EFFECT OF CARBON CONTENT ON GRAIN REFINEMENT THROUGH THERMAL CYCLING IN PLAIN CARBON STEEL

BY

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CERTIFICATION

This is to certify that this research work, carried out by **Adebiyi Damilola Isaac,** of the **Department of Metallurgical and Materials Engineering,** has not been submitted elsewhere for the award of a degree and is accepted as meeting part of the requirements for the award of Master of Engineering (M. Eng.) degree in Metallurgical and Materials Engineering.

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DEDICATION

This research work is dedicated to **God Almighty** in whom is hidden all the treasures of wisdom and knowledge; **Jesus Christ** my Saviour, the centre of my joy, **Holy Spirit** my Inspiration and Comforter.

"You are the love of my life

You are the hope I cling to

You mean more than this world to me

I won't trade You for silver or gold

I won't trade You for riches untold

You are; You are my everything".

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ABSTRACT

The work investigated the grain refinement achievable in carbon steels by re-austenitization and alternate thermal cycling treatments and the properties developed in these carbon steels after cycling at different austenitizing temperatures. Re-austenitization thermal cycling consists of repeated heating to the austenite range, soaking for a short time at the peak temperature and then quenching in water, while the process of alternate thermal cycling consists of alternate heating between the austenite range and the two phase ($\alpha+\gamma$) range with intermediate quenching in water. The mechanical and microstructural properties of carbon steel with six different carbon levels (0.1%C, 0.22%C, 0.39%C, 0.54%C, 0.81%C, and 1.04% C) were studied at different austenitizing temperatures of 880 ^{O}C, 900 ^{O}C and 920^{O}C. The soaking times were 5, 10 and 15 minutes for 2, 4 and 6 number of thermal cycles. The effect of carbon content on grain refinement of the carbon steels was investigated. It was observed that an optimum temperature exists for grain refinement by thermal cycling treatments and this temperature decreases with increasing number of thermal cycles. For all the holding times investigated, grain size increased with increasing soaking time at the peak temperature, while higher number of cycles requires less holding time. However, with favorable condition of soaking time and peak temperature, grain size decreased with increasing number of thermal cycle up to a certain number after which further cycling does not provide any observable grain refinement but grain coarsening. Repeated austenitization, though accompanied by poor ductility gave higher strength than alternate thermal cycling but the latter produced slightly better grain refinement and better combination of strength and ductility.

CHAPTER ONE

INTRODUCTION

The attainment of finer ferrite grain structure in carbon steel is of interest because significantly higher yield strength and lower ductile-brittle transition temperatures can be achieved at the same time with ultra-fine grain sizes. The properties of various types of steel and of any given steel alloy at varying temperatures depend primarily on the amount of carbon present and how it is distributed in the iron. Refining of the final grain size of steels is one of the biggest challenges in order to produce high strength steels with improved toughness and ductility.

In the past few years, the challenges of obtaining ultra fine grain sized materials with minimum alloy cost have been pursued with great interest. This is generated by the need for economical higher strength steels with good ductility and toughness simultaneously, as well as today's energy and resource conservation.

Sequel to these requirements, research efforts have focused attention, among others, on the detailed physical metallurgy of carbon steels, thus enabling greater exploitation of their potentials for higher strength along with ductility for a wide range of applications.

Grain size is one of the microstructural features, which influence, the properties and response of metals to heat treatment more than others. This is particularly true for steel because the grain size, be it austenite or ferrite, influences not only the yield and tensile strengths, but also the ductility, ductile-brittle transition temperature, creep strength, and high-temperature ductility. Also, the austenite grain size affects the transformation characteristics of steel and through them the hardenability of the steel. It is

therefore no exaggeration to state that control of the grain size enables the control of the whole response of steel to heat treatment, and the consequent mechanical properties.

Traditionally, grain size control in an alloy is obtained by the addition of grain growth inhibitors such as aluminum, titanium, zirconium and vanadium. The fine grain thus obtained promotes a low ductile-to-brittle transition temperature, tends to reduce the hardenability of the steel, but does not significantly affect the strength of the steel.

Improved techniques to achieve ultra fine grains in an industrial process are expected to be developed to further increase material strength. This will require better understanding of such basic phenomenon as diffusion, recrystallization, grain growth, precipitation and transformation, as these can produce grain refinement. Also, partitioning in duplex microstructures, and pearlite dispersions, which are useful methods of restricting grain coarsening, must be understood. However, the method selected for grain refinement and grain-coarsening control for a specific alloy still depends on the character of the alloy.

A relatively new and more efficient technique for grain refining is the thermal cycling. The method consists of repeated cycles of rapid cooling from the austenite and (α+γ) phase range. It utilizes the fact that an austenite grain transforms to several ferrite grains on cooling whereas a single ferrite grain transforms to many austenite grains on heating. The method produces better grain refinement in most steels hardenable by heat treatment. The steels become ultra fine grained and hence exhibit increased strength and toughness.

1.1 Objectives of the research

This present work is an attempt to apply the two thermal cycling methods to study the grain refinement characteristics in plain carbon steels. The main objective, however is to investigate the effect of carbon content on the grain refinement characteristics of plain carbon steel when subjected to thermal cycling treatments.

Other specific objectives are to:

(i) Investigate the microstructural evolution of plain carbon steel subjected to thermal cycling treatments.

(ii) Determine the influence of number of heating cycles, peak temperature and soaking times on the mechanical and microstructural properties of plain carbon steels.

(iii) Determine optimum conditions of peak temperature, soaking time, number of cycles and carbon content for effective grain refinement in plain carbon steels.

1.2 Justification of the work

The properties of various types of steel and of any given steel alloy at varying temperatures depend primarily on the amount of carbon present and how it is distributed in the iron. The output of this research would:

(i) Enable a better appreciation of the potential of plain carbon steel for strengthening through thermal cycling

(ii) Serve as foundation for further research in grain refinement characteristics of plain carbon steels

CHAPTER TWO

LITERATURE REVIEW

For a given composition of ferritic and pearlitic steel, the most important factor controlling the strength and associated ductility is grain size (Irvine 1962 and Smith, *et al*, 1954). The increase in strength is based on the concept that grain boundaries act as barrier to dislocation motion, which is brought about when a material is subjected to stress. Thus smaller grain size i.e. increase in the number of grain boundaries leads to higher strength.

In order to achieve fine grain size, complex thermo-mechanical treatment is usually employed. But recent studies have shown that similar result can be achieved by rapid thermal cycling.

According to Pan (2004), by repeating the low temperature re-heating cycle (thermal cycling) to refine the prior austenite grain size, an ultra-fine ferritic (UFF) grain can be obtained in steels.

2.1 Grain size effect

The size of the grain in polycrystalline metal greatly influences a number of its mechanical and chemical properties. In pure metals and single-phase alloys, the advantages of a small grain size over a large one have long been recognized. Hall (1951) and Petch (1953) proposed a linear relationship between flow stress and square root of grain diameter. The relationship popularly known as the Hall-Petch equation is written as:

$$\sigma_0 = \sigma_1 + k_y D^{-1/2} \quad \text{.....2.1}$$

Where

σ_0 = yield stress

σ_i = friction stress which opposes dislocation movement.

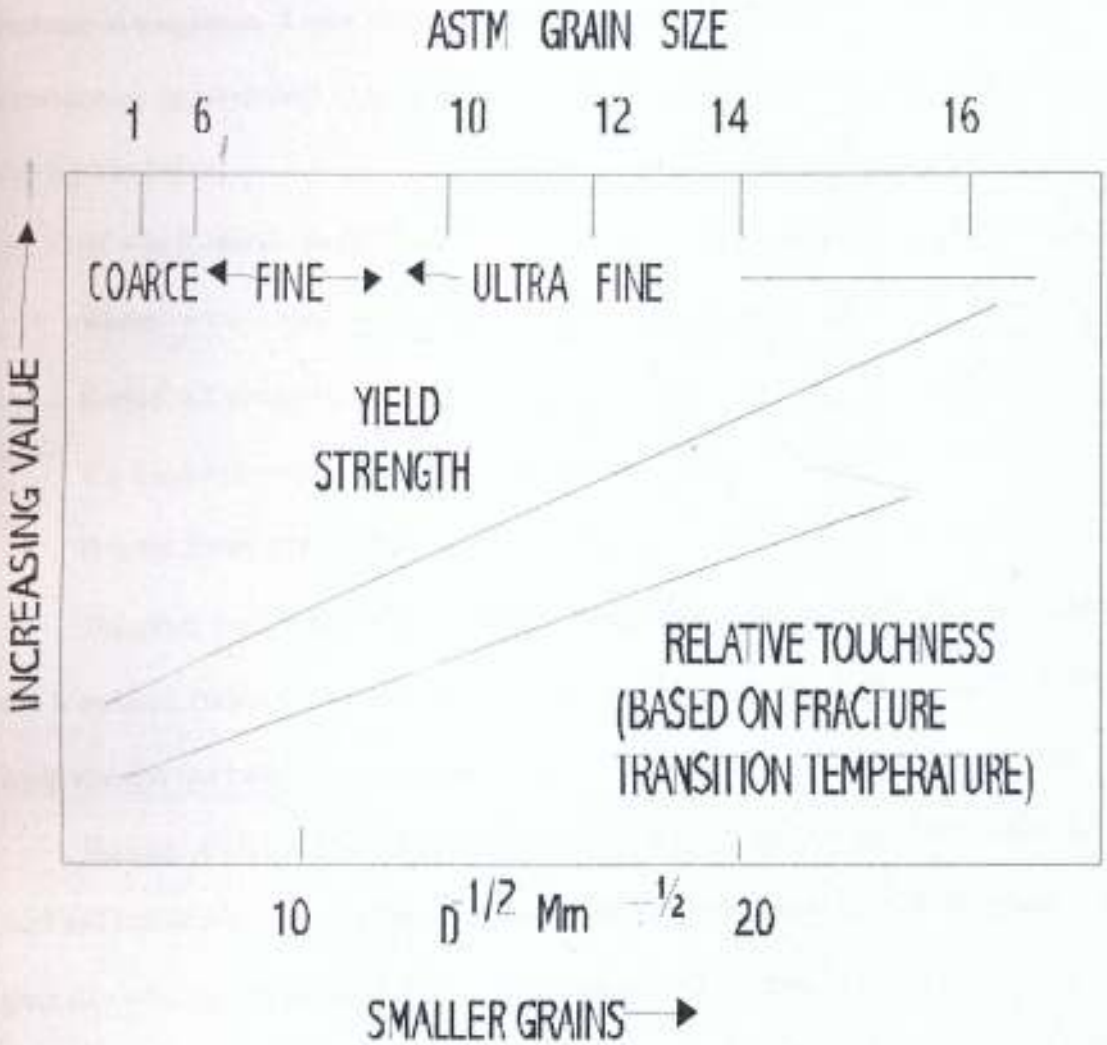
k_y = constant often called dislocation locking term.

D = grain size

The additional contribution to the yield strength from the $k_yD^{-1/2}$ term increases as the grain size is reduced. The strengthening effect for a given grain size depends on the magnitude of the constant, k (Raghavan, 1989).

Therefore the finer the grain size, the higher will be the yield stress. In fact grain refinement is the only commercially available conventional technique, which improves strength and ductility at the same time (Rajan, Sharma and Sharma, 1988).

Grange (1971), demonstrated an analogous increase in yield strength with the refinement of the prior austenite grain size in steel hardened by heat treatment. As shown in figure 2.1, both yield strength and notch toughness measured by fracture transition temperature progressively increased with grain refinement. The straight-line relation however, may be an over simplification. The important inference to be drawn is that grain refinement carried out to the greatest possible extent is a means of increasing useful strength of steel.



- Schematic representation of the effect of austenite grain size on strength and toughess
(GRANGE; 1971)

According to Llewellyn (1992), whereas strengthening effect usually leads to a decrease in toughness, it was shown that refinement of ferrite grain size also produced simultaneous improvement in toughness. The Petch equation linking toughness to grain size is given below:

$$\beta T = \ln \beta - \ln C - \ln D^{-1/2} \quad \ldots\ldots 2.2$$

Where

β and C are constants

T is the ductile-brittle transition temperature

D is the ferrite grain size

Therefore the ductile-britlle transition temperature decreases as the ferrite grain size is reduced. Below the equi-cohesive temperature, fine-grained steels exhibit superior creep strength and have higher fatigue strength as compared with coarse-grained steel.

Fujioka, *et al* (1979) investigated the effect of grain size on the creep behaviour of steel and reported that at low temperature the rupture life increases with decrease in the grain size whereas at high temperature, it decreases with decreasing grain size.

Grain size also affects the deformation behaviour of ultra fine-grained medium carbon steel; the yield drop of the steel increases with grain refinement, the total elongation remains unaffected, Luder's elongation and post-neck elongation increase, uniform elongation is reduced and the strain hardening exponent, n, reaches a near limiting value as the grain size is reduced.

The cyclic-stress-strain response of low carbon steel is affected by the grain size. Yokobori, *et al* (1979), who investigated the response, reported that the cyclic yield stress

increased as the ferrite grain size is decreased while the strain-hardening exponent decreased as the grain size is decreased.

Figure 2.2 shows the relationship between the grain size and strength obtained from measurement of stainless steel of the same chemical composition and various grain sizes. The figure shows that the yield stress is increased by about 350 megapascals due to grain refinement from 20μm to 1μm. This increase means that the yield stress is almost double in plain carbon steel. The increase in strength levels-off for grain sizes less than 0.5μm because sub-grains with a small angle grain boundary contribute less to strengthening than normal grains. (JFE 21st Century Foundation, 2003).

Valiev *et al* (2000) also reported that ultra-fine grained materials offer significant advantages in terms of high strain rate super-plasticity and improved mechanical properties compared with materials of conventional grain sizes.

There are four methods of increasing the strength of steel materials: Solid solution hardening, precipitation hardening, work hardening and hardening by grain refinement. Solid solution hardening, precipitation hardening and work hardening cause decrease in elongation and toughness, although strength is increased. In contrast, strengthening by grain refinement does not have this undesirable effect

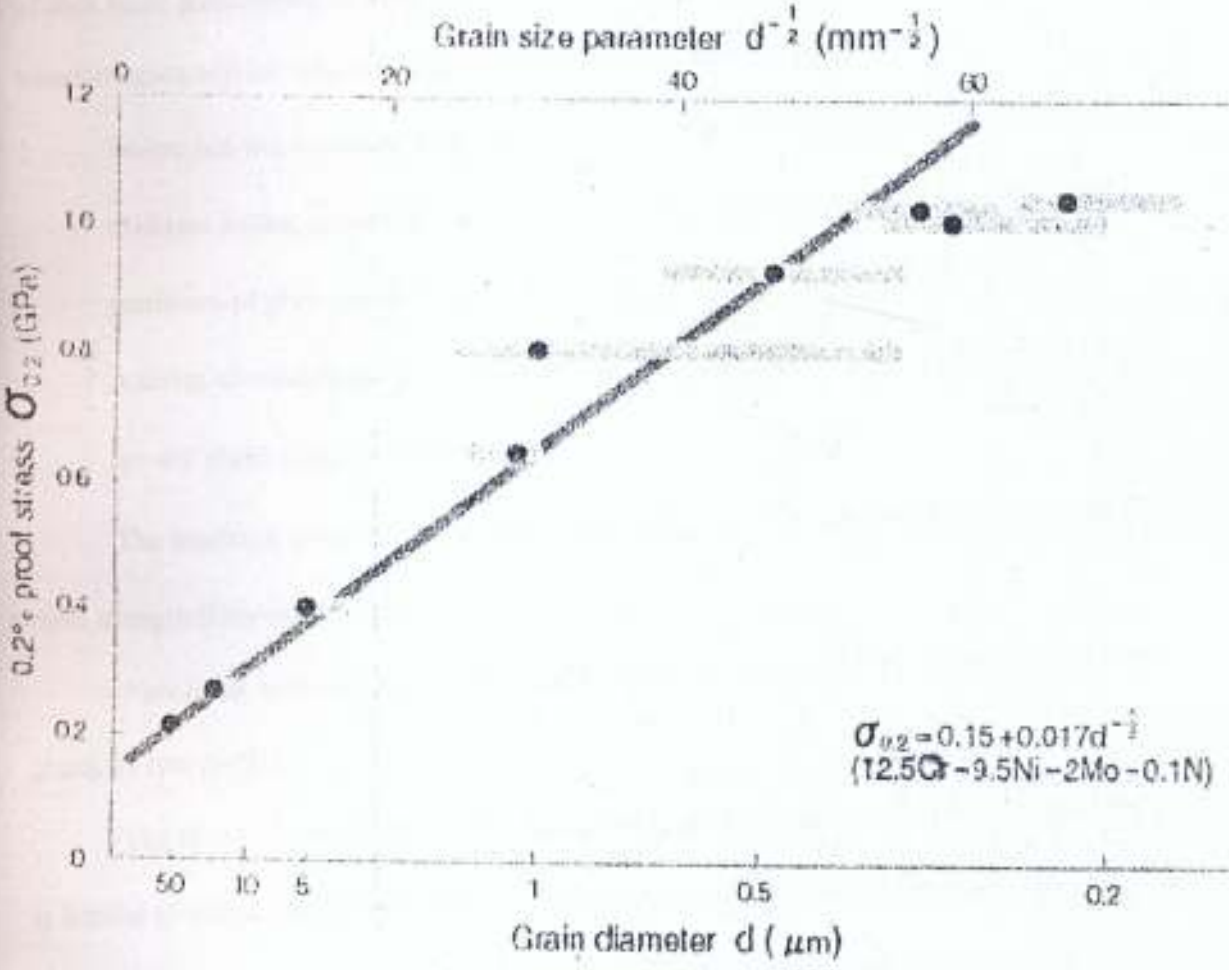


Fig.2.2 Relationship between the grain size and strength of stainless steel
JFE, Kawasaki steel, (21st Century Foundation, 2003)

2.2 Grain refinement

The techniques to achieve much finer grain in an industrial metalworking process are desirable to further increase material strength. To achieve this, a better understanding of such basic phenomena as diffusion, re-crystallization, grain growth, precipitation, and transformation will be required. Grain refinement is achieved either by;

1. Severe hot deformation in the austenitic condition followed by controlled rolling to obtain further grain refinement from $\gamma \longrightarrow \alpha$ phase transformation with a minimum of grain growth; or
2. A series of rapid heating and cooling cycles to produce sequence of $\alpha \longrightarrow \gamma$ and $\gamma \longrightarrow \alpha$ phase transformation.

