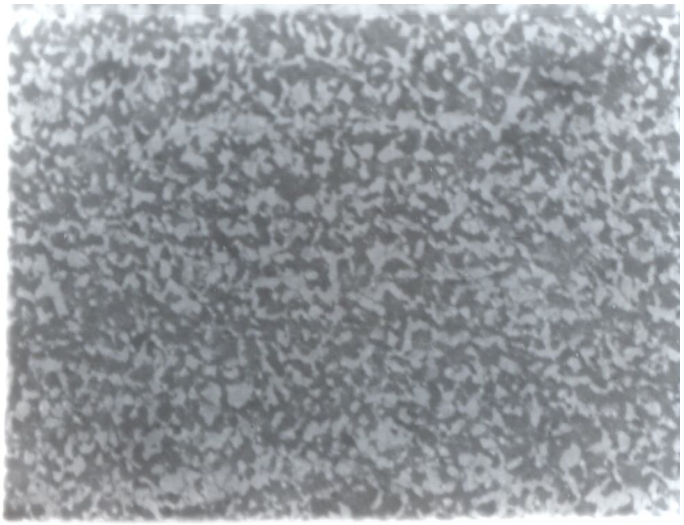
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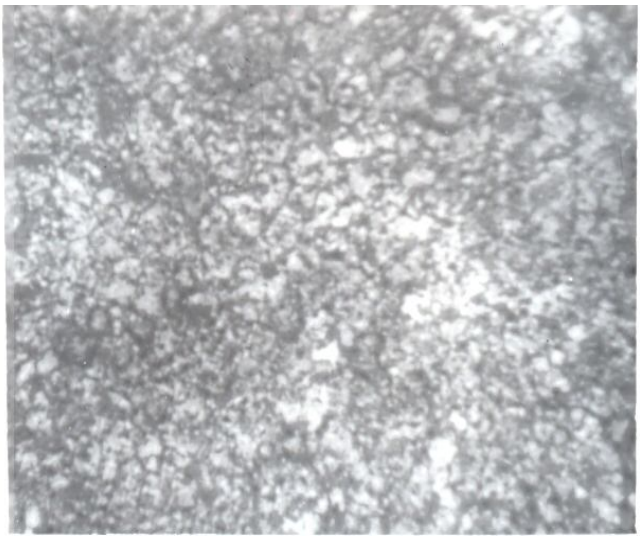
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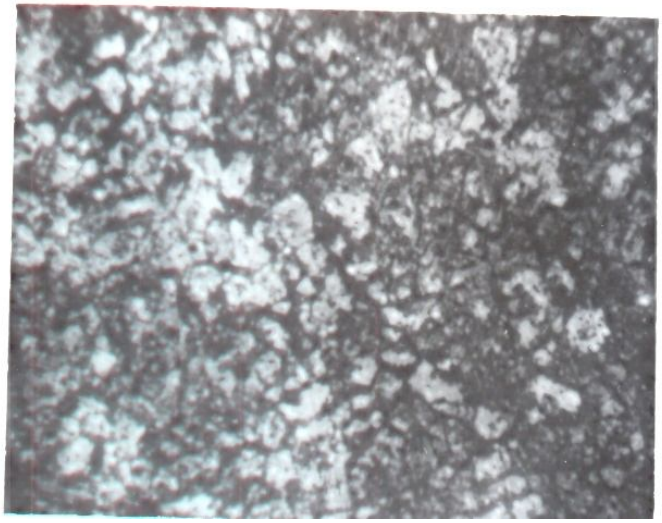
APPENDIX A



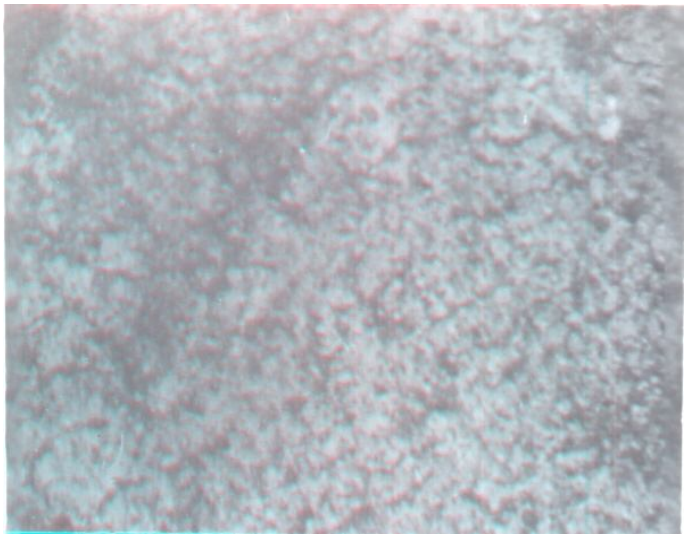
Micrograph A1: Microstructure of as-received microalloyed steel sample.The structure consists of ferrite (light) and pearlites (dark).2% Nital etch. ×100.