The resulting grain size is in the range near 1μm and substantially increases the yield strength (Guy and Hyren, 1974).

According to Rollason (1977), large grains in metals can be converted to small grains by two methods:

The first which depends on the (allotropic) transformation occurring in solid state, is limited to a few metals and alloys, notably steels, aluminum and bronze, whereas the second method which is refinement of grain size is applicable to all metals.

It consists of annealing heavily cold-worked materials at a suitable temperature so as to form small equi-axed crystals, which are usually independent of the old shattered grains. This phenomenon is called recrystallization and is used extensively in non-ferrous metals and a great deal in the heat treatment of steel sheets.

2.2.1. *Allotropic transformations*

At different temperatures, many metals exist in different crystalline forms, or as they are sometimes called, allotropic (or polymorphic) modifications. As a result of polymorphic transformation, the atoms of a crystalline body having a lattice of one body are re-arranged to form a lattice of another type. At room temperature for example, pure iron has a body-centered cubic (BCC) crystal form called ferrite. Ferrite is stable up to 910^{O}C when it changes to face-centred cubic (FCC) structure called austenite. A further change takes place at 1390 ^{O}C when FCC reverts to BCC form called σ – ferrite (Lakhtin, 1979). As in solidification from the liquid phase, a certain super-cooling (or super-heating) is required with respect to the equilibrium temperature for a polymorphic transformation to proceed.

Polymorphic transformation, for instance, the ferrite-austenitic (Feα ⟶ Feγ) transformation in iron always proceeds in two stages: the nucleation of a new phase of a different lattice than that of the matrix and the growth of the gains (crystalline) of the new phase, first at the expense of the disappearing phase and latter by consuming other grains of that new phase (Gorelik, 1981). The new crystalline grains are of different size and shape and for this reason such transformations are also called recrystallization. If a metal is heated to a temperature slightly exceeding the temperature of a polymorphic transformation (critical point), a very fine grain can be obtained. This is made use of to refine the coarse grain structure obtained in solidification from the liquid state or in pre-heating from a very high temperature.

2.2.2. *Recrystallization*

Recrystallization is the nucleation and growth of new strain-free crystals from the cold worked metal. When a strain-hardened metal is heated, the old grains are not restored. Instead entirely new grains are formed and their sizes differ essentially from those of the initial grains. The formation of new equi-axed grains in place of the oriented fibrous structure is called recrystallization treatment or primary recrystallization.

The size of the grain after cold working and recrystallization may be larger or smaller than that of the initial grains. The grain size depends upon the temperature of re-crystallization annealing (Figure 2.3a), the holding time at this temperature, (Figure 2.3b), the degree of previous deformation (Figure 2.3c), the chemical composition of the alloy and the size of the initial grains. (Lakthin, 1979).

Reis (2004), explored the possibility of generating ultra-fine grains by cold rolling and annealing treatments and reported that annealing treatments which allow the static recrystallization to continue until full completion (i.e. end of primary re-crystallization but before the start of normal grain growth) can not achieve the target of a substantial grain refinement.

2.3 Methods of grain refinement

The problem of obtaining fine grain microstructure can be divided into two parts:

- The achievement of high nucleation density during recrystallization or transformation and
- The prevention of subsequent grain growth.

These objectives are achieved through a process of thermo-mechanical treatment or by rapid heat-treatment process.

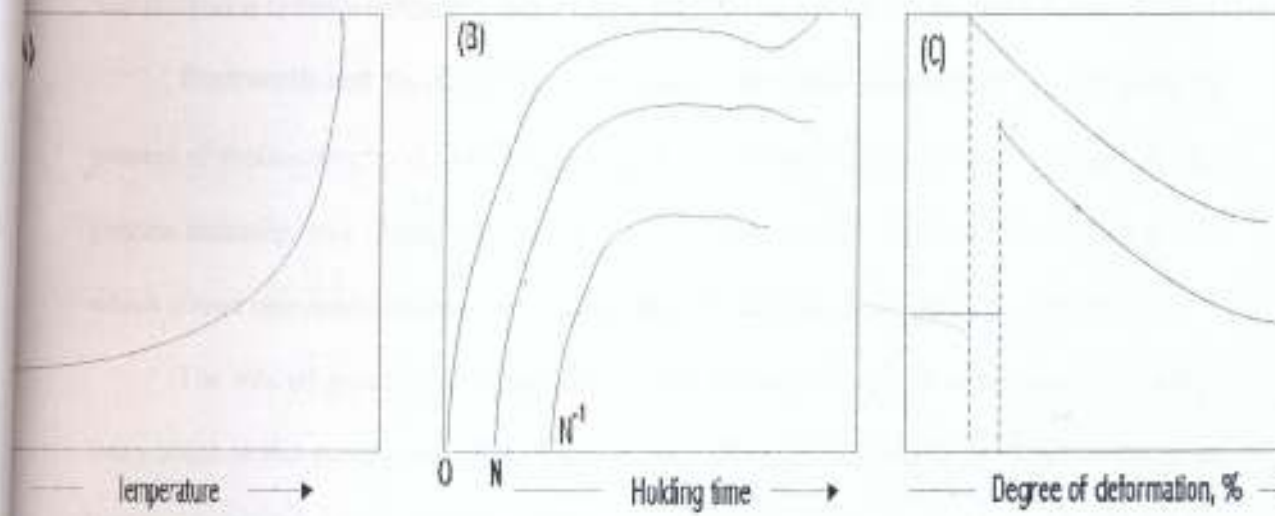


Fig. 23 Schematic representation of the effect of the temperature, holding time and degree of previous deformation on the size of recrystallized grains ($l < l' < l''$) (on and on' lengths of the incubation periods I and I' critical degrees of deformation)
(Y.U. LAKHTIN, 1977)

2.3.1. Thermo-mechanical treatment

Thermo-mechanical treatment involves the simultaneous application of heat and a deformation process to an alloy in order to change its shape and refine the microstructure. The method is rather unorthodox but technologically viable for grain refinement.

Duckworth and Baird (1969) investigated the grain refinement in mild steel by process of thermo-mechanical treatment and a considerable success was reported. In this process austenite was rolled just above the AC_3 temperature and then cooled at a rate, which allows recrystallization to take place, but restricts the time for grain growth.

The role of processing in creating a fine grain size is very significant. Although every stage in the processing chain from ingot casting to cooling from final working or heat treating step may exert some influence, by far most important role is played by the temperature at which hot-working is completed or final annealing temperature for cold worked material. According to Burke and Weiss, (1970), fine grain size is favoured by a low finishing temperature and a rapid cooling rate from the working temperature.

Kamma (1990), investigated the optimum conditions for grain refinement in carbon steels by thermo-mechanical treatment. Various microstructures - martensite, tempered martensite and pearlite/ferrite-serving as starting point phases were cold-worked to varying degrees and then subjected to re-crystallization annealing. The cyclic process of deformation and heating was respectively carried out on each of the initial microstructure, with deformation and annealing temperature serving as variable parameters. The result showed that cycling application of deformation and heat treatment enhanced the refinement tendencies of each microstructure. In all, pearlite/ferrite structure exhibited the largest degree of grain refinement while the smallest grain size

was obtained from a one-cycle re-crystallization annealing treatment of a tempered martensitic structure.

With adequate deformation while the steel is in a metastable condition, strength levels not attainable by standard quench-and-tempered treatments have been obtained, quite often with higher ductility and fracture toughness than those normally expected at these very high strength levels. However, thermo-mechanical treatments are not widely used commercially, presumably because of practical difficulties in adapting experimental techniques to actual production (Metals Handbook, 1978).

Pan (2004), investigated methods of producing ultra-fine (UFF) structures in four low carbon steels and reported that steels with UFF grain sizes (i.e. grain seizes below 3μm) can be obtained by two methods, more or less convenient to mass production: Deformation-induced ferrite transformation from fine to ultra-fine grained austenite and the static recrystallization of the various heavily cold worked initial microstructures. He concluded that by using the complex pre-treatment to refine the prior austenite grain size, (cold rolling), repeating the low temperature reheating cycle (thermal cycling) or using martensite initial microstructure, UFF grain size can be obtained in these steels.

2.3.2. Rapid heat-treatment of steel.

The heat-treatment of steel most often involves controlling by suitable temperature-time cycle solid-state phase changes or precipitation and coalescence of carbide particle with the aim of developing a microstructure having the combination of mechanical properties best suited for the intended use of the product. In designing a specific heat treatment cycle, temperature and time are inter-dependent variables. Solid-

state reactions involved in austenitizing occur faster as the temperature is higher. A modest increase in temperature may be equivalent to an order of magnitude increase in time in an isothermal treatment. This situation makes it possible to speed up a variety of heat-treatment process. (Grange, 1971).

Shortening the austenitizing cycle, in addition to saving of time minimizes oxidation and carburization and controlled atmospheres will often be unnecessary. An additional advantage would seem to be in the probability that rapid austenitizing will minimize growth of austenite grains and hence lead to a finer grained heat treatment product.

Grange (1971), carried out an experiment on 0.2%C steel on the possibility of refining austenite grains by a rapid austenitizing cycles. One was made according to so-called coarse-grained particle (Si-killed) and the other to fine grained particles in which killing with silicon was followed by additional de-oxidation with aluminum (Al-killed). Small samples were quenched into lead at 870^{O}C for various time periods in the range 5 seconds to 20 minutes. With short austenitizing time, the Si-killed steel was almost as fine as the Al-killed steel whereas with a 20 minutes austenitizing representative of conventional slow austenitizing, the two are very different as shown in figure. 2.4

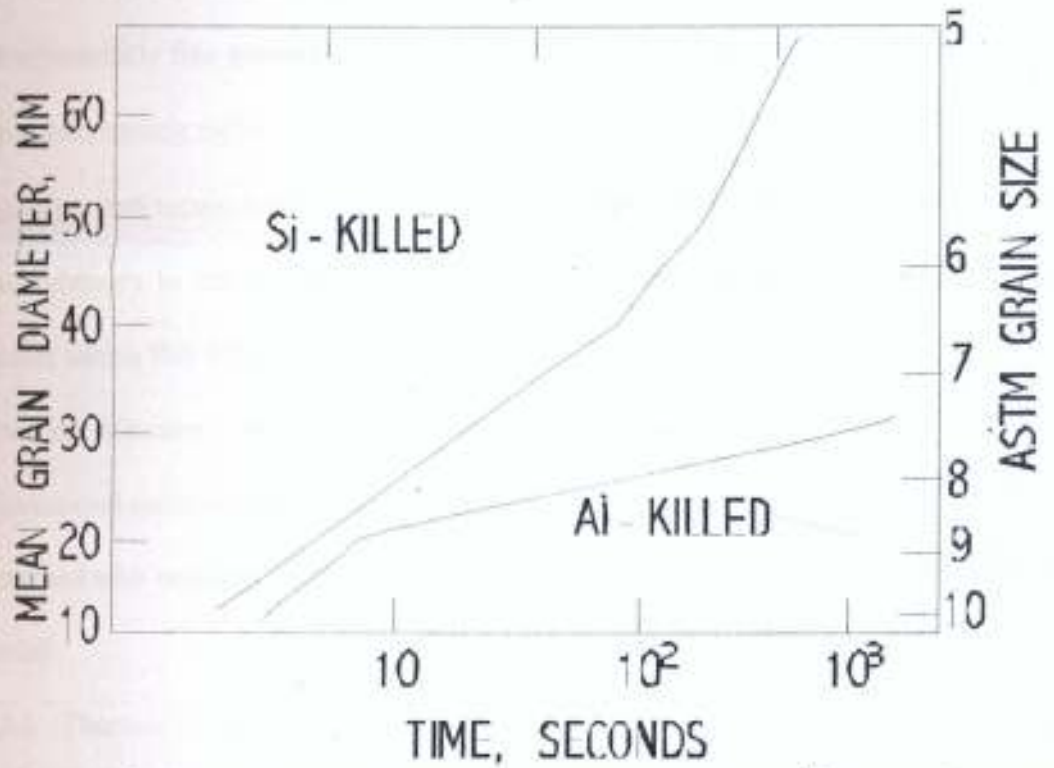


Fig. 2.4: Austenite grain growth with time at 870°C AISI 1020 steels (GRANGE, 1971)

With a rapid austenitizing cycle, regardless of de-oxidation practice, both steels are substantially fine grained than when austenitized conventionally (Grange, 1971). In a rapid austenitizing cycle, the steel is rapidly heated starting at room temperature or at any higher pre-heat temperature below A_1 to a temperature above A_3 but preferably no higher than necessary to ensure complete austenitization. As soon as the centre of the cross-section attains this temperature, the steel is immediately quenched or otherwise cooled. Thus no intentional hold at temperature or soaking is employed. In contrast, in a conventional austenitizing treatment, the steel is heated more slowly through the A_1 to A_3 range and after reaching furnace temperature, is held for an appreciable time before being cooled.

2.3.3. Thermal cycling

A single-cycle rapid austenitizing results in appreciable grain refinement over conventional austenitizing only if the initial microstructure is favourable. According to Speich and Szirmae, (1969), the preferred starting structure for carbon steel is a hot rolled structure, whereas Grange (1971), reported that martensite or fine ferrite-carbide aggregate structures are desirable. In treating a coarse undesirable initial microstructure by rapid austenitizing, a preliminary cycle can be given such that the microstructure is martensite at the start of the second cycle. This becomes a multiple thermal cycling. It suggests naturally therefore from the degree of grain refinement which results from two-cycle treatment that additional cycles might develop even smaller austenite grains.

Grange (1971), has devised an austenitizing cycle that result in considerable further grain refinement. The rapid heat treatment process originally developed by Grange (1971), consists of repeated cycles of rapid austenitizing each of very short

duration at a temperature barely sufficient to austenitize and followed by intermediate drop quenching. Austenite nuclei are more numerous as the prior austenite grain size of the sample being rapidly heated decreases. Therefore, progressive grain refinement on cycling repeatedly can be expected as long as each successive cycle treats a sample with decreasing prior austenite grain size.

According to Grange (1971), in his report on the investigation carried out on the response of several different steels to multi-cycle rapid austenitizing, additional grain refinement usually results from two or more cycles and most steels hardenable by heat treatment become ultra-fine grained and hence exhibit increased strength and toughness.

The report of the study carried out by Karlson (1973), on grain refinement of plain hypo-eutectoid steel by means of the repetitive short term austenitization shows that for carbon levels of 0.38, 0.18 and 0.05 wt% up to 1:10 in ferrite-pearlite condition for original grain sizes of 50-100μm is obtainable.

Koo and Thomas (1976) applied a non-standard thermal cycling process to low carbon Fe-1.12%C – 0.5% Mn steel. The process consists of alternate thermal cycling in the austenite range and two phase ($\alpha+\gamma$) range with intermediate quenching so as to produce a micro-duplex fine grained mixture of martensite and pro-eutectoid ferrite. Accordingly, the technique produces better grain refinement than obtained by repeated rapid austenitizing. It appears that the second phase (pro-eutectoid ferrite) may effectively inhibit grain growth of austenite in the two-phase range. Moreover, the second phase rendered the grain size refinement to be less sensitive to holding time, whereas the degree of refinement by the repeated austenitizing method is very sensitive to slight changes in holding times at the peak temperature (Grange, 1971). However, Grange

(1971) and Karlson (1979), reported that thermal cycling process will not refine the grain size if carried beyond a limited number of cycles. The instability of ultra-fine austenite grains limits the degree of grain refinement. Eventually, the grains become so small and hence so unstable that their growth nullifies any further refinement. This limited number of cycle is called "stand-off" point and the number of cycles required to reach this point varies with several factors, including the particular steel being treated, the mass of the sample and the rapidity of heating attained in each cycle (Grange 1971). Grange reported that for the variety of steels investigated, the "stand-off" point often occurs in three or four cycle and Koo and Thomas (1979) reported that for the low carbon, Fe - 0.12%Co-5%Mn studied, multi-cycling more than two cycles did not provide additional grain refinement. However, a higher degree of grain refinement is likely with a given cycle whether one or more, if the steel contains effective dispersion of particles, which inhibits grain boundary movement.

Porter and Dabkowoski (1970) reported that 5Ni-Mo-Cr-V steel, when subjected to rapid heat treatment at a high heating rate shows the "stand-off" point between 7 and 8 cycles. In the case of Fe-15Ni-5-30CO-5-10MO-0.2Ti alloys subjected to cyclic re-austenitization by Nakazawa *et al*, (1978), the grain size of all the alloys was reduced to less than 50μm by cycling five times at a suitable austenitizing temperature.

Badmos (1993), investigated the effect of prior cold work on grain refinement of mild steel and reported that multi-cycling more than four cycles did not provide additional grain refinement but rather further cycling improved the uniform grain size distribution.

In plain carbon steel, the size reduction is generally more pronounced for steels with higher carbon content; and this is probably due to the fact that, in a material with higher carbon content, cycling increases the number of very fine carbide particles which inhibit grain growth.

2.4 Mechanism of grain refinement by thermal cycling

Grain coarsening of austenite is reversible as several new grains of austenite are nucleated in the volume that had been occupied by one old austenite grain (Samuels, 1980). During grain refinement, an austenite grain transforms to several ferrite grains on cooling whereas a single ferrite grain transforms to one austenite grain on heating (Nicholson 1970).

When cooled below the A_3 temperature, there are pronounced morphological changes that take place in steel which in general apply to hypo- and hyper-eutectoid phases. The austenite becomes supersaturated in carbon.

Kalson (1972), asserts that the leading role in grain refinement is played by ferrite nucleation from austenite. As a result of a survey of the behaviour of plain carbon steel, Dube proposed a classification of morphologies of ferrite which occur as the $\gamma \longrightarrow \alpha$ transformation temperature is lowered (Honeycombe, 1981).

Ferrite crystals normally nucleate at the austenite grain boundaries, called grain boundary allotrimorphs, on further cooling below A_1 the carbon enriched regions of the remaining austenite provide nucleation site for pearlite. The ferrite, growing from the grain boundaries along well-defined matrix planes often appears in a plate-like shape called Widmannstatten plate. There is also nucleation of roughly equi-axed crystals

within the austenite grains and can grow as Widmannstatten plates or blocky ferrite and this morphology is referred to as intergrannular idimophs.