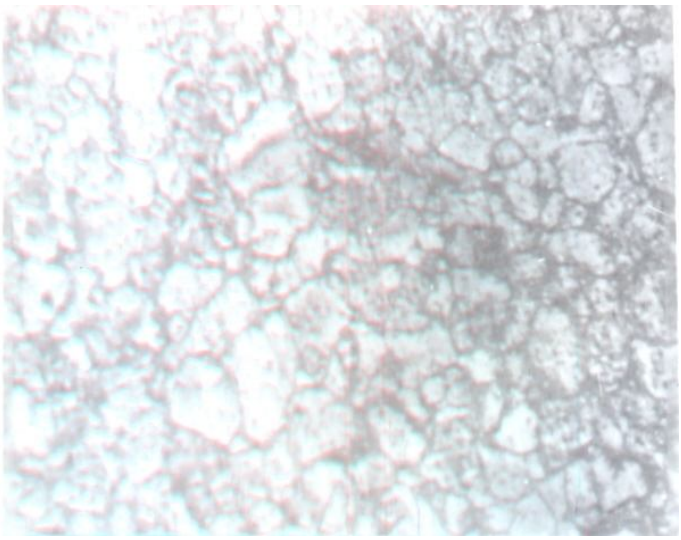
Micrograph A2: Microstructure of normalized steel sample. The structure consist fine ferrite (light) and pearlite (dark). 2% Nital etch.×100.



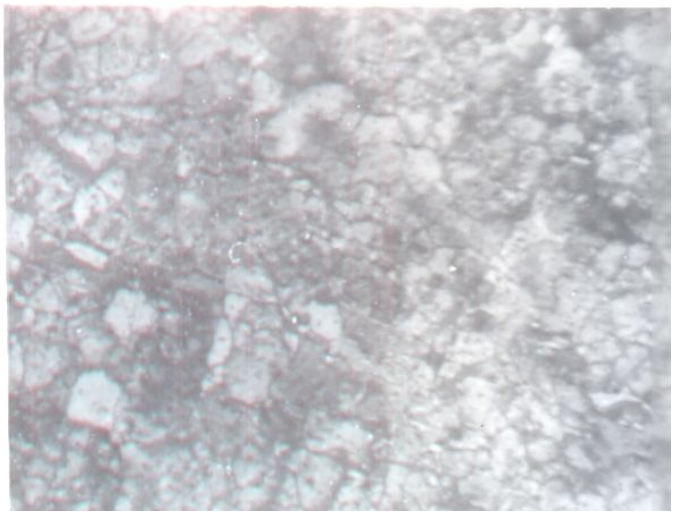
Micrograph A3: Microstructure of as-quenched (iced brine) steel sample structure consisting of coarse martensite (black) and the retained austenite (white). 2% Nital etch.×100.



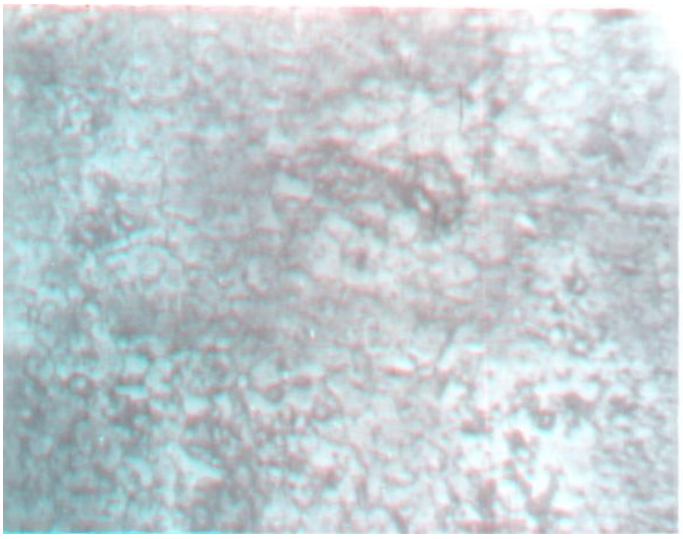
Micrograph A4: Microstructure of IQ-treated HMDP steels intercritically annealed at 730^{o}C for 30minutes showing distribution of martensite (black), undissolved carbide (black dot) and ferrite (white). 2% Nital etch. ×100.