Increasing under-cooling will enhance homogenous nucleation of ferrite entirely within the austenite grain boundary. During the quenching part of a cycle, ferrite will form exclusively at the austenite grain boundaries with cementite finely dispersed at the ferrite boundaries. During the heating in the next cycle, new austenite will nucleate preferentially on cementite particles and so on (Kalson, 1973).

However, Grange (1971) supposes that ferrite nuclei form preferentially at the boundaries of an original austenite grain that has been transformed into several ferrite grains once it has been found that the number of austenite nuclei per unit volume is markedly higher for smaller original austenite grains. Each ferrite then transforms to a single austenite grain, which in turns transforms into several ferrite grains and so on.

Cota (2004), investigated the microstructure and mechanical properties of a microalloyed steel with Ni and V in its composition, after thermal treatments, and observed that, the niobium atoms which dissolved during austenitizing, are retained in the martensite after quenching and are available to produce hardening during other heat treatment cycles.

2.5 Microstructure developed by thermal cycling

In addition to grain refinement, rapid austenitizing provides opportunities for developing different microstructural morphologies than those resulting from slow conventional austenitizing. Grange, (1971), reported that, often, the transformation products of ultrafine-grained austenite appear substantially different from those of fine or coarse-grained austenite and no doubt have a direct morphological effect on mechanical

properties. Apart from this effect, rapid austenitizing can be conducted so as to retain a very fine uniform dispersion of undissolved carbides, which with slow heating would coalesce into fewer and larger particles (Grange, 1967). In the hardened steel, it is reasonable to assume that very fine carbide particles may be more desirable than relatively large, spheroidal carbide particles. Under service stresses, which plastically deform, large carbides may crack and initiate failure. Also, a very fine dispersion should promote grain refinement by increasing the number of austenite nuclei formed on heating above AC_1 (Speich and Szirmae, 1969), and by inhibiting growth of austenite grains after impingement (Grange, 1971).

However, while a unique microstructure of the sort discussed above can be developed also in medium carbon steels, it is more readily formed in high carbon steels (Grange, 1971). It is almost obvious; therefore, according to Grange (1971) that morphology obtained in thermally cycled microstructure differs from the conventionally hardened microstructure.

The formation of ferrite and pearlite in rapidly heat-treated hypereutectoid steels was reported by Karlson, (1973). When cooled below AC_3 temperature the austenite becomes supersaturated in carbon. Ferrite normally nucleates at the austenite grain boundaries. On further cooling below A_1 the carbon enriched regions of the remaining austenite provide nucleation sites for pearlite. The ferrite, growing from the grain boundaries, often appears in a plate-like shape (Widmannstatten plates). In large grains, ferrite can nucleate also within the austenite grains and can grow as Widmannstatten plates or blocky ferrite. Increased undercooling will enhance homogeneous nucleation of ferrite relative to grain boundary nucleation, producing further refinement of the ferrite-

pearlite structure. The report, therefore, concluded that the rapid nucleation of ferrite during the rapid cooling from the austenite temperature is important for the mechanism of grain refinement.

Brzuszek (2002), investigated the microstructural evolution of a low carbon 0.15%C - 1.39%Mn steel after an ice-brine quenching from 1200^{O}C and reported that a fully martensitic microstructure was formed with distinct blocks which consisted of fine martensite laths (low carbon steels) parallel each other and they were delineated by the different orientations of the laths, according to the parent austenite grains. He also reported an increase of ferrite volume fraction with decreasing quenching temperature because less austenite is transformed to martensite and the microstructure was so refined. He observed that the ferritic grain refinement was better with the sample submitted to annealing during one hour because a great refinement with a more uniform structure was reached while short annealing time promoted a finer grain but with non-homogenous microstructure.

Cota *et al* (2004), studied microstructure and mechanical properties of microalloyed steel after thermal treatments, and reported that the microstructure of the steel after intercritical treatments revealed martensite thin layer thickening on the ferrite grain boundaries with time, until it formed a continuous network around the ferrite grains. This, he said was a result of the increase in the martensite volume fraction with intercritical treatment time. He also observed that there was no variation of the ferrite grain size, and its average value stabilized.

Cota further observed martensite structure in samples austenitized at different temperatures prior to quenching. The structure, according to him, showed a cluster of

needles typical of low carbon martensite. He also observed that an increase in intercritical time led to an increase in the martensite volume fraction.

Although Steels (1990) explained that the martensite hardness is usually expected to decrease as the austenite grain size increases, Cota *et al* (2004) observed the opposite tendency i.e. as the austenitizing temperature is increased, both the austenite grain size and the martensite hardness increased. This he said is probably due to the formation of small amounts of bainite during quenching of the sample austenitized at $900^{O}C$ and $1000^{O}C$. Therefore, the microstructures of sample austenitized at $900^{O}C$ and $1000^{O}C$ consisted of martensite and bainite and the microstructure of samples quenched at $1100^{O}C$ is mainly martensite.

Brzuszek (2002), reported that for samples quenched after soaking conditions, the final microsructure was formed by a mixture of ferrite, matensite and traces of bainite

2.6. Factors affecting grain refinement during thermal cycling

A fine austenite grain size results in the formation of a fine ferrite grain size on cooling to room temperature. Although, the austenite grain size is of major importance, other factors also play a part in developing a fine ferrite grain size. Thus, the presence of elements such as carbon and manganese or an increase in the cooling rate from the austenite range will lead to a refinement of the ferrite grains (Llewellyn, 1992).

Grain refinement by thermal cycling is influenced by a number of factors which include:

1. Chemical composition of steel.
2. Initial microstructure.
3. Number of cycles

4. Rate of heating.

5. Peak temperature and soaking time.

2.6.1. Chemical composition of steel

The chemical composition of an alloy strongly affects the degree of grain refinement and the minimum grain size attainable in it.

Increasing carbon decreases austenite to ferrite transformation temperatures and so causes grain refinement. Kalson (1973) investigated the responses of steels with carbon content 0.38, 0.18 and 0.5 wt% and reported that the size reduction was more pronounced for steel with higher carbon content.

A strong tendency to coarsen exists for austenite grains as they become smaller. Thus, a grain growth inhibitor exerts an important influence on the degree of grain refinement. Grain size control in steel has traditionally been obtained by the addition of grain growth inhibitors such as aluminum, titanium, zirconium and vanadium; which form nitrides, carbonitrides or carbides that are stable at austenitizing temperatures and thus act as barriers to grain growth.

Grain refinement is promoted in high-strength maraging steel by increasing cobalt and titanium content whereas increasing molybdenum content suppresses it (Nakazawa 1978). Some steels are treated during ingot de-oxidation with elements such as Al, Nb, V, T, and Zr, the presence of which inhibit austenite grain growth. The austenite grain size after heating at normal austenitizing temperature is then a good deal smaller than normal steel. Samuels, (1980).

2.6.2. Initial microstructure

In hypereutectoid steels, the austenite grain size can be refined adequately only if a speheroidized structure of eutectoid and pro-eutectoid cementite is established (Samuels 1980).

Speich and Szermae (1969), have shown that the preferred starting microstructure for carbon steel is a hot rolled structure. According to them, the structure is a very open bainite with small patches of auto-tempered martensite and the ferrite grain boundaries heavily outlined with carbides. Other factors such as alloy segregation in the hot rolled structure, according to Speich and Szermae (1969) could promote austenite nucleation and help to suppress grain growth of the austenite after impingement, and thus could be important in the superior performance of the hot rolled starting microstructure.

However, Grange (1971), investigated the effect of microstructure on the grain refinement of AISI 5140 steel (C-0.399, Mn-0.02%, S-0.007%, Si-0.23%, Cr-0.97%) and reported that martensite or fine ferrite aggregate such as tempered martensite are desirable. According to his explanation, with a relatively fine ferrite-carbide aggregate such as tempered martensite, the diffusion distances required to distribute carbon atoms uniformly in austenite are small and hence complete austenitization is accomplished quickly at a lower temperature and this minimizes grain growth after impingement. Secondly austenite nucleates on rapid heating at many more locations when the initial microstructure is a fine aggregate. The result is smaller austenite grains early in the cycle before austenite grain growth becomes important.

2.6.3. Number of cycles

A small austenitic grain size is not usually recovered in a single re-austenitizing treatment of a very coarse grain structure. Moreover, the austenitic grain size is likely to be rather mixed after a single grain-refining treatment and more uniform after two. (Samuels 1980). The austenite grain size decreases with increasing number of autenitizing cycles but up to a certain limit. Badmos (1993), investigated the grain refinement of ST44-2 steel and concluded that multi-cycling more than four cycles did not provide additional grain refinement but further cycling improved the uniform grain size distribution.

As explained by Grange (1971), in the first circle, austenite nuclei are relatively few and the first nuclei grow considerably before impingement with the result that when austenitization is complete the grains are of mixed sizes and, on the average, not very fine. In the second cycle austenite nucleates at many locations so the grain remains small when austenitization is completed. However, the cyclic process ceases to be effective after a limited number of cycles called "stand-off" point and the grains eventually become so small and unstable that their growth nullifies any further refinement.

2.6.4 Rate of heating.

Heating rate has been shown to affect the grain size and structure of some steels treated with various heating processes. Zhou *at al*, (1981) confirmed the phenomenon that slow heating leads to heredity of grain size and heating up to recrystallization temperature causes grain refinement.

Porter and Dabkowoski (1970), investigated the effect of heating rate and the number of cycles of rapid heat treatment of 5Ni-Cr-Mo-V steel and concluded that for a particular number of cycles, grain refinement increases with heating rate

2.6.5 Peak temperature and soaking time.

In hypo-eutectoid steel, several new grains of austenite can be nucleated in the volume that had been occupied by one old austenite grain and the size to which this new grain grows depends principally on the new austenitizing temperature. Thus the new austenite grain size will generally be smaller than the old austenite size if the new austenitizing temperature is lower. (Samuels, 1980). Rollason, (1973), investigated the effect of duration and temperature of annealing on the grain size of 0.18% carbon steel. He reported that the grain size increases as the temperature increases, and at a given temperature the grain size increases with time, rapidly at first, then slowly.

The instability of ultra fine austenite grains limits the degree of grain refinement attainable in them by multi-cycle rapid austenitizing and, all other factors being equal, this instability is greater the higher the temperature necessary for complete austenitization (Grange, 1971).

Badmos (1993), in the study conducted on the grain refinement of ST 44-2 steel by thermal cycling reported that cyclic re-austenitization produced better grain refining effect with austenitizing temperature just slightly above the AC_3 and holding time of about 40 seconds.

Low austenitizing temperature and short holding time are desirable in multi-cycle rapid austenitizing because of the favourable effect on resistance to grain growth. Therefore, for any appreciable grain refinement in a multi-cycle rapid austenitizing

process, the program must be properly designed with respect to the austenitizing temperature and holding time. Lower austenitizing temperature decreases the austenite grain size, but increases the presence of bainite, leading to lower hardness of the steel. The increase in the time of intercritical treatment at 750^{O}C shows little effect on the ferrite grain size, but it raises the martensite volume fraction and, as a result the hardness of the steel. Cota *et al* (2004).

Brzuszek (2004), reported that short annealing time at 800^{O}C produced a heterogeneous microstructure. As shown in Figure 2.5, by Cota *at al* (2004), the hardness and austenite grain size are functions of the austenitizing temperature

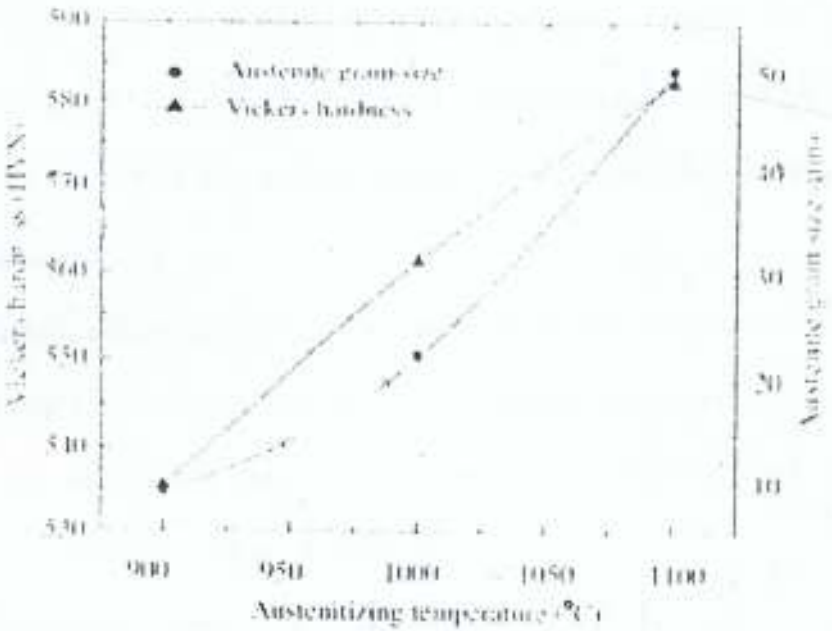


Fig. 2.5 Vickers hardness and austenite granite grain size as functions of the austenitizing temperature. Standard deviations are below 10%.
Cota, (2004).

2.7 Grain size measurement

Grain size is measured with light microscope by counting the number of grains within a given area, by determining the number of grains (or grain boundaries) that intersect a given length of random lines, or by comparing with standard grain-size chart. Most grain size measurements invoke assumptions relative to the shape and size distribution of the grains and, therefore, must be interpreted with some degree of caution. (Dieter, 1988). As pointed out by DeHoff and Rhines (1968), the most applicable technique is that which provides structural information which may be correlated with property data and which may be accomplished by relatively simple measurement on the polished surface.

Methods are available for measuring the grain size of metals, some of which have been in use for a considerable time. All of the methods involve a measurement taken on a plane section through the metal and the results are usually quoted directly without correlation to the respective spatial diameter.

2.7.1 Comparison method

The specimen is prepared and etched for microscopic study. The image of the microstructure projected at a magnification of 100x, or a photomicrograph of the structure at the same magnification, is compared with a series of graded standard grain-size chart.(Avner, 1974). By trial and error, a match is secured and the grain size of the metal is then designated by a number corresponding to the index number of the matching point. The ASTM grain-size number may be obtained as follows:

$$N = 2^{n-1} \quad \text{.....2.4}$$

Where,

N = index number of the matching point

n = ASTM grain size number of the steel

Hence

$$N = I + \log N / \log_2 \quad \text{.....2.5}$$

The number of grain per square millimeter NA can be calculated if the ASTM grain size number is known and Pascoe (1978), gave this as:

$$NA = 8 \times 2^n \quad \text{.....2.6}$$

2.7.2 Intercept (or Heyn) method

The grain size is estimated by counting on a ground-glass screen, or photomicrograph, or on the specimen itself, the number of grains intersected by one or more straight lines. Grains touched by the end of the lines count only as half grains. Counts are made on at least three fields to assure a reasonable average. (Avner, 1974). The length of the line in millimeters divided by the average number of grains intercept by it gives the average intercept length or grain 'diameter' (Avner, 1974); i.e. average linear intercept, l, is given by

$$l = L/n \quad \text{.....2.7}$$

The average linear intercept L is less than the diameter of the grain in the plane of sectioning, and in fact, L is smaller than the diameter of the circle of equivalent area to the grain in the plane of the polish (Weinberg, 1970). This method developed by Heyn, (1903), can be used to determine the grain size both in single-phase and multiphase alloys.The determination of the mean linear intercept in two-phase structure is basically similar to that of single-phase alloys. Consider that there are nα grains of the α-phase and nβ grains of the β-phase on an intercept of length L. The length Lα of intercept occupied by the α-phase is given by the product of the intercept length and the proportion of α-phase, Pα.

$$L\alpha = LP\alpha \quad (2.8)$$

Similarly

$$L\beta = LP\beta \quad (2.9)$$

The mean linear intercept of α and β grains $L\alpha$ and $L\beta$ are therefore given by:

$$L\alpha = LP\alpha/n\alpha \quad (2.10)$$

$$L\beta = LP\beta/n\beta \quad (2.11)$$

The proportion of phases is obtained from a method known as linear analysis.

2.7.3. Hilliard's circular test method

Hilliard's circular test (Hilliard, 1964) is a very quick and useful method for determining an equivalent ASTM, G by means of the P_L count. The method consists in superimposing a test circle of known length on the micrographs, and the number of intersections of the grain boundary per unit length of test circle P_L is measured. The ASTM grain size number G is calculated using the Hilliard's equation:

$$G = -10.00\text{-}6.64 \log L_3 \quad (2.12)$$

Where

$$L_3 = L_T/PM \quad (2.13)$$

P is the total number of grain-boundary intersections made by a test circle laid several times to give a total length, L_T on a field viewed at any magnification, M.

2.7.4. Circle intercept method.

This method is based on calculations of the average cross-sectional area F_K of a circle whose diameter is d (mm) drawn on a micrograph of known magnification V, to determine the average number of grains per unit area NA.

Hence

$$F_K = \frac{\pi d^2}{4} (mm^2) \quad \text{2.14}$$

average cross-sectional area of grain (f_m) is given as:

$$F_m = F_K.10^6/(0.67n + 7)V^2 \quad \text{2.15}$$

$$NA = \sqrt{Fm}\,(mm) \quad \text{2.16}$$

Where;

Z = number of grains lying completely within the circle.

n = number of grains intercepted by the circle.

0.67 = oertal factor.

2.7.5. Planimetric method

This is the most accurate of all the methods of determining of the average number of grains per unit area. The method originated by Jefferies *et al* (1916), measures the area of one or more groups of grains using the area integration. By dividing the number of grains by the measured area, the mean value of grains per unit area is obtained. The measurements are made from several locations on the polished and etched microstructure.

2.8. Plain carbon steel.

Plain carbon steels are alloys of iron and carbon in which carbon varies from traces to about 2 percent by weight. The upper limit of carbon in plain carbon steel corresponds to the maximum solubility of carbon in austenite. (Rajan, Sharma and Sharma, 1988). Steel is considered to be plain carbon steel when no minimum content is specified or required for chromium, cobalt, columbium (niobium), molybdenum, nickel, titanium, tungsten, vanadium or zirconium or any other element to be added to obtain a desired alloying effect; when the specified level for copper does not exceed 0.40 percent; or when the maximum content specified for any of the following elements does not

exceed the percentages noted: manganese 1.65, silicon 0.60 copper 0.60 (The American Iron and Steel Institute (AISI))

Plain carbon steels are classified according to various de-oxidation practices, and this will have effect on the properties of the steel. However, variations in carbon content have the greatest effect on mechanical properties, with increasing carbon content leading to increased hardness and strength. The properties of plain carbon steel are primarily derived from the presence of carbon. Other elements such as manganese, silicon, phosphorus and sulphur may be present in relatively small amounts, but their purpose is not principally that of modifying the mechanical properties of the steel. (Clerk and Verney, 1961). By varying the amount of carbon in steels and by selecting a heat treatment programme suited to the carbon content, it is possible to produce a vast range of different mechanical properties such as are not available in other metallic alloy. Moreover, carbon steel is relatively inexpensive when compared with non-ferrous alloys (Higgins, 1993).