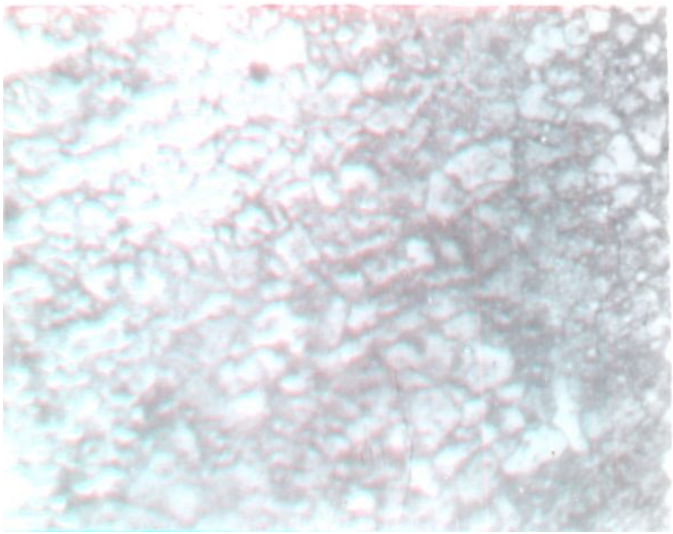
Micrograph A5: Microstructure of IQ-treated HMDP steels intercritically annealed at 730^{o}C for 60minutes showing distribution of martensite (black), undissolved carbide (black dot) and ferrite (white). 2% Nital etch. ×100.



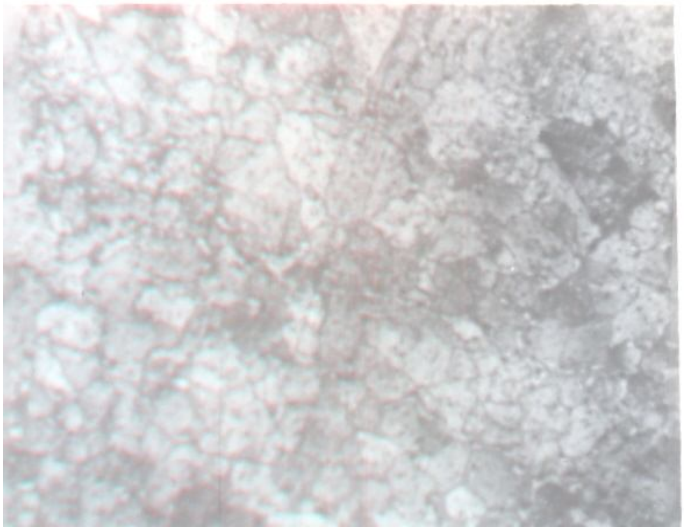
Micrograph A6: Microstructure of IQ-treated HMDP steels intercritically annealed at 730^{o}C for 90minutes showing distribution of martensite (black), undissolved carbide (black dot) and ferrite (white). 2% Nital etch. ×100.



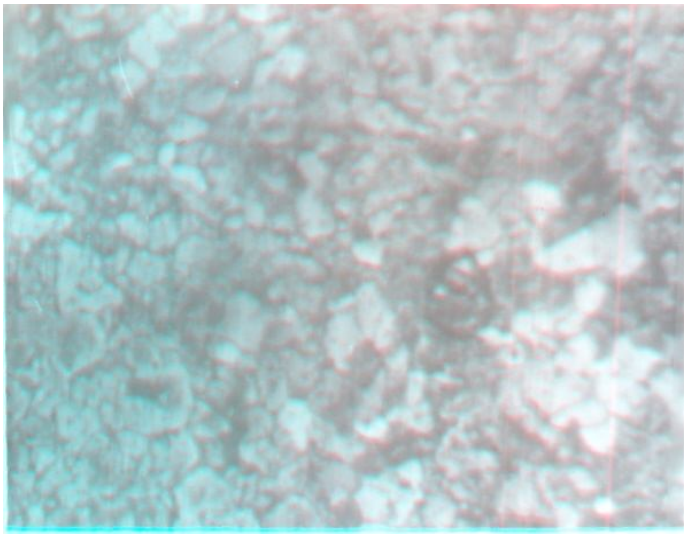
Micrograph A7: Microstructure of IQ-treated HMDP steels intercritically annealed at 750^{o}C for 30minutes showing distribution of martensite (black), undissolved carbide (black dot) and ferrite (white). 2% Nital etch. ×100.



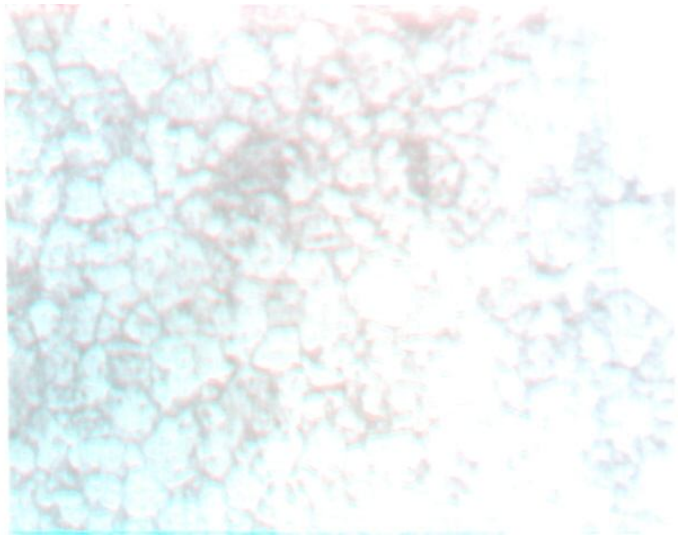
Micrograph A8: Microstructure of IQ-treated HMDP steels intercritically annealed at 750^{o}C for 60minutes showing distribution of martensite (black), undissolved carbide (black dot) and ferrite (white). 2% Nital etch. ×100.



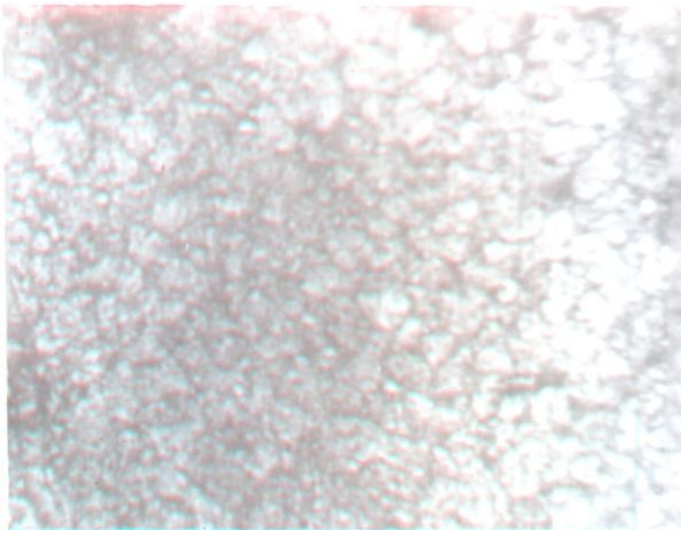
Micrograph A9: Microstructure of IQ-treated HMDP steels intercritically annealed at 750^{o}C for 90minutes showing distribution of martensite (black) and ferrite (white). 2% Nital etch. ×100.



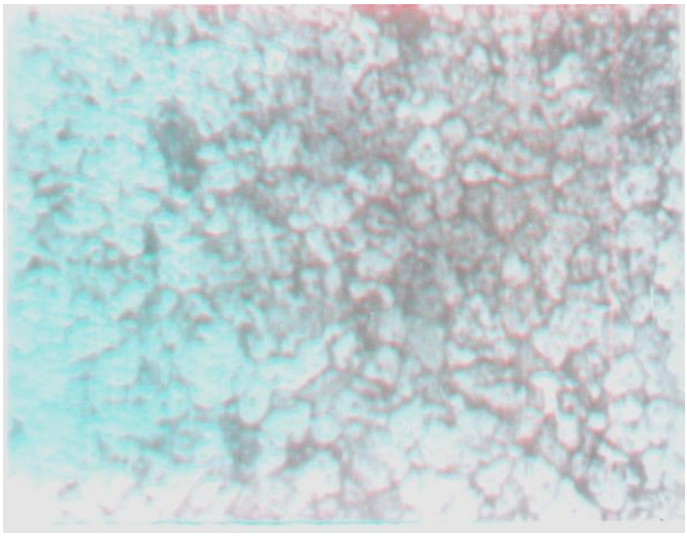
Micrograph A10: Microstructure of IQ-treated HMDP steels intercritically annealed at 770^{o}C for 30minutes showing distribution of martensite (black) and ferrite (white). 2% Nital etch. ×100.



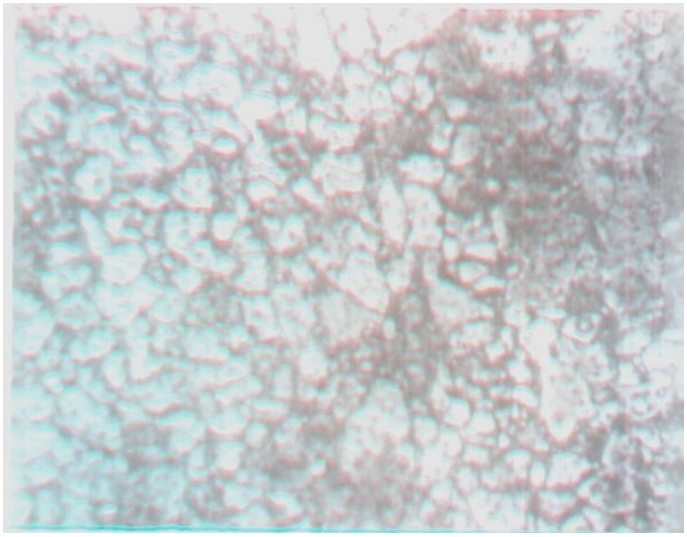
Micrograph A11: Microstructure of IQ-treated HMDP steels intercritically annealed at 770^{o}C for 60minutes showing distribution of martensite (black) and ferrite (white). 2% Nital etch. ×100.