2.9 Structure of plain carbon steel

The physical properties of various types of steel and any given steel alloy at varying temperatures depend primarily on the amount of carbon present and how it is distributed in the Iron. Before heat treatment, most steels are a mixture of three substances: ferrite, pearlite and cementite. Ferrite is iron containing small amount of carbon and other elements in solution and is soft and ductile. Cementite, a compound of iron containing about 7 per cent carbon, is extremely brittle and hard. Pearlite is an intimate mixture of ferrite and cementite having a specific composition and characteristic structure, and physical characteristics intermediate between its two constituents.

The toughness and hardness of a steel that is not heat-treated depend on the proportion of these three ingredients. As carbon content of steel increases, the amount of ferrite present decreases and the amount of pearlite increases until when the steel has 0.8 per cent carbon, and then, it is entirely composed of pearlite. The various microstructures which may be obtained by heat treatment of normal steel when slowly cooled from the austenite range depend on the carbon content.

Plain carbon steel can be divided into four classes namely; Low carbon steel, medium carbon steel, high carbon steel and ultra-high carbon steels.

2.9.1 Low carbon steel

Low carbon steels contain up to 0.30 %C (by Wt). They possess good formability and excellent weldability. Low carbon steels exhibit poor response to heat treatment as a means for improving mechanical properties, particularly tensile and yield strength; the major micro-constituents being ferrite (Rajan, Sharma and Sharma, 1988).

During the freezing of low carbon steel, dendrites of δ-iron form, the composition of which is represented eventually by C (0.07%), and the liquid enriched in carbon crystals form austenite. The diffusion of iron occurs as the solid alloy cools to the ferrite solubility line, which denotes the commencement of precipitation of ferrite from austenite. Here α-ferrite commences to be ejected from the austenite, eventually the remaining solid solution is enriched in carbon until a point is reached at which cementite can also be precipitated. The alternate formation of ferrite and cementite at 625^{o}C gives rise to pearlite. The structure finally consists of masses of pearlite embedded in the ferrite.

The magnitude of difference in mechanical properties increases with rise in carbon content. Heat treatment processes are an integral part of the manufacturing and fabrication process of low carbon steel. Low carbon steels can be grouped into two classes:

1. Low carbon steels up to 0.10 per cent carbon and
2. Low Carbon steel with carbon from 0.10 to 0.30 per cent.

Low carbon steel with less than 0.10 percent carbon has excellent formability. For this reason these steels are employed for general engineering constructional work involving severe cold deformation such as bending, riveting and deep drawing. These steels, in form or cold-rolled sheets find applications in automobile, furniture and refrigeration industries. Annealing plays an important role during the manufacture of cold-rolled sheets. The manufacturing process essentially consists of a combination of cold-working and annealing treatments.

Steels with 0.10 – 0.30 per cent carbon possess high strength and toughness values. On case carburizing, these steels develop a hard and wear resistant case with tough core. Carburized steel is subjected to further heat treatment to refine case or core or both. These steels are very well suited for light engineering purpose such as welded structures, riveted structures, forgings and machine parts.

2.9.2 Medium carbon steel

Steels with carbon content varying 0.30 per cent to 0.60 per cent are referred to as medium carbon steels. The response of this steel to heat treatment is much better than that of low carbon steel. In order to have maximum advantage, these steels are often used in heat treated condition. Dendrites of austenite usually form during the cooling of medium

carbon steel and it finally freezes as cored solid solution, which on cooling through the critical range (750-695^{O}C) decomposes into ferrite and pearlite.

The first group consists of steels with 0.30 – 0.40 per cent carbon whereas the second group consists of steels with 0.40-0.60 per cent carbon. Normalizing treatment is employed for improving the machinability of steels included in the first group whereas the machinability of the second group of steel is increased by annealing. The steels in the second group are generally used in the hardened and tempered conditions. The desired levels of strength, ductility and toughness can be obtained by proper combination of hardening and tempering treatment for these steels.

Steels in the first group are suitable for moderately stressed components such as railway coupling, driving rings and flanges for bicycle, automobile and aircraft. Steels of the second group have higher strength than those of the first group, and for this reason, can be subjected to relatively higher stress. Typical applications include spindles of machine tools, gears, bolts, shaft, axle pinion, cylinders, riffle barrels and ball mills.

2.9.3 High carbon steel

The carbon content of high carbon steel generally varies between 0.60 to 1.20 percent. High carbon steel solidifies as cored solid solution at the beginning of the cooling stage but latter the cementite starts to be ejected and the steel becomes increasingly poorer in carbon until a point is reached when both cementite and ferrite form in juxtaposition. The structure then consists of free cementite and pearlite. The higher the carbon, the more is the strength with attendant brittleness. Therefore, steel with more than 1.3 percent carbon are rarely used in practice.

Machinability and formability of these steels can be improved to a greater extent by a heat treatment process known as spheroidizing annealing. Steels with 0.65–1.00 percent carbon are frequently used for manufacturing springs, gauges, machine knives, piston rings, saw, cutting tools.

2.9.4 Ultra high carbon steel

These steels are experimental alloys containing 1.20-2.0% carbon. They are thermo-mechanically processed to produce microstructures that consist of ultra-fine equiaxed grains of spherical discontinuous pro-eutectoid carbide particles.

They have fair formability, high hardness and wear resistance, poor hardenability and low toughness. They are not recommended for welding and are usually joined by brazing with low temperature silver alloy, making it possible to repair or fabricate tool-steel parts made with them without affecting their heat treated condition.

Their uses include punches for metal, rock drills, shear blades, cold chisels, rivet sets, springs, cutting tools, press tools, striking dies, taps, milling cutters, knives, cold cutting dies, wood working tools, files, and reamers. They are also used where a keen cutting edge is necessary such as razors, saws, and where wear resistance is important.

CHAPTER THREE

EXPERIMENTAL WORK

3.1 Materials

The materials used for this investigation were hot rolled plain carbon steels with varying carbon contents. The Chemical composition (wt %) and the structural conditions of the steel samples are as shown in table 3.1:

Table 3.1 Chemical composition and structural conditions of the steel samples.

Element	**Composition (wt %) and structural condition**					
	A	C	D	K	N	P
Carbon	0.100	0.220	0.390	0.540	0.810	1.040
Silicon	0.170	0.170	0.230	0.160	0.250	0.180
Manganese	0.370	0.600	0.600	0.72	0.660	0.600
Sulphur	0.026	0.041	0.024	0.033	0.049	0.038
Phosphorus	0.026	0.027	0.024	0.039	0.030	0.030
Structural Condition	Normalized at 900^{O}C	Normalized at 880^{O}C	Normalized at 860^{O}C	Oil quenched from 840^{O}C, Normalized at 550^{O}C	Normalized at 830^{O}C	Normalized at 880^{O}C

3.2 Heat treatment

Sample K, which was initially oil quenched from 840^{O}C and normalized at 550^{O}C, was normalized at 840^{O}C to bring it to the same heat treatment and structural condition with others samples, which were used directly without any initial heat treatment.

The heat treatment operation was performed at the Research and Quality Control Laboratory of Ajaokuta Iron and Steel Complex. The Laboratory is equipped with heat treatment facilities, which were found adequate for this investigation. The heat treatment was accomplished by charging the specimen into the furnace at room

temperature, heated to the desired temperature and held at this temperature for a desired period of time. They were then quenched in water at room temperature.

The furnace used was the Box-type resistance furnace with maximum temperature of 1200^{O}C, which was considered desirable for this research.

3.2.1 Determination of operating temperature

The annealing temperature AC_1 that defined the ferrite + austenite ($\alpha+\gamma$) region was determined by the Andrew's empirical formula (Gorni, 2004) as shown below:

$$Ae_1 = 723 - 10.7Mn + 29.1Si + 16.9Cr - 16.9Ni + 290As + 6.38W \quad \ldots\ldots 3.1$$

This temperature was found to be 723.99 for sample A. Therefore the inter-critical treatment was performed at 740 ^{O}C

3.2.2 Alternate thermal cycling

In this method the samples were heated alternatively in the austenite range and the two-phase ($\alpha+\gamma$) range with intermediate water quenching The austenitizing temperatures that were studied are 880^{O}C, 900^{O}C and 920^{O}C and the two phase range temperatures studied is 740 ^{O}C. The holding times were 5, 10 and 15 minutes and the numbers of cycles were 2, 4, and 6 cycles

3.2.3 Re-austenitization thermal cycling

Apart from the alternate thermal cycling treatments, re-austenitizing heat treatment was also carried out on other samples. Re-austenitizing thermal cycling consisted of repeated cycles of rapid austenitizing each of very short duration at a temperature sufficient to austenitize, and followed by intermediate water quenching, For samples austenitized at 900 ^{O}C, the holding times were 5, 10 and 15 minutes, and the numbers of cycles were 2, 4, and 6 cycles. The comparison of the two forms of thermal cycling is shown schematically in Figure 3.1

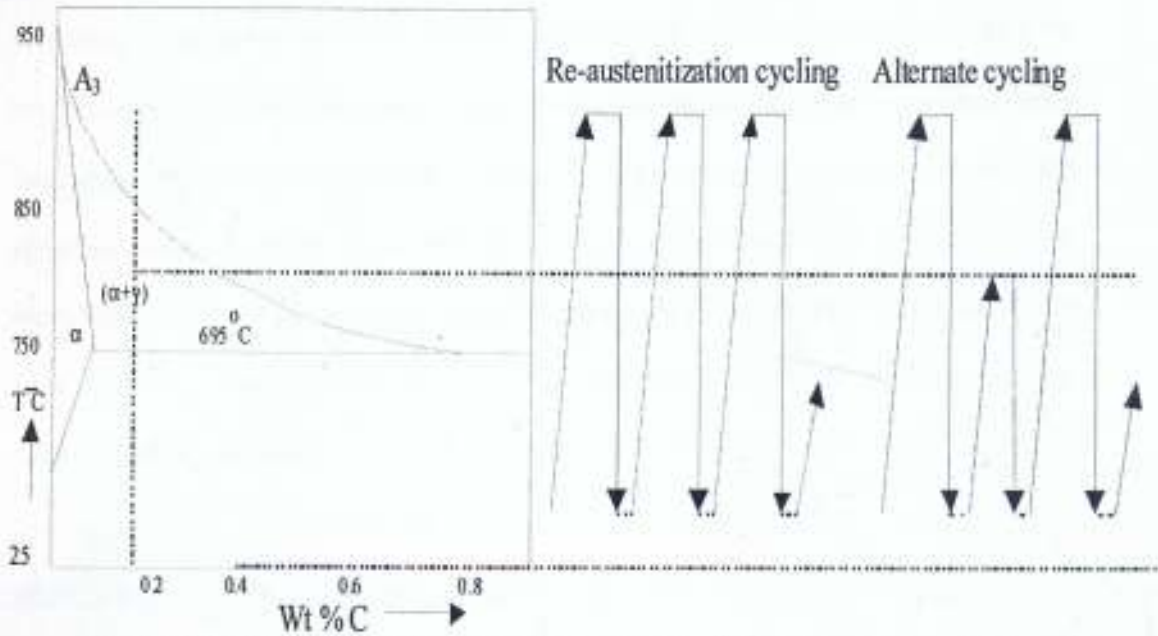


Fig 3.1 Schematic representation of cyclic re-austenitization in comparison with alternate cycling; referred to part of the FeC_3 equilibrium phase diagram

3.3 Tensile testing

The effect of thermal cycling on the tensile properties of the plain carbon steel samples was studied by preparing standard tensile samples from the as-received samples, subjecting them to various forms of heat treatment and testing in tension to failure on the Monsanto type W-type Tensometer. The initial gauge length and diameter were measured before subjecting them to tension. The yield and maximum loads were recorded directly from the tensile machine. At fracture the broken ends of the specimen were fitted together and the final gauge length and also the smallest diameter of the local neck were measured. The readings thus obtained were used in the determination of the following properties.

(i) Yield strength

(ii) Ultimate tensile strength

(iii) Percentage elongation

(iv) Percentage reduction in area

$$\text{Yield strength} = \frac{\text{Yield load}}{\text{Original cross-sectional area}} \quad \ldots\ldots 3.2$$

$$\text{Ultimate tensile strength} = \frac{\text{Maximum load}}{\text{Original cross-sectional area}} \quad \ldots\ldots 3.3$$

$$\text{Percentage elongation} = \frac{\text{Final gauge length-Original gauge length}}{\text{Original gauge length}} \quad \ldots\ldots 3.4$$

$$\text{Percentage reduction in area} = \frac{\text{Original area} - \text{Final Area}}{\text{Original Area}} \quad \ldots\ldots 3.5$$

3.4 Metallography

Specimens for the optical microscopic investigation were prepared by standard metallographic technique. Samples were sectioned using an abrasive cut-off machine. To enhance easy handling, the specimens were mounted in hot phenolic powder. The specimens were ground on a water lubricated hand grinding set-up of abrasive papers, progressing through from the coarsest to the finest grit sizes. The 240, 320, 400 and 600 grades were used in that order. Polishing was carried out on a rotating disc of a METASERVE universal polisher. Rough polishing was done with a synthetic velvet polishing cloth impregnated with 1 micron alumna paste. Final polishing was carried out with diamond paste. The specimens were then etched with the standard 2% nital so as to reveal the ferrite grain boundaries needed for the subsequent grain size measurement.

The optical microscopic examinations were carried out on an Olympus metallurgical microscope at a magnification of 100x. The specimens were illuminated with a 100 killowatts detachable quartz iodine lamp

3.5 Grain size measurement

The Jefferies Planimetric method (Vander Voort, 1999) was used to measure the grain sizes of the heat-treated samples. A circle of diameter 79.8mm was drawn on the micrograph and this defines an inscribed area of 5000mm^2. The number of grains completely inscribed in the circle and the numbers of grains intersecting the perimeter of the test area were counted. The number of grains per mm^2 was computed using equation 3.6

$$N_A = \frac{M^2}{5000}\left(n_1 + \frac{n_2}{2}\right) \quad \text{.....3.6}$$

where M = magnification of the photographic image,

n_1 = number of grains completely in the inscribed area and

n_2 = number of grains intersecting the perimeter of the test area.

The ASTM grain size number was then computed using the equation

$$G = [3.322 \log N_A] - 2.95. \quad \text{.....3.7}$$

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Result

4.1.1 *Tensile properties*

The tensile properties of the steel samples subjected to various forms of thermal cycling were assessed and the results are as contained in the Tables 4.1-4.8

Table 4.1: Yield strength of the steel samples after various numbers of alternate thermal cycling at constant temperature and holding time

			Yield strength (N/mm^2)			
Number of cycles			0	2	4	6
Samples	Temperature (^OC)	Holding time (Mins)				
A	900	5	405.06	415.19	420.11	384.81
C	900	5	384.81	390.21	396.30	394.84
D	900	5	486.07	490.11	497.82	495.55
K	900	5	433.88	439.82	445.74	440.36
N	900	5	415.19	420.34	424.18	420.91
P	900	5	495.87	499.98	503.24	500.11

Table 4.2 Ultimate tensile strength of the steel samples after various numbers of alternate thermal cycling at constant temperature and holding time

			Ultimate tensile strength / (Nmm^2)			
Number of cycles			0	2	4	6
Samples	Temperature (^OC)	Holding time (Mins)				
A	900	5	445.51	460.80	475.95	474.91
C	900	5	526.58	549.38	568.31	569.10
D	900	5	617.72	634.11	656.32	655.98
K	900	5	723.14	738.36	755.62	756.10
N	900	5	810.13	831.41	853.86	853.12
P	900	5	826.45	854.40	870.31	869.98

Table 4.3 Percentage elongation of the steel samples after various number of alternate thermal cycling at constant temperature and holding time.