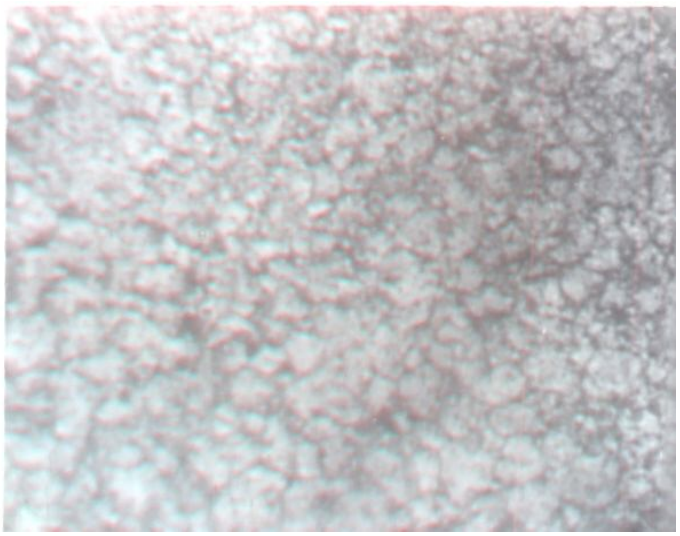
Micrograph A12: Microstructure of IQ-treated HMDP steels intercritically annealed at 770^{o}C for 90minutes showing distribution of martensite (black) and ferrite (white). 2% Nital etch. ×100.



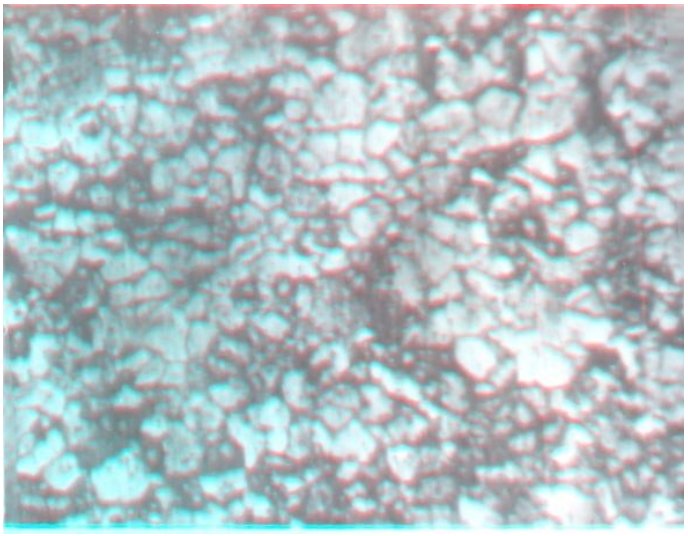
Micrograph A13: Microstructure of IQ-treated DP steels intercritically annealed at 790^{o}C for 30minutes showing distribution of martensite (black) and ferrite (white). 2% Nital etch. ×100.



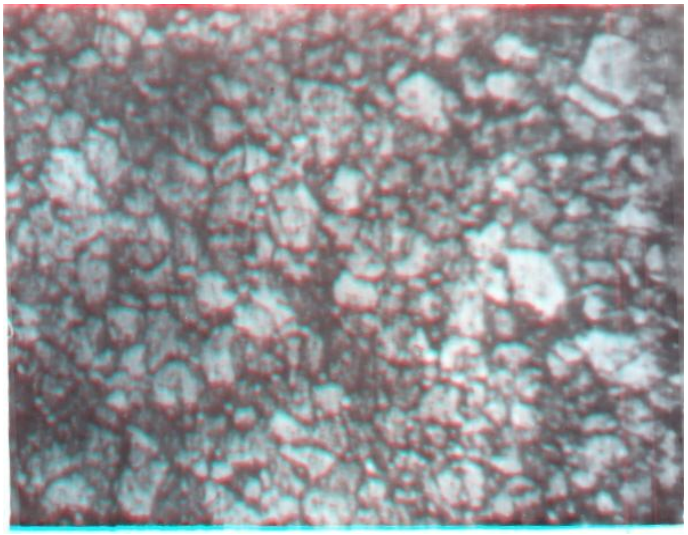
Micrograph A14: Microstructure of IQ-treated HMDP steels intercritically annealed at 790^{o}C for 60minutes showing distribution of martensite (black), and ferrite (white). 2% Nital etch. ×100.



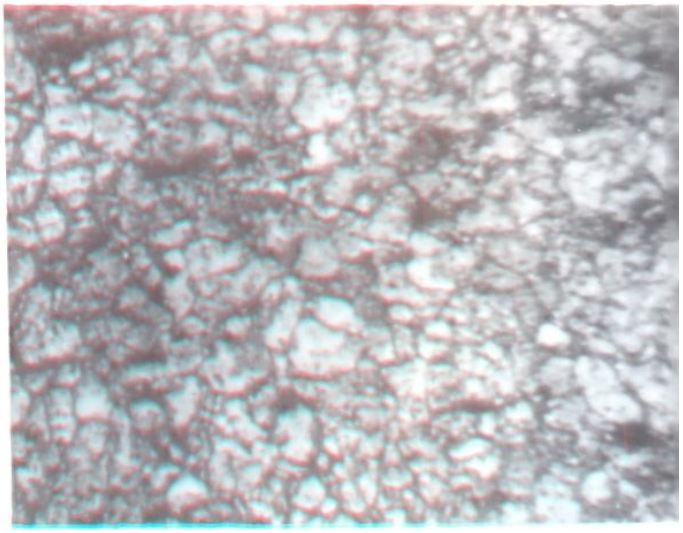
Micrograph A15: Microstructure of IQ-treated HMDP steels intercritically annealed at 790^{o}C for 90minutes showing distribution of martensite (black) and ferrite (white). 2% Nital etch. ×100.



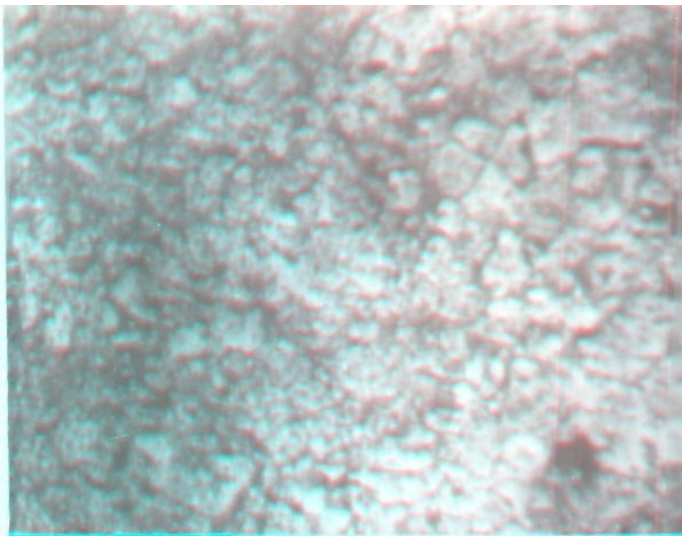
Micrograph A16: Microstructure of IQ-treated HMDP steels intercritically annealed at 810^{o}C for 30minutes showing distribution of martensite (black) and ferrite (white). 2%Nital etch. ×100.



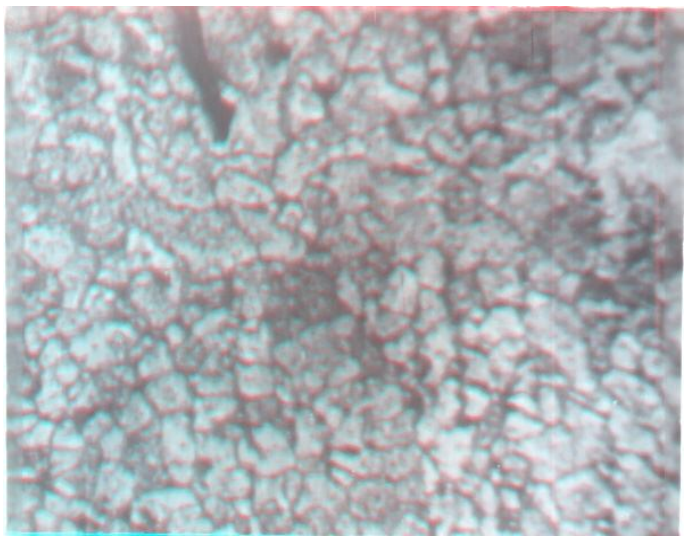
Micrograph A17: Microstructure of IQ-treated HMDP steels intercritically annealed at 810^{o}C for 60minutes showing distribution of martensite (black) and ferrite (white). 2% Nital etch ×100.



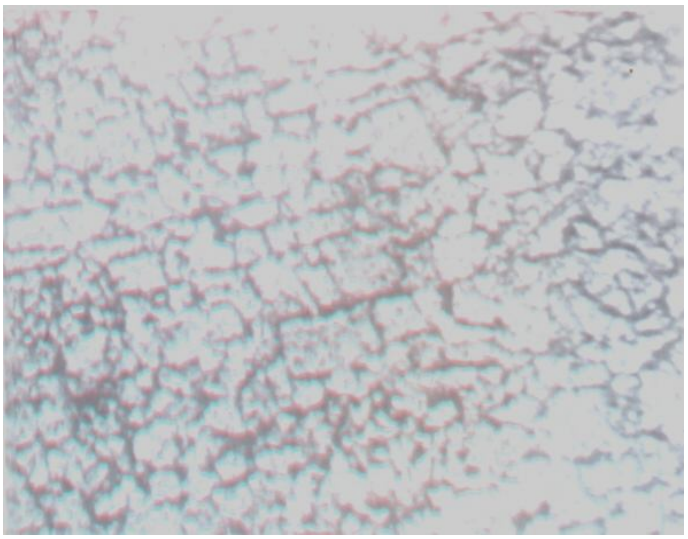
Micrograph A18: Microstructure of IQ-treated HMDP steels intercritically annealed at 810^{o}C for 90minutes showing distribution of martensite (black), and ferrite (white). 2% Nital etch. ×100.