Number of cycles			Percentage elongation (%)			
			0	2	4	6
Samples	Temperature (^{O}C)	Holding time (Mins)				
A	900	5	21.48	17.31	15.01	18.50
C	900	5	30.74	26.81	20.14	23.78
D	900	5	27.81	23.44	21.84	24.90
K	900	5	14.81	13.94	11.94	12.12
N	900	5				
P	900	5	18.15	15.73	13.82	14.74

Table 4.4 Percentage reduction in area of the steel samples after various number of alternate thermal cycling at constant temperature and time

			Percentage reduction in area (%)			
Number of cycles			0	2	4	6
Samples	Temperature (^{O}C)	Holding time (Mins)				
A	900	5	46.00	44.56	37.45	41.46
C	900	5	42.00	40.48	41.07	41.80
D	900	5	38.00	36.91	34.67	37.90
K	900	5	25.71	24.11	23.32	24.12
N	900	5				
P	900	5	28.57	25.55	25.41	27.10

Table 4.5 Yield strength of the steel samples after various numbers of re-austenitization thermal cycling at constant temperature and holding time

			Yield strength (N/mm^2)			
Number of cycles			0	2	4	6
Samples	Temperature (^oC)	Holding time (Mins)				
A	900	5	405.06	415.19	419.30	451.81
C	900	5	384.81	394.60	681.82	395.89
D	900	5	486.07	495.50	687.60	602.33
K	900	5	433.88	442.70	645.90	660.80
N	900	5	415.19	423.91	625.77	663.10
P	900	5	495.87	503.77	605.92	542.31

Table 4.6 Ultimate tensile strength of the steel samples after various number of re-austenitization thermal cycling at constant temperature and holding time

			Ultimate Tensile Strength (N/mm^2)			
Number of cycles			0	2	4	6
Samples	Temperature (^OC)	Holding time (Mins)				
A	900	5	445.51	906.33	516.48	542.17
C	900	5	526.58	624.40	729.67	775.32
D	900	5	617.72	770.81	916.91	882.96
K	900	5	723.14	1000.91	1406.47	1354.11
N	900	5	810.13	1212.74	1510.81	1394.38
P	900	5	826.45	1336.77	1618.72	1410.34

Table 4.7 Percentage elongation of the steel samples after various numbers of re- austenitization thermal cycling at constant temperature and holding time

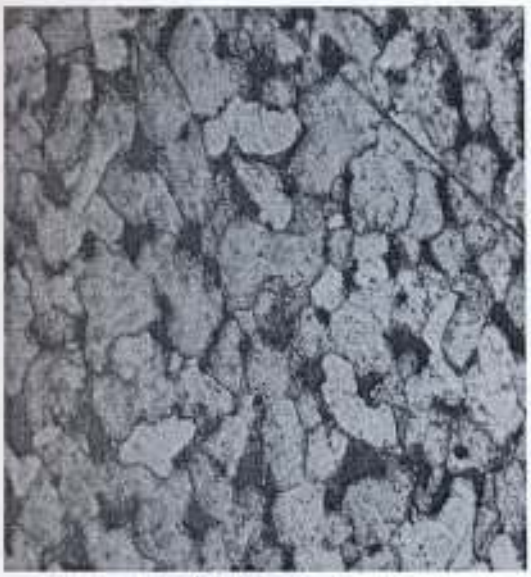
			Percentage elongation (%)			
Number of cycles			0	2	4	6
Samples	Temperature (^{O}C)	Holding time (Mins)				
A	900	5	21.48	18.30	18.01	15.50
C	900	5	30.74	27.71	23.14	20.78
D	900	5	27.81	24.44	24.48	21.90
K	900	5	14.81	14.94	3.99	12.12
N	900	5	24.81	22.94	21.46	20.93
P	900	5	18.15	16.73	15.80	13.82

Table 4.8 Percentage reduction in area of the steel samples after various number of alternate thermal cycling at constant temperature and holding time

			Percentage reduction in area (%)			
Number of cycles			0	2	4	6
Samples	Temperature (^{O}C)	Holding time (Mins)				
A	900	5	66.00	64.77	63.01	62.90
C	900	5	72.00	66.82	64.24	63.96
D	900	5	71.40	70.01	68.44	67.91
K	900	5	68.00	66.82	64.24	63.96
N	900	5	65.71	64.10	63.30	62.78
P	900	5	68.57	66.92	65.46	64.89

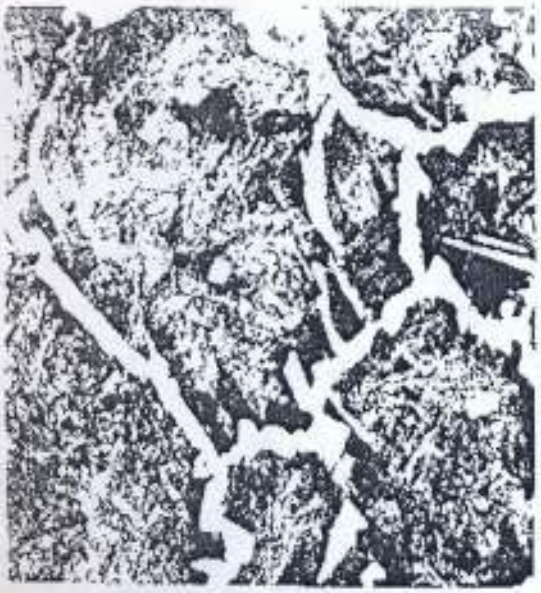
4.1.2 Photomicrographs

The photomicrographs of the steel samples before and after the various forms of thermal cycling treatments are shown in Plates 4.1 to 4.6:

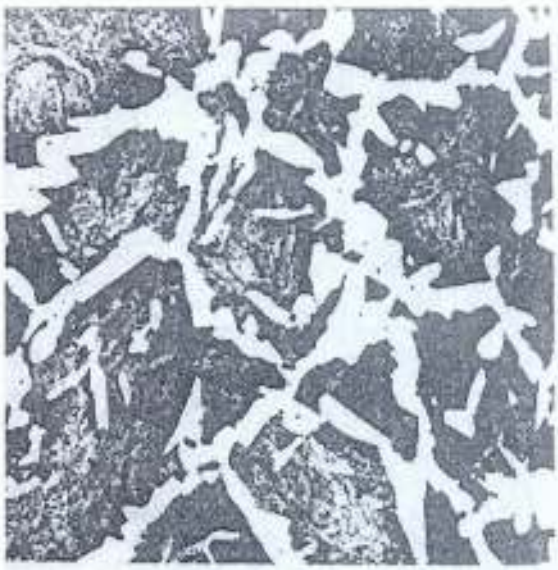


4.1 (a)

4.1 (b)



4.1 (c)



4.1 (d)

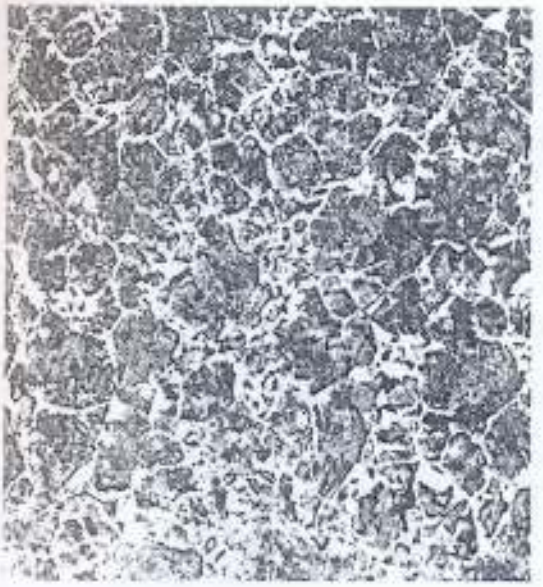
Plate 4.1: Optical photomicrograph of the steel samples before heat treatment

(a) A (0.10% Wt C) (b) D (0.45% Wt C)

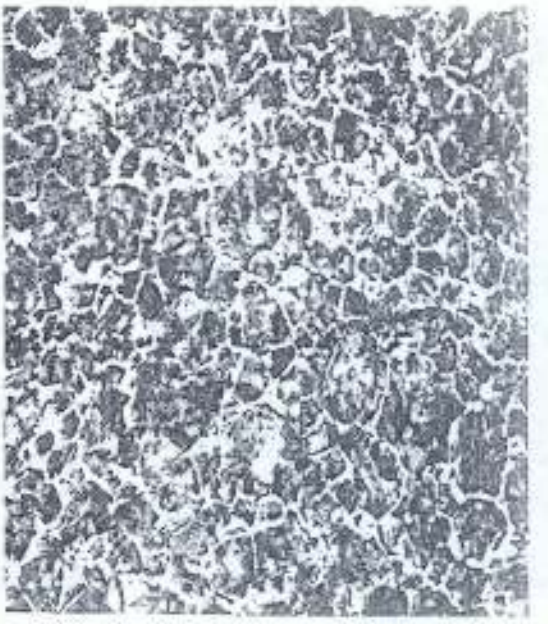
(c) N (0.81% Wt C) (d) P (1.04% Wt C)

Light areas are ferrite grains and dark areas the pearlite phase.

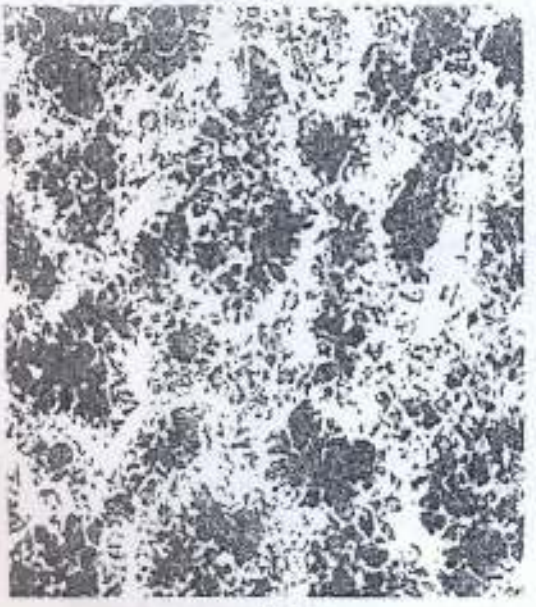
Etchant: 2% Nital X 100



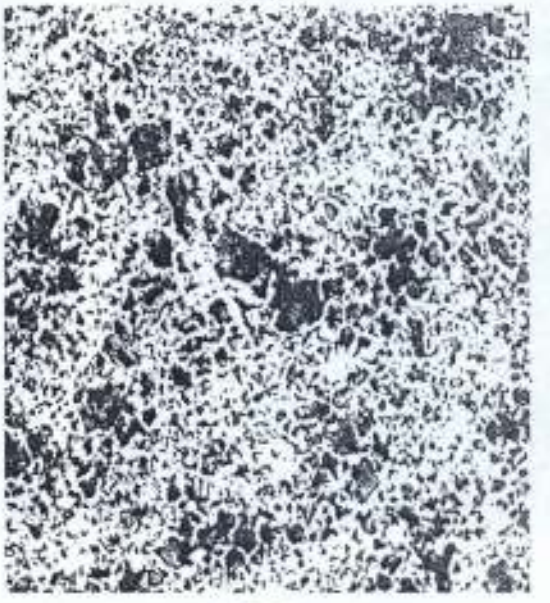
4.2 (a)



4.2 (b)



4.2 (c)



4.2 (d)

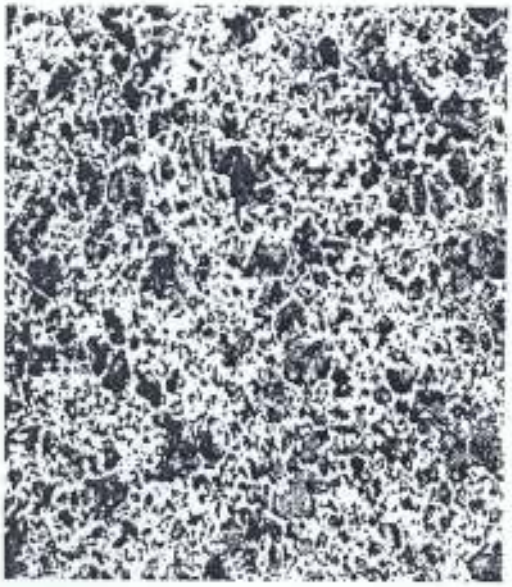
Plate 4.2: Optical photomicrograph of the steel samples after 2 cycles of alternate thermal cycling heat treatment at 900^{O}C and 740 ^{O}C with intermediate water quenching

(a) A (0.10% Wt C) (b) D (0.45% Wt C)

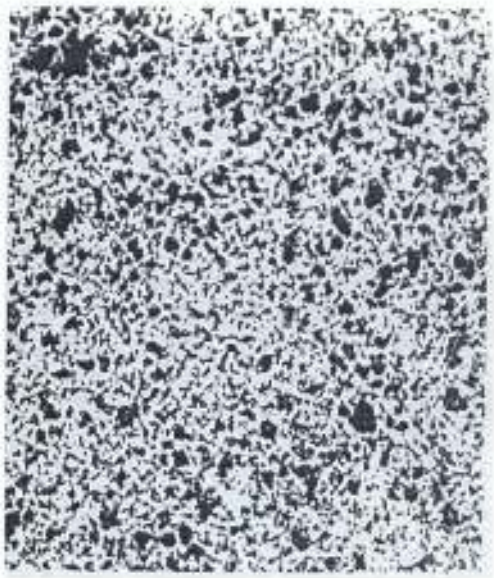
(c) N (0.81% Wt C) (d) P (1.04% Wt C)

Light areas are ferrite grains and dark areas are pearlite phase.

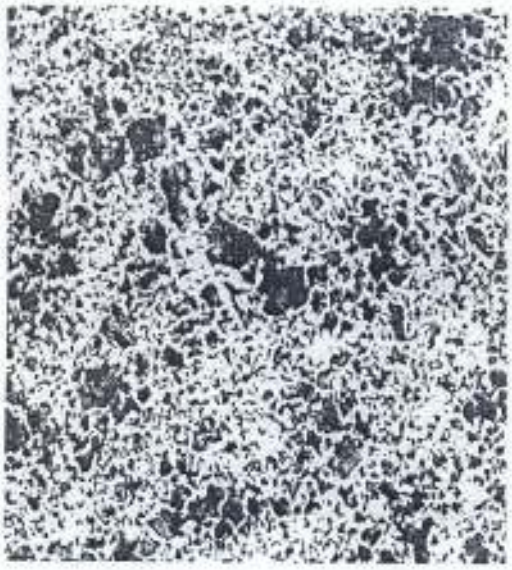
Etchant: 2% Nital X 100



4.3 (a)



4.3 (b)



4.3 (c)

4.3 (d)

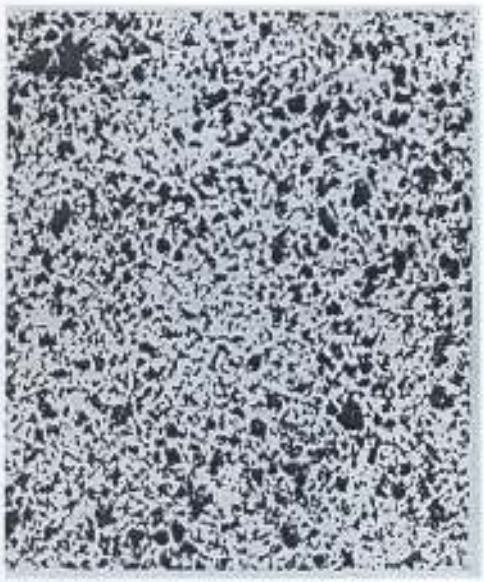
Plate 4.3: Optical photomicrograph of the steel samples after 4 cycles of alternate thermal cycling heat treatment at 900^{O}C and 740 ^{O}C with intermediate water quenching

(a) A (0.10% Wt C) (b) D (0.45% Wt C)

(c) N (0.81% Wt C) (d) P (1.04% Wt C)

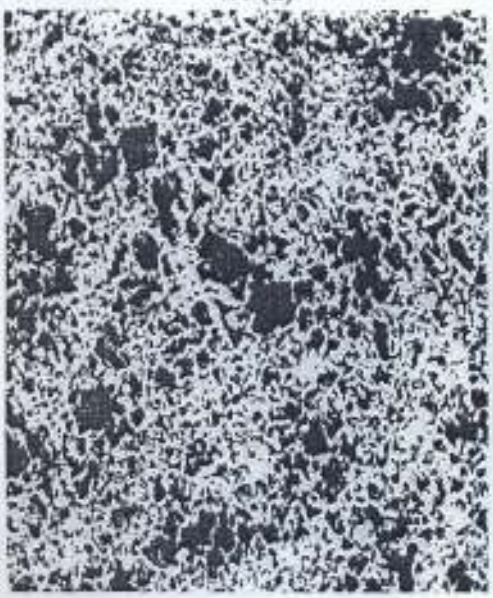
Light areas are ferrite grains and dark areas are pearlite phase.

Etchant: 2% Nital X 100

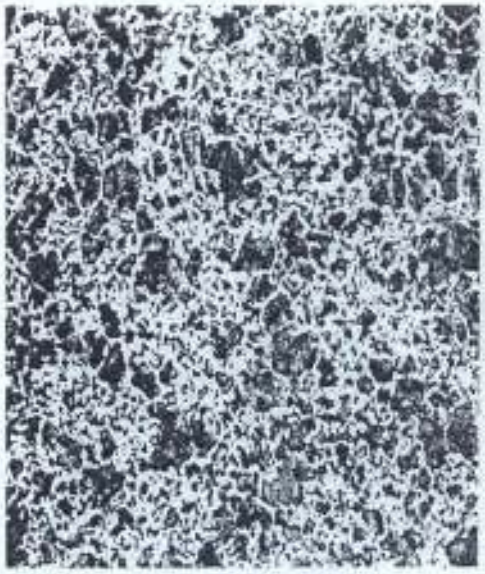


4.4 (a)

4.4 (b)



4.4 (c)



4.4 (d)

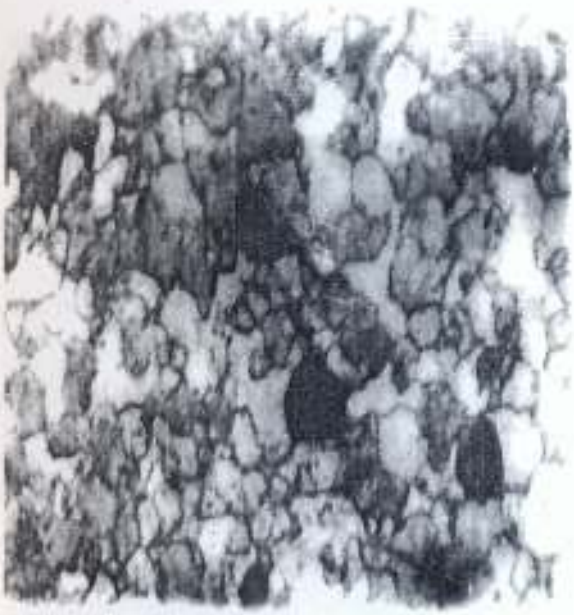
Plate 4.4: Optical photomicrograph of the steel samples after 6 cycles of alternate thermal cycling heat treatment at 900^{O}C and 740 ^{O}C with intermediate water quenching

(a) A (0.10% Wt C) (b) D (0.45% Wt C)

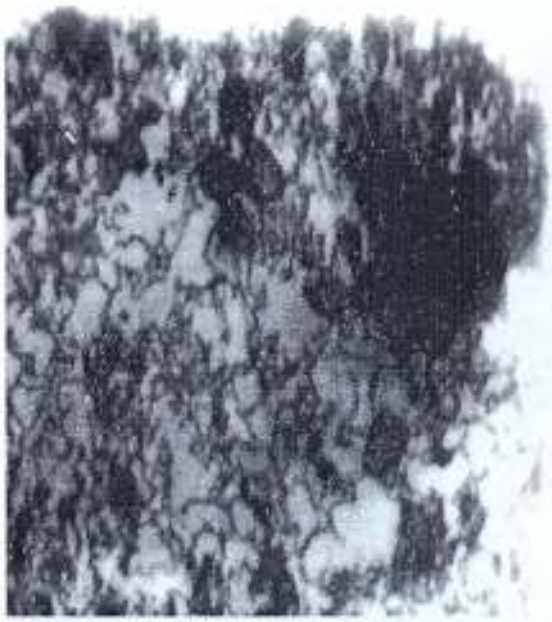
(c) N (0.81% Wt C) (d) P (1.04% Wt C)

Light areas are ferrite grains and dark areas are pearlite phase.