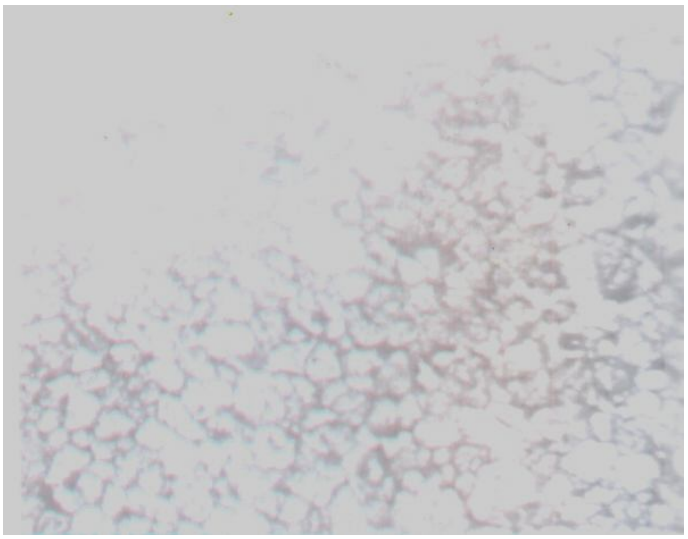
Micrograph A19: Microstructure of as-quenched and tempered steel sample at 250^{o}C for 1hour. 2%Nital etch. ×100.



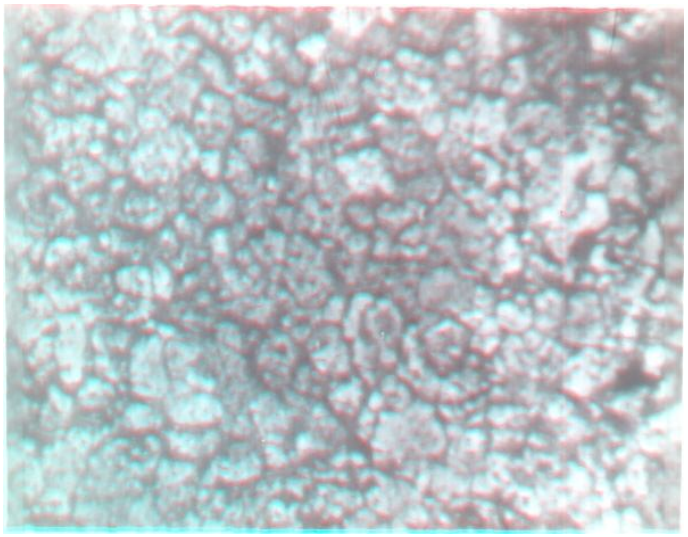
Micrograph A20: Microstructure of as-quenched and tempered steel sample at 350^{o}C for 1hour. 2%Nital etch. ×100.



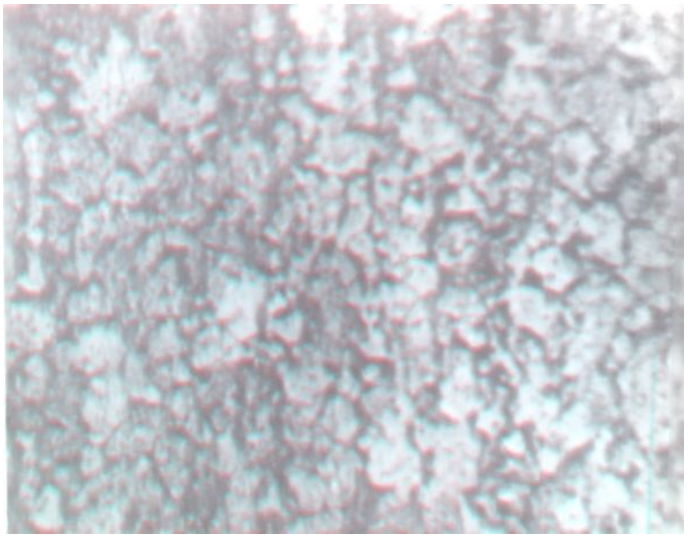
Micrograph A21: Microstructure of as-quenched and tempered steel sample at 450^{o}C for 1hour. 2% Nital etch. ×100.



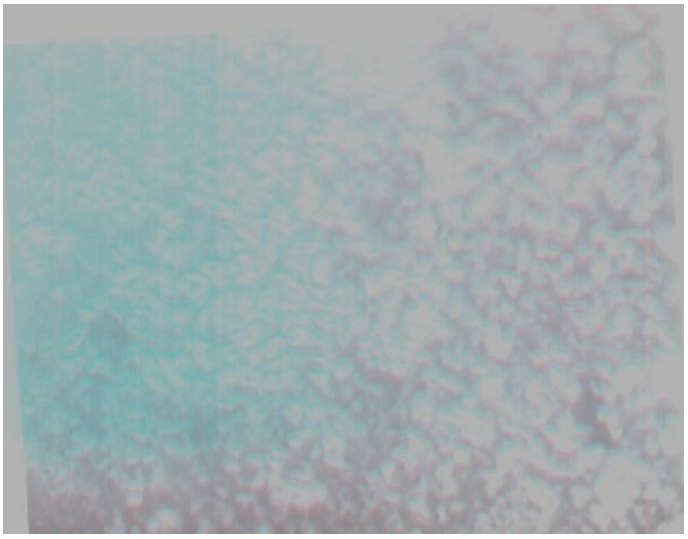
Micrograph A22: Microstructure of as-quenched and tempered steel sample at 550^{o}C for 1hour. 2% Nital etch. ×100.



Micrograph A23: Microstructure of IQ-treated HMDP steel sample intercritically annealed at 770^{o}C for 60minutes and tempered at 250^{o}C. 2% Nital etch. ×100.



Micrograph A24: Microstructure of IQ-treated HMDP steel sample intercritically annealed at 770^{o}C for 60minutes and tempered at 350^{o}C. 2% Nital etch. ×100.



Micrograph A25: Microstructure of IQ–treated steel sample intercritically annealed at 770^{o}C for 60minutes and tempered at 450^{o}C. 2% Nital etch. ×100.



Micrograph A26: Microstructure of IQ–treated steel sample intercritically annealed at 770^{o}C for 60minutes and tempered at 550^{o}C. 2% Nital etch. ×100.

APPENDIX B

Table B1: Mechanical properties of as-received, normalized and brine-quenched specimens

Specimen	Tensile strength (N/mm^2)	Yield strength {N/mm^2)	Elongation (percent)	Impact strength (J)	Hardness (HVN)
As-received	387	213	18.8	89	I57
As-normalized	546.14	310.7	24.8	114	136
Brine-quenched	861.48	636.32	13.85	46.10	297

Table B2: Variation of yield strength values of IQ-treated HMDP steel with intercritical temperature and time

Temperature, ^oC	Yield strength (N/mm^2)		
	30minutes	**60minutes**	**90minutes**
730	328	362.17	412.63
750	352	380	401
770	367	420	433.07
790	453	477	524
810	401	361	353.64

Table B3: Variation of tensile strength values of IQ-treated HMDP steel with intercritical temperature and time

Temperature, ^oC	Tensile Strength (N/mm^2)		
	30minutes	**60minutes**	**90minutes**
730	499	541	590
750	523	588	640
770	547	682	763
790	643	689	795
810	587	547	523

Table B4: Variation of percentage elongation values of IQ-treated HMDP steel with intercritical temperatures and times

Temperature, ^{o}C	Elongation (%)		
	30minutes	60minutes	90minutes
730	12.70	15.62	16.97
750	14.47	16.07	18.10
770	16.32	18.10	20.48
790	21.13	22.40	22.80
810	19.07	17.42	16.30

Table B5: Variation of hardness values of IQ-treated HMDP steel with intercritical temperature and time

Temperature, ^{o}C	Hardness (HVN)		
	30minutes	60minutes	90minutes
730	175	194	208
750	187	206	226
770	203	235	268
790	227	245	274
810	211	199	185

Table B6: Variation of impact values of IQ-treated HMDP steel with intercritical temperature and time

Temperature, ^{o}C	Impact Energy (joules)		
	30minutes	60minutes	90minutes
730	29.80	29.80	37.96
750	38.00	39.00	50.85
770	61.00	71.00	87.00
790	83.00	87.00	107.11
810	83.00	75.00	66.44

Table B7: Effect of tempering temperature on the tensile strength, yield strength and elongation properties of as-quenched steel

Tempering temperature, ^{o}C	Tensile strength N/mm^2	Yield strength N/mm^2	Elongation (percent)
250	657.75	480.66	15.67
350	609.76	455.99	17.23
450	509.00	376.45	18.2
550	449.24	331.12	19.78

Table B8: Effect of tempering temperature on the hardness values of as-quenched (brine) and IQ-treated HMDP steels intercritical at 770^{o}C for 60 minutes

Tempering temperature, ^{o}C	Hardness (HVN)	
	As-quenched	IQ-treated HMDP steel
250	240	217
350	214	205
450	185	193
550	167	176

Table B9: Effect of tempering temperature on the tensile strength, yield strength and elongation properties of IQ-treated HMDP steel intercritical at 770^{o}C for 60 minutes

Tempering temperature	Tensile strength N/mm^2	Yield strength N/mm^2	Elongation (percent)
250	622.48	392.26	18.90
350	570.56	357	21.09
450	540	336.85	22.40
550	523	324.10	23.40

Table B10: Effect of tempering temperature on the impact strength of as-quenched (brine) and IQ-treated HMDP steel intercritical at 770^{o}C for 60 minutes

Tempering temperature ^{0}C	Impact (joules)	
	As-quenched	IQ-treated steel
250	55	67
350	76	100
450	89	111
550	96	114