Etchant: 2% Nital X 100



4.5 (a)



4.5 (b)

4.5 (c)

4.5 (d)

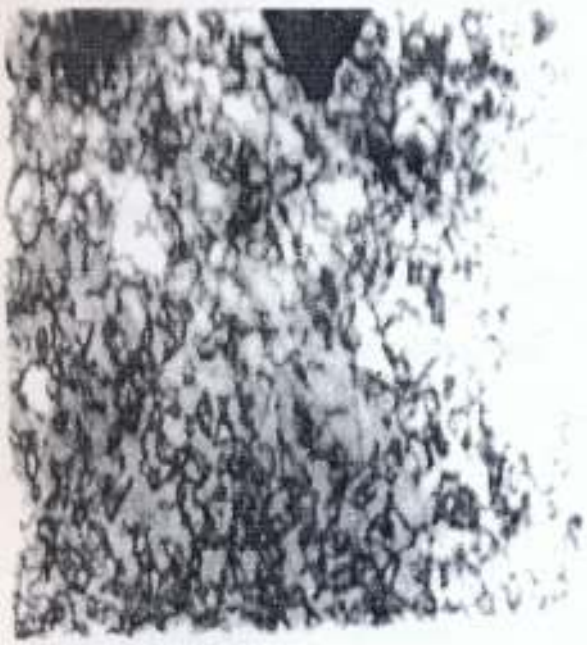
Plate 4.5: Optical photomicrograph of the steel samples after 2 cycles of re-austenitization thermal cycling heat treatment at 900^{O}C and 740 ^{O}C with intermediate water quenching

(a) A (0.10% Wt C) (b) D (0.45% Wt C)

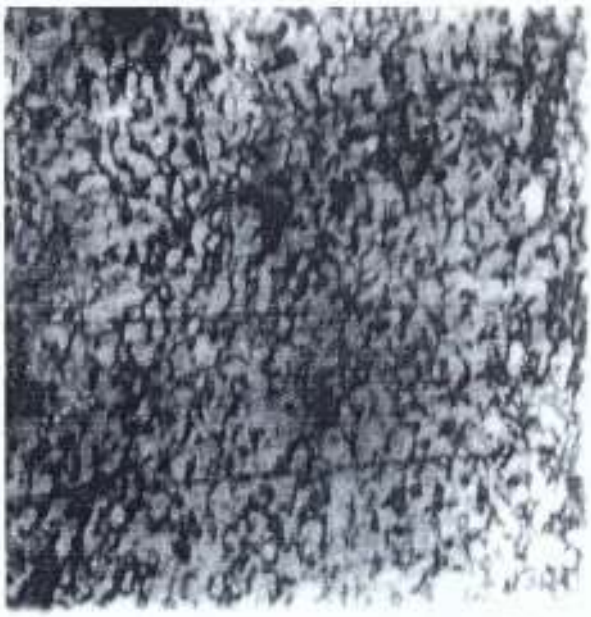
(c) N (0.81% Wt C) (d) P (1.04% Wt C)

Light areas are ferrite grains and dark areas are pearlite phase.

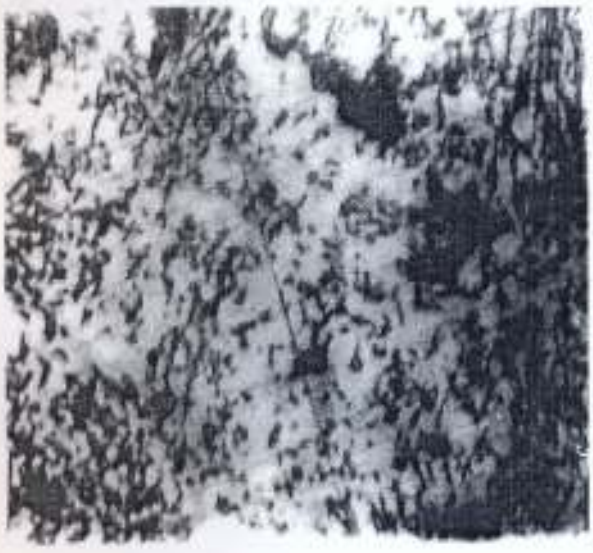
Etchant: 2% Nital X 100



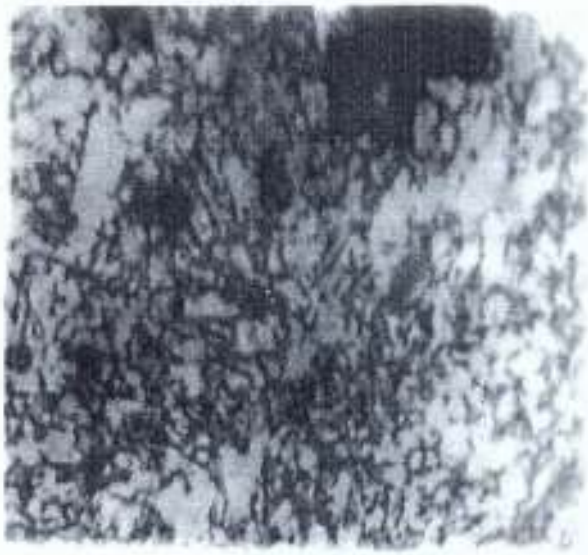
4.6 (a)



4.6 (b)



4.6 (c)



4.6 (d)

Plate 4.6: Optical photomicrograph of the steel samples after 4 cycles of re-austenitization thermal cycling heat treatment at 900^{O}C and 740 ^{O}C with intermediate water quenching

(a) A (0.10% Wt C) (b) D (0.45% Wt C)

(c) N (0.81% Wt C) (d) P (1.04% Wt C)

Light areas are ferrite grains and dark areas are pearlite phase.

Etchant: 2% Nital X 100

4.1.3 Grain sizes

The ferrite grain size was measured by the Jefferies Planimetric method as outlined in section 3.5 and the results are as contained in the tables below:

The effect of temperature of austenitization on the grain size after 2, 4, and 6 cycles are shown in tables 4.9-4.12

Table 4.9 Average grain size after 4 cycles of alternate thermal cycling at constant temperature and different holding times

			Grain size (mm) x10^{-3}			
Holding time (Mins)			**0**	**5**	**10**	**15**
Samples	**Temperature (^{O}C)**	**Number of cycle**				
A	900	4	12.50	11.00	11.40	11.70
C	900	4	12.80	10.50	11.10	11.40
D	900	4	12.85	10.35	12.20	12.60
K	900	4	12.18	10.11	10.50	10.90
N	900	4	12.60	10.42	11.40	11.80
P	900	4	12.30	10.80	11.70	12.10

Table 4.10 Average grain size after 4 cycles of alternate thermal cycling at constant holding time and different temperatures

			Grain size (mm) $x10^{-3}$			
Temperature (^{O}C)			0	880	900	920
Samples	Holding time (Mins)	Number of cycles				
A	5	4	12.50	11.50	11.00	11.31
C	5	4	12.80	10.90	10.50	10.60
D	5	4	12.85	10.70	10.35	10.40
K	5	4	12.18	10.62	10.11	10.40
N	5	4	12.60	10.80	10.42	10.60
P	5	4	12.30	11.36	10.80	11.00

Table 4.11 Average grain size for different numbers of cycles of alternate thermal cycling at constant holding time and peak temperature

			Grain size (mm) $x10^{-3}$			
Number of cycles			0	2	4	6
Samples	Holding time (Mins)	Temperature (^{O}C)				
A	5	880	12.50	11.90	11.50	11.61
C	5	880	12.80	11.40	10.90	10.98
D	5	880	12.85	11.74	10.70	10.72
K	5	880	12.18	11.44	10.62	10.71
N	5	880	12.60	11.73	10.80	10.89
P	5	880	12.30	11.46	11.36	11.64

Table 4.12 Average grain size after 4 cycles of re-austenitization thermal cycling at constant holding time and peak temperature

			Grain size (mm) $x10^{-3}$			
Number of cycles			0	2	4	6
Samples	Holding time (Mins)	Temperature (^{O}C)				
A	5	900	12.50	10.51	9.54	9.59
C	5	900	12.80	10.44	9.59	9.60
D	5	900	12.85	10.53	9.67	9.71
K	5	900	12.18	10.23	9.25	9.46
N	5	900	12.60	10.61	9.13	9.23
P	5	900	12.30	10.20	8.95	9.20

4.1.4 Hardness properties

The hardness of the steel samples subjected to the various forms of thermal cycling treatments was assessed. The tables 4.14 and 4.15 show the data obtained from the hardness measurement

Table 4.13 Brinell hardness number after various number of alternate thermal cycling at constant holding time and teak temperature

			Brinell hardness number		
Number of cycles			**0**	**2**	**4**
Samples	**Holding time (Mins)**	**Temperature (^{O}C)**			
A	5	900	100	124	144
C	5	900	120	137	174
D	5	900	141	160	177
K	5	900	152	168	190
N	5	900	161	180	223
P	5	900	173	193	250

Table 4.14 Brinell hardness number after various numbers of re-austenitization thermal cycling at constant holding time and peak temperature

			Brinell hardness number		
Number of cycles			0	2	4
Samples	Holding time (Mins)	Temperature (^{O}C)			
A	5	900	100	110	141
C	5	900	120	144	178
D	5	900	141	163	182
K	5	900	152	174	194
N	5	900	161	184	226
P	5	900	173	198	254

4.2 Discussion of result

4.2.1 Effect of temperature

Table 4.10 shows the average grain size obtained after four cycles of alternate thermal cycling at different temperatures. The dependence of grain size on the austenitizing temperature for different number of alternate cycles is shown in Figure 4.1. It was observed that for all numbers of cycles investigated, grain size initially appeared to decrease slightly with increased temperature up to a certain temperature, but above this temperature, the grain size increased at an appreciable rate with increasing temperature.

At temperatures below the optimum, the recrystallization of austenite is incomplete, giving a structure with a grain boundary that is unchanged before and after cycling, or a mixed grain structure of fine and large grains is obtained. The result in both cases is inefficient grain refinement. On the other hand, at temperature above the optimum, grain size increases with increasing temperature. This is a result of the free migration of grain boundaries to attain the equilibrium grain size when the temperature is raised. Grain growth is a thermally activated process, which depends both on time and temperature. The steels were observed to show gradual growth of all grains as the temperature is increased.

The fall in optimum temperature as the number of cycles is increased is due to the increasing grain growth tendency the finer the austenite grains become after each effective austenitizing cycle. The instability is observed to be greater with higher temperature thus the finer the starting microstructure, the lower will be the optimum temperature of austenitization.

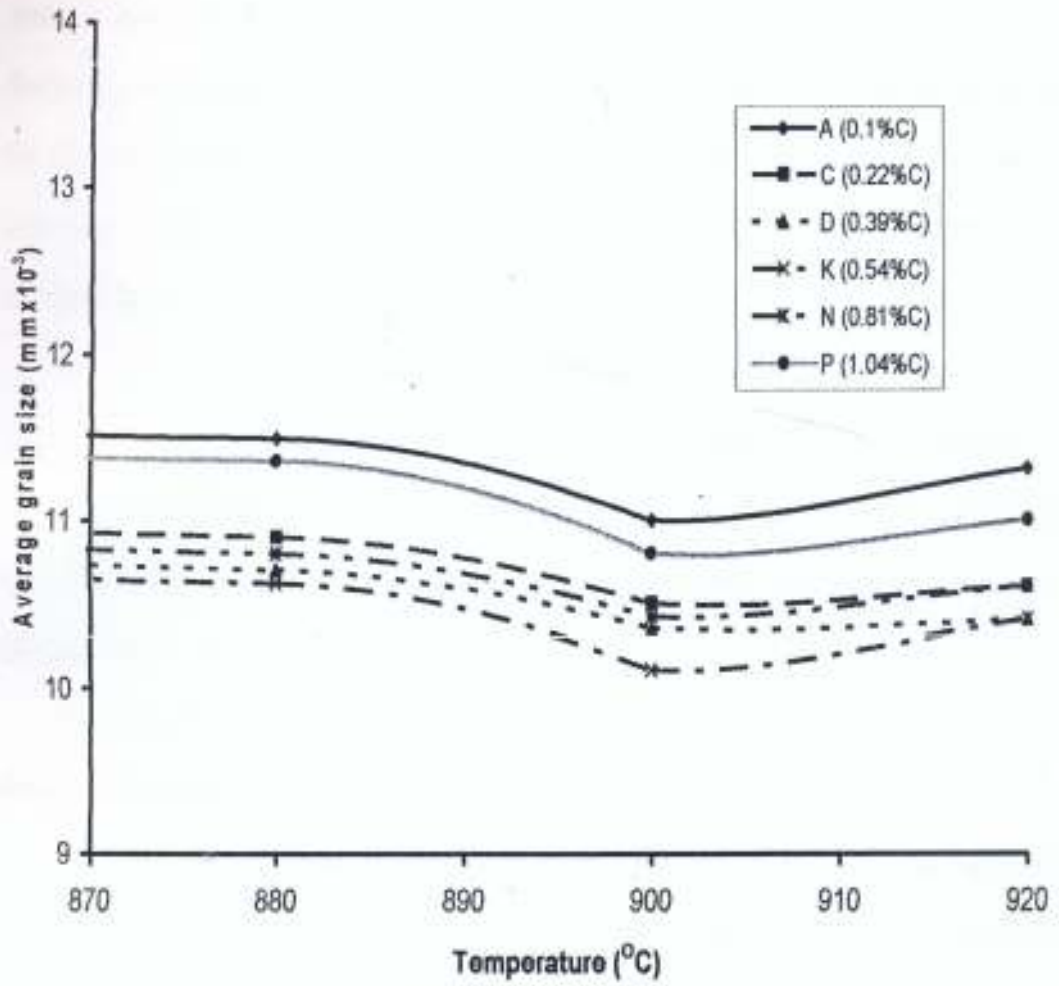


Figure 4.1: Effect of peak temoerature on grain size after 4 cycles of alternate thermal cycling

4.2.2 Effect of holding time

The average grain size obtained after four alternate cycles for different holding times is shown in Table 4.9. Figure 4.2 shows the effect of holding time at the peak temperature of austenitization for four alternate thermal cycles. The behaviors observed for the various carbon levels appeared to be alike. There was only a slight decrease in grain size with holding time of less than 6 minutes, but above this, the grain size increased significantly with increasing holding time.

This behaviour agreed with the established fact. Grain growth is a thermally activated process and the grain size, which is developed, depends both on temperature and time. Miller (1992), studied kinetics of grain growth in the austenite range and found that at any given temperature, the average grain diameter (D), depends upon time, according to the following equation.

$$D = Kt^n \quad \ldots\ldots 4.1$$

Where K and n are constants.

The austenite grain growth is soon inhibited by mutual grain impingement after long time of exposure at the peak temperature of austenitization. This explains why after 6 minutes, the rate of decrease in grain size with time slows down as shown in Figure 4.2. However, as evident from the curve, there appeared to be a steady increase throughout. This is due to the instability of very fine grains so that at impingement the unstable fine grains only dissolve into bigger grains and led to continuous, though slow increase in average grain size.

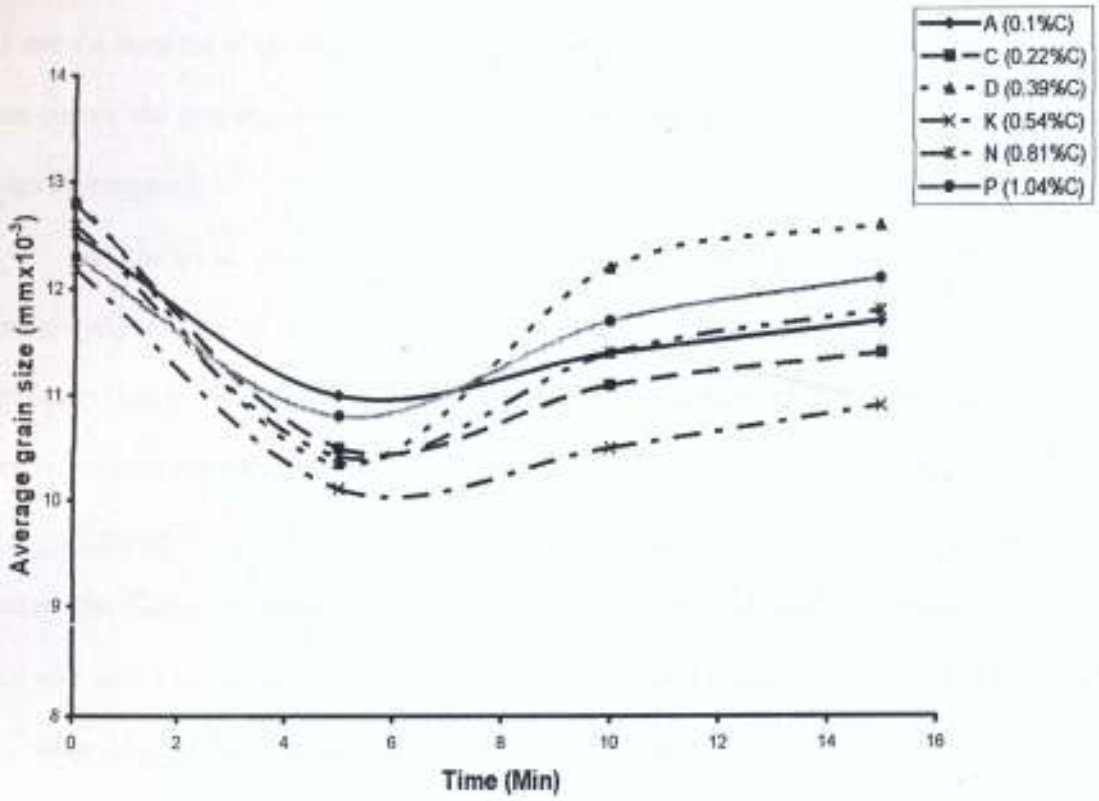


Figure 4.2: Effect of soaking time on grain size after 4 cycles of aternate thermal cycling

4.2.3 Effect of numbers of cycles

The results obtained after six cycles each of alternate thermal cycling and re-austenitization thermal cycling are shown in Tables 4.11 and 4.12 respectively. Figures 4.3 and 4.4 show the effect of the number of austenitization cycles on the grain size. In these curves, the temperature and time conditions were the same for all austenitization cycles investigated.

As shown in the figures, grain size decreased with increasing number of thermal cycle up to a certain number after which further cycling did not produce any significant changes in grain size. This behaviour is in agreement with the general observation from the earlier works.

Cycling is effective because, with each successive cycle up to a certain number, the number of austenite nuclei formed above AC_1 increases as the austenite grain size after each preceding cycle is smaller. With more nuclei, the austenite grain size, when impingement occurs, is smaller. After a limited number of cycles, the austenite grains become so unstable due to their small size, that rapid grain growth prevents additional refinement.

Refinement in the first cycle can also be attributed to multiple nucleation of ferrite in the large austenite grains and that in the following cycles, where the refinement effect was less, the growth of transformed regions both in the ferrite and in the austenite state seemed to determine the final grain size.

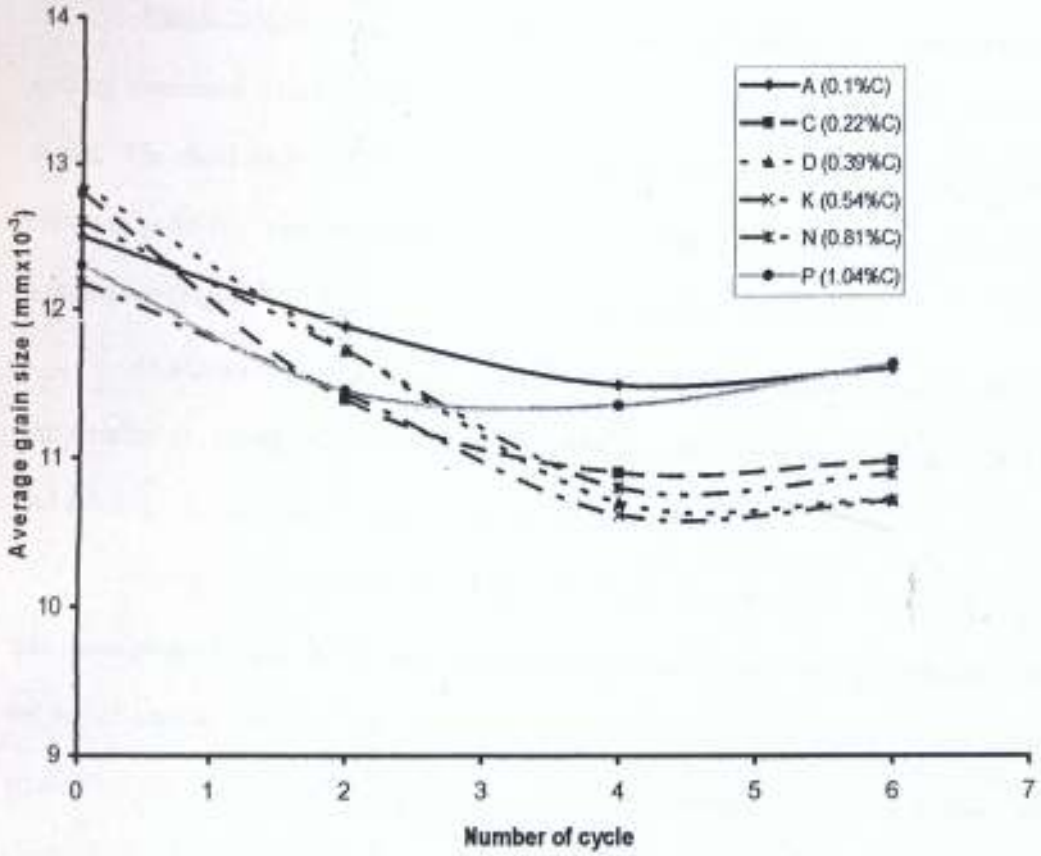


Figure 4.3: Effect of number of cycles on grain size at constant holding time and peak temperature

4.2.4 *Effect of carbon content*

Tables 4.9, 4.10 and 4.11 give the grain sizes obtained after the thermal cycling treatments while varying the peak temperatures, holding times and number of cycles. The dependence of grain refinement on the carbon content is shown on the curves of grain size versus number of cycle (Figure 4.4), grain size versus austenitizing temperature (Figure 4.5) and grain size versus holding time (Figure 4.6)

As shown in Figure 4.4 for 2, 4 and 6 cycles investigated, average grain size was smaller, the higher the carbon content. A similar behavior is observed in figures 4.5 and 4.6. Higher carbon content gave finer grain size.

However, as observed from Figures 4.4 and 4.6, the effect of carbon content was more pronounced at lower austenitization temperature and holding time, especially for lower carbon content. Also observed was the diminishing effect of the carbon content as the number of austenitizing cycles was increased beyond 4 cycles. This observation is obvious in Figure 4.4.

It was observed that for a given number of austenitizing cycles the average grain size decreased with increased carbon content for the hypoeutectoid steel with sample D (0.39% C) having the greatest refinement after four cycles (Figure 4.11). As shown in figure 4.11, for samples A (0.1%C), C (0.22%C), and D (0.39%C), grain size reduction was observed to more pronounced for steel with higher carbon content. This is probably due to the fact that cycling increases the number of very fine carbide which inhibits grain growth. However, for samples K (0.54%C), N (0.81%C), and P (1, 04%C) the reduction is less pronounced the higher the carbon content. This is probably due to higher growth tendencies the higher carbon contents.

The effect of carbon content however diminished with increasing number of austenitizing cycles. Also observed was the effect of carbon content on the number of

cycles required before the minimum grain size was obtained. It was noted that fewer number of cycles is required the higher the carbon content

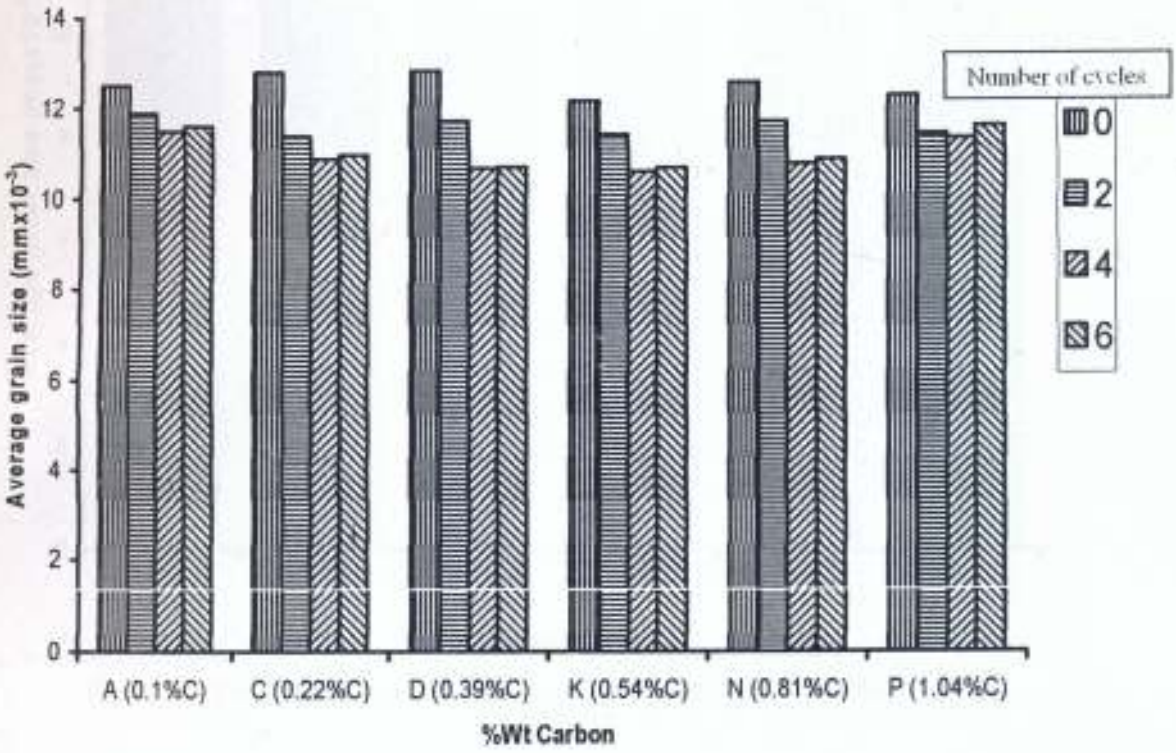


Figure 4.4: Effect of carbon content on grain size after various numbers of cycle of alternate thermal cycling

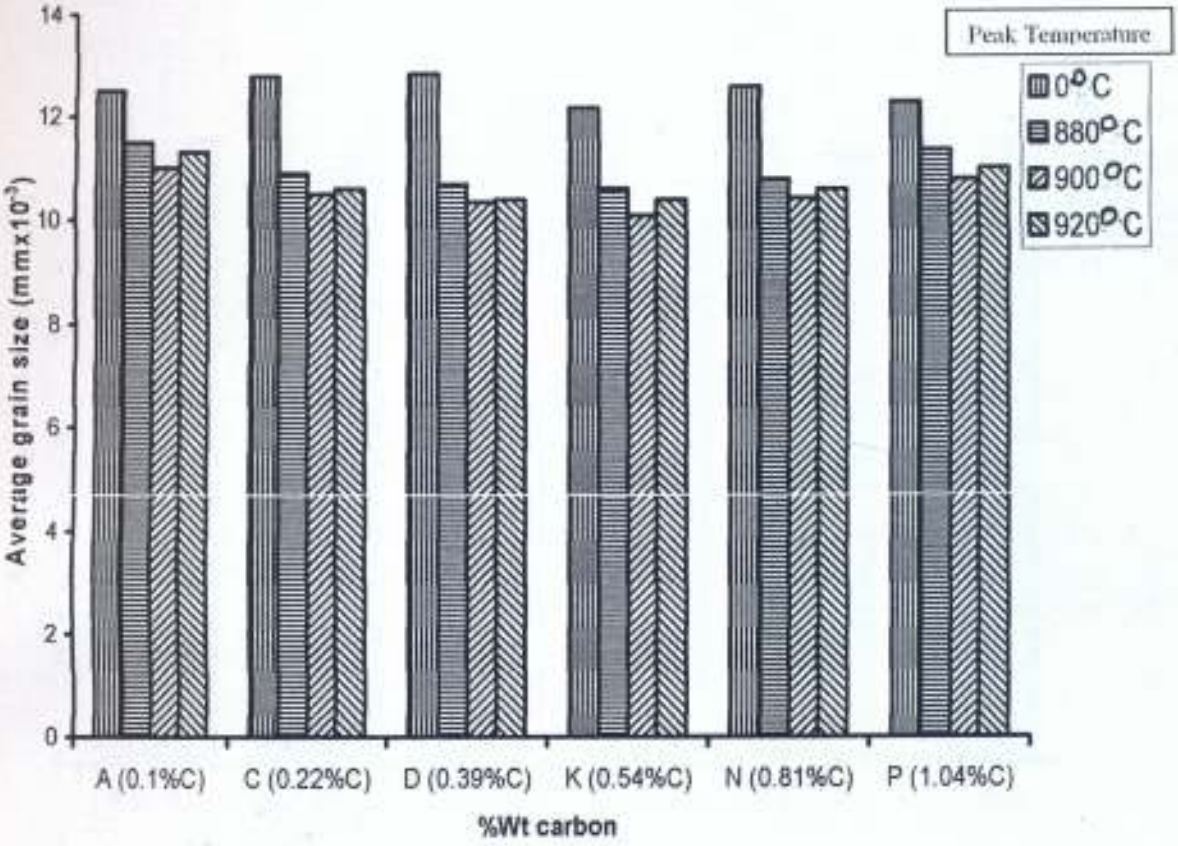


Figure 4.5: Effect of cabon content on grain size at different peak temperatures

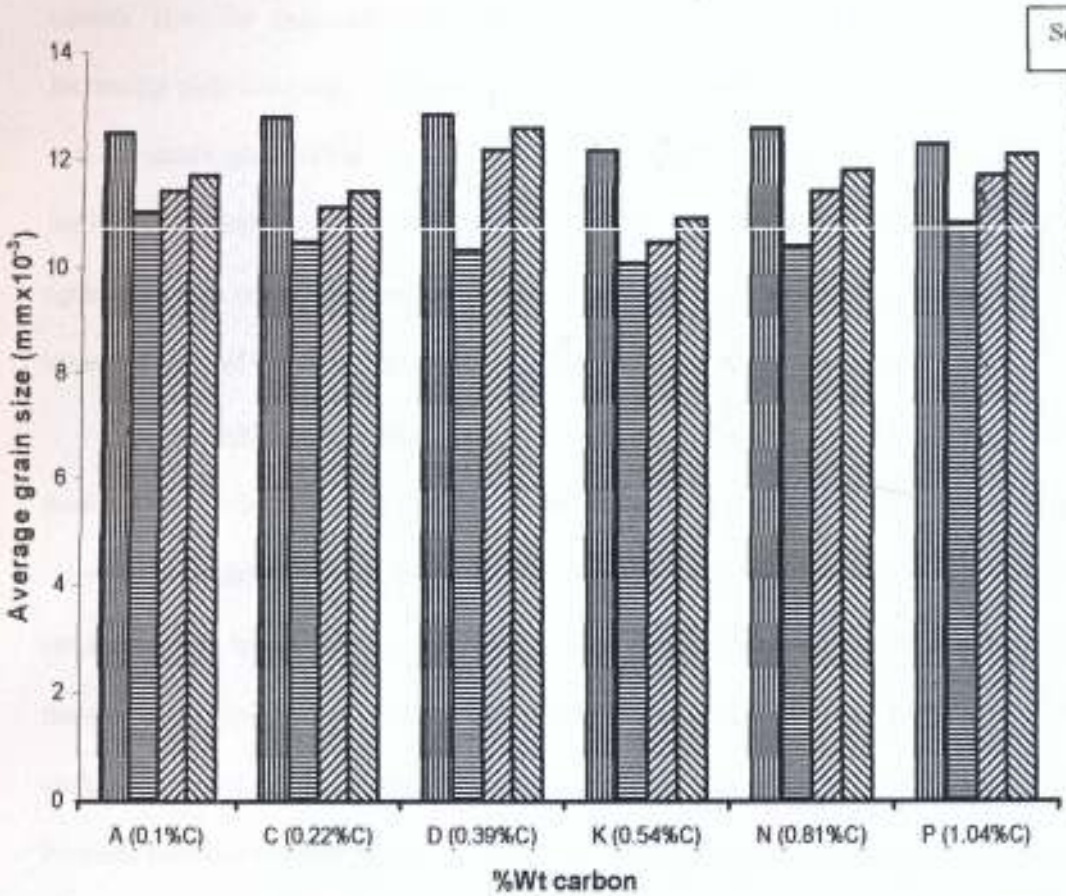


Figure 4.6: Effect of carbon content on grain size for varios holding times at 900^{O}C

The increasing degree of grain refinement observed with increasing carbon content (for the hypoeutectoid samples) conformed to certain established fact. Increasing carbon content decreases the austenite to ferrite transformation temperature and so causes grain refinement. Carbon also increases hardenability of steel and so facilitates formation of martensite during cooling from the austenite phase, thus the finer grain size obtained from higher carbon content and the general increase in grain size regardless of the carbon content as temperature and holding time increased.

The diminishing effect of carbon content noted as the temperature and holding time increased was as a result of the increasing growth tendency as the temperature is raised or the holding time is increased. In cycling, with each successive cycle up to a certain number, depending on the grain growth characteristic of the various steels, the number of austenite nuclei formed above AC_1 increases if the austenite grain size after each preceding cycling is smaller. In other words, there must be a direct relation between austenite nucleation and the finess of the microstructure being rapidly heated.

According to VanAken, (2003), strengthening in Fe-C is also a combination of solute strengthening and a refinement in grain size. The strength of martensite increases proportionally to the square root of the carbon content. For sample A and C, where carbon content is less than or approximately equal to 0.2%wt, increase in strength is probably due to carbide precipitation while in samples D, K, N, and P, the increase in strength is obtained in the as-quenched, martensitic condition i.e. solute strengthening is greater than that obtained by carbide precipitation.

4.2.5 Tensile properties of the steel samples

The tensile properties of the steel samples after various numbers of cycles of alternate and re-austenitization cycling are shown in Tables 4.1 and 4.8. Figures 4.7 and 4.8 present the yield and ultimate tensile strengths of the steel samples as a function of

the number of alternate thermal cycles. The percentage elongation and percentage reduction in area after various numbers of alternate cycles are presented in Figures 4. 9 and 5.10 respectively.

Figures 4.11 and 4.12 give the yield and ultimate tensile strengths of the steel samples as a function of the number of rapid austenitization cycling. The percentage elongation and percentage reduction in area after four re-austenitization cycles are presented in Figures 4.13 and 4.14 respectively.

As can be seen from Figures 4.7, 4.8, 4.11 and 4.12, yield and ultimate tensile strength increased with increasing number of cycles of alternate cycling for all the steel samples up to four cycles after which further cycling did not produce any observable increase in strength. This observation is consistent with the changes in grain sizes as can be observed in Figure 4.3

The explanation of this behaviour is found on the dislocation theory. Hall and Petch, (1953) empirically established that the tensile stress is related to grain size by

$$\sigma_o = \sigma_1 + Kd^{-1/2} \quad \ldots\ldots 4.2$$

Where:

σ_o = flow stress

σ_1 = frictional stress porosity motion of dislocation

K = unpinning constant measuring the extent to which dislocations are pilled up at barriers

d = grain diameter.

The flow stress σ_o increases with dislocation density because dislocations pile up on slip planes at barriers in the crystal and these pile-ups will produce a back stress, which opposes the applied stress on the slip plane Brandon and Nutting (1960), have observed

that the dislocation density is an inverse function of the grain size. The finer the grain size, the higher the dislocation density, and hence, the higher the strength.

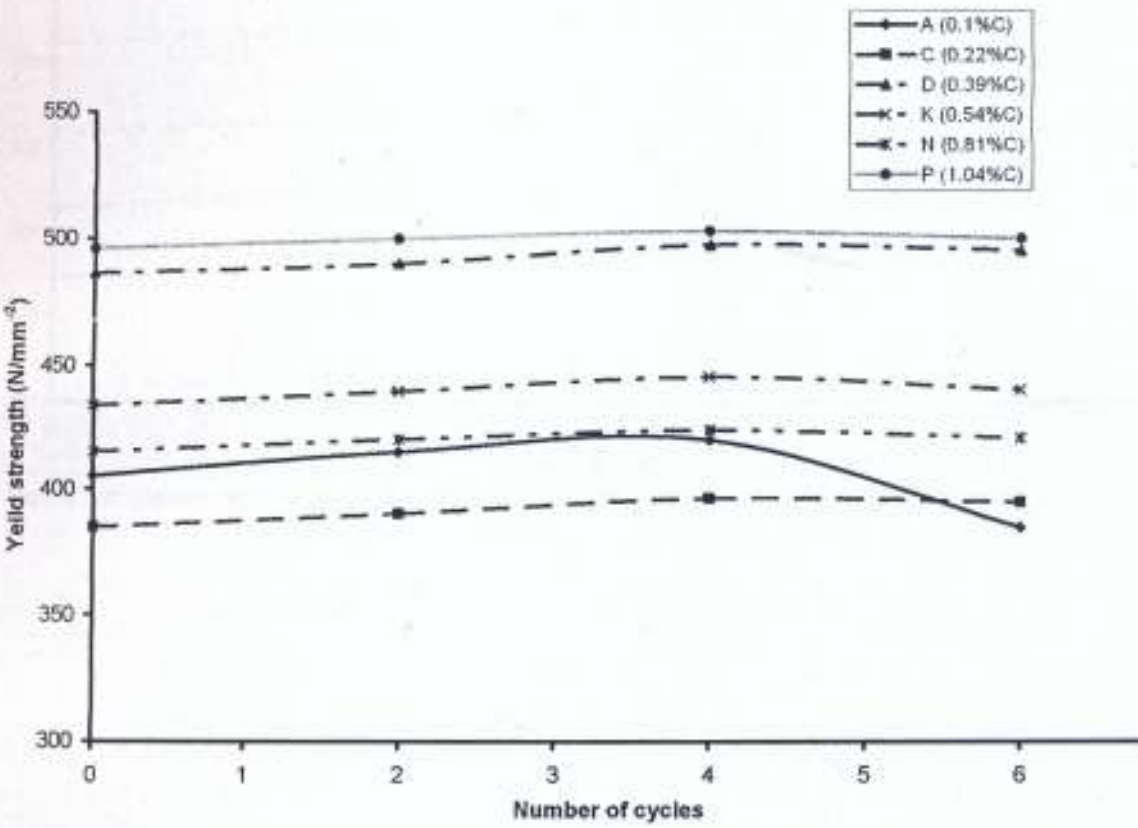


Figure 4.7: Yield strength of the steel samples after six cycles of alternate thermal cycling

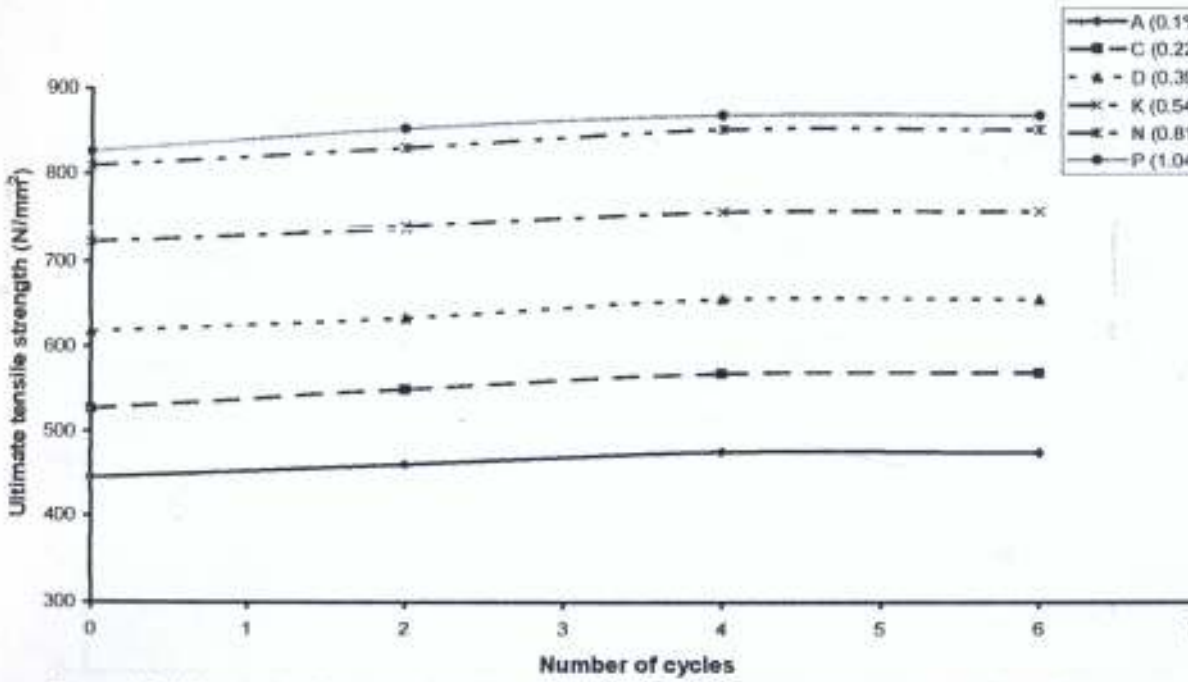


Figure 4.8: Ultimate tensile strength of the steel sample after six cycles of alternate thermal cycling

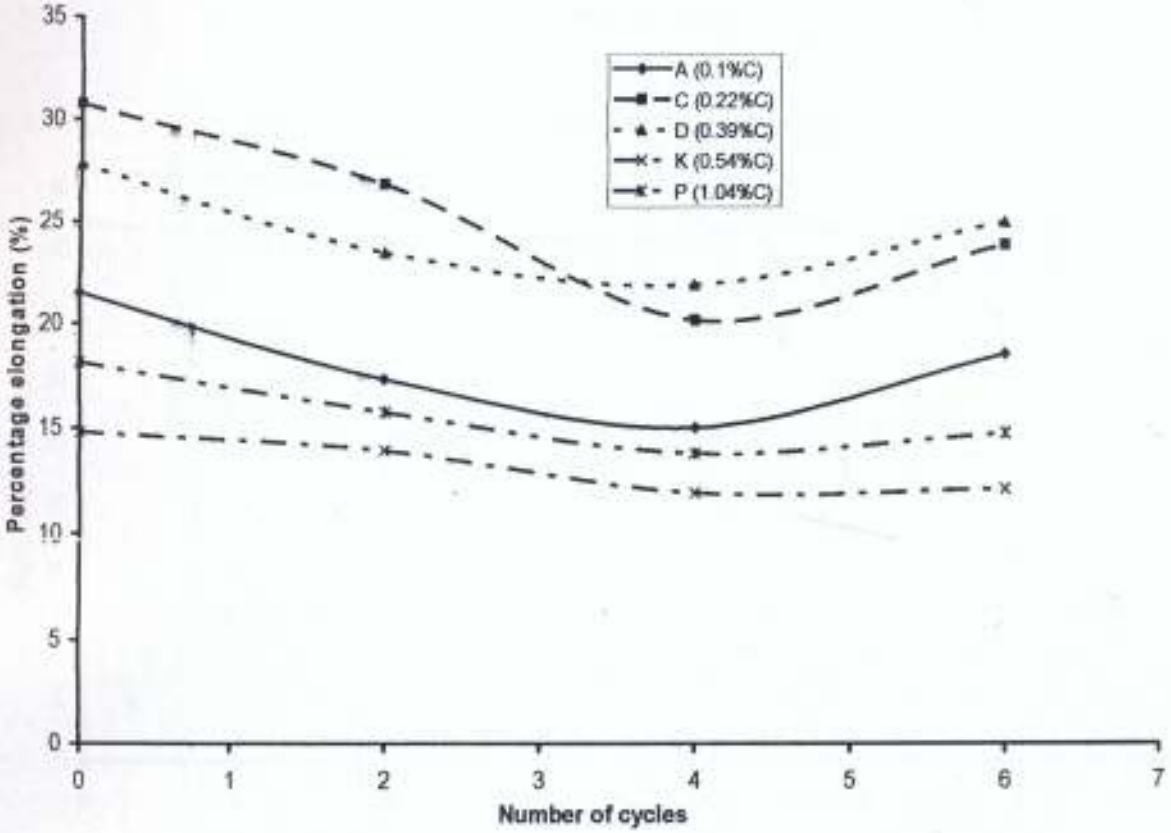


Figure 4.9: Percentage elongation in area of the steel samples after six cycles of alternate thermal cycling

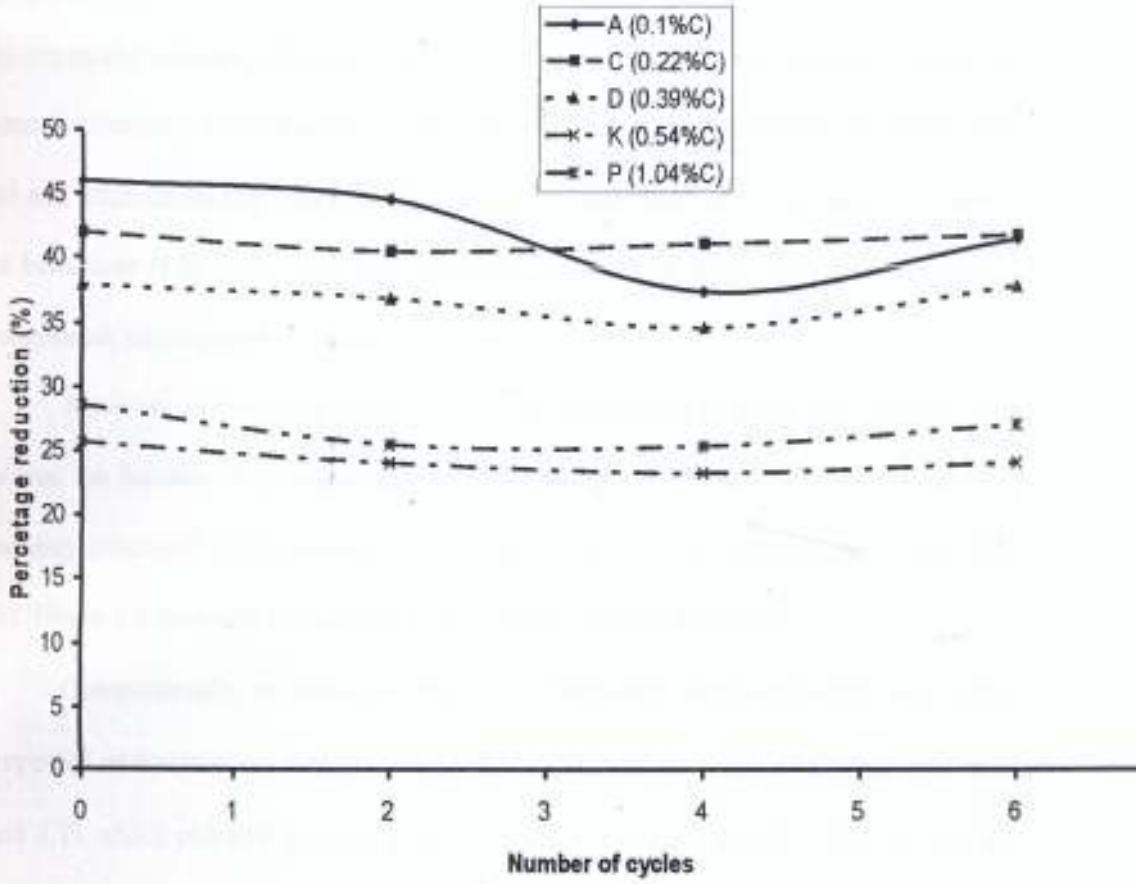


Figure 4.10: Percentage reduction in area of the steel samples after six cycles of alternate thermal cycling

The ductility, as shown in Figures 4.10 and 4.14 decreased for all the samples with increasing number of cycles up to 4 cycles. The higher the carbon content the greater the decrease in the ductility. These changes were consistent with the changes in yield and ultimate tensile strengths. The ductility decreased as the strength increased. This behaviour is in order with the generally known fact, that increasing strength is accompanied, usually with decreasing ductility.

However it would be expected that the refinement in grain size should have improved the ductility. It is suspected that the effect of brittle martensite resulting from quenching treatment predominated. Thus higher the carbon content, the more this effect. Hence the decrease in ductility as the carbon content increases.

Comparatively, as shown in Figure 4.12 ultimate tensile strengths are higher for repeated austenitization than for alternate thermal cycling. This is in agreement with Figure 4.11 which presents average grain sizes after the same number of cycles for the two cycle patterns. On the other hand, some improvement in ductility was observed with alternate thermal cycling than repeated austenitization cycling.

These observations are better explained by considering the well-known rule of two mixtures using the following equations:

$$\sigma_{uc} = \sigma_{uf} v_f + \sigma_m (1 - V_f) \quad \text{4.3}$$

Where:

σ_{uc} - tensile strength of thermally cycled specimen

σ_{uf} - tensile strength of martensite

σ_m - stress of the matrix (ferrite) at the ultimate tensile strain of martensite

V_f - volume fraction of martensite.

According to Koo and Thomas, 1976, if both σ_m and σ_{uf} are the same for sample of steel treated by austenitization, and those by alternate thermal cycling, σ_{uc} would be higher

for the repeated austenitized samples on account of the higher volume fraction of the higher strength martensite. In the same vein, the ductility is better in samples treated by alternate thermal cycling because of smaller volume fractions of hard and brittle martensite.

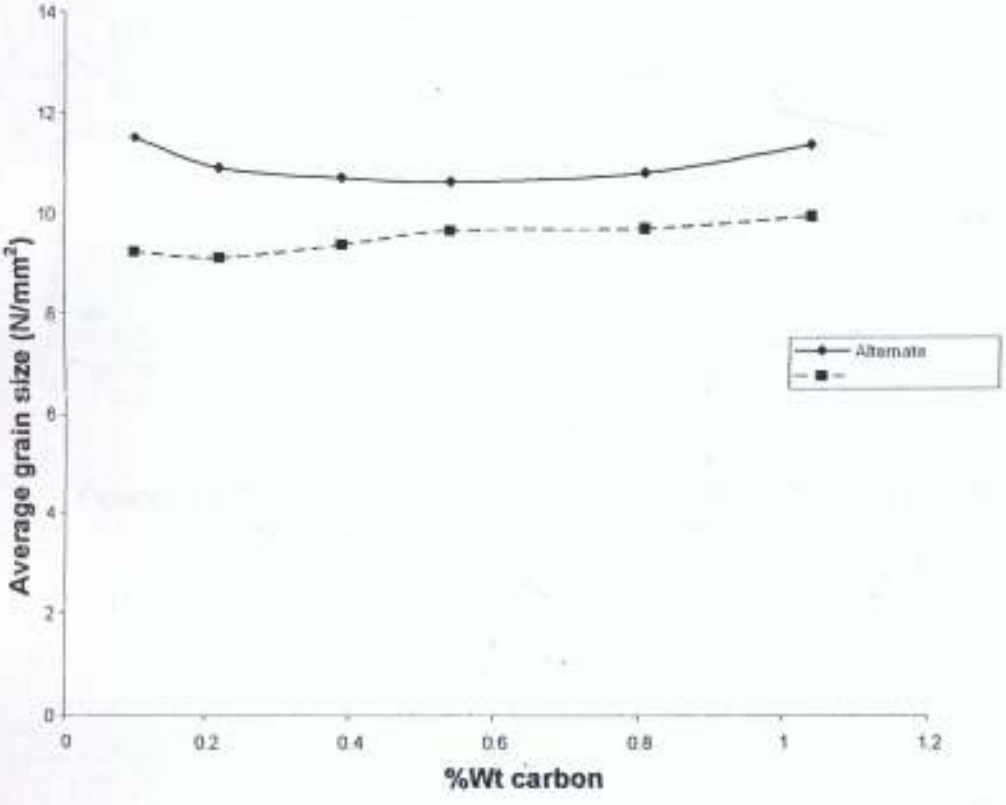


Figure 4.11: Average grain size after 4 cycles of alternate and re-austenitization thermal cycling at 900°C

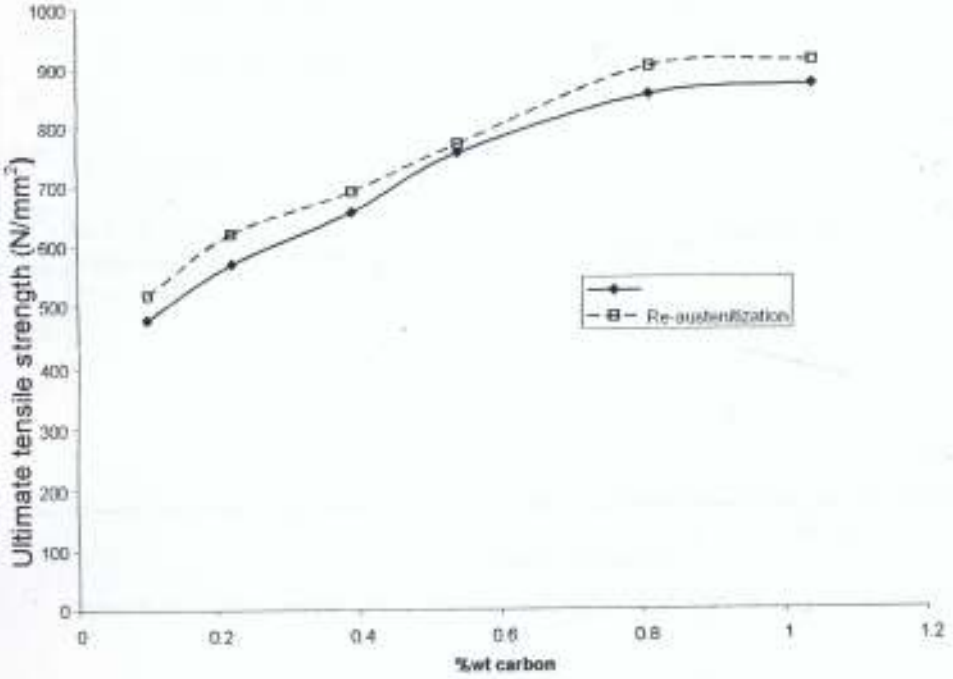


Figure 4.12: Ultimate tensile strength after 4 cycles of alternate and re-austenitization thermal cycling at 900°C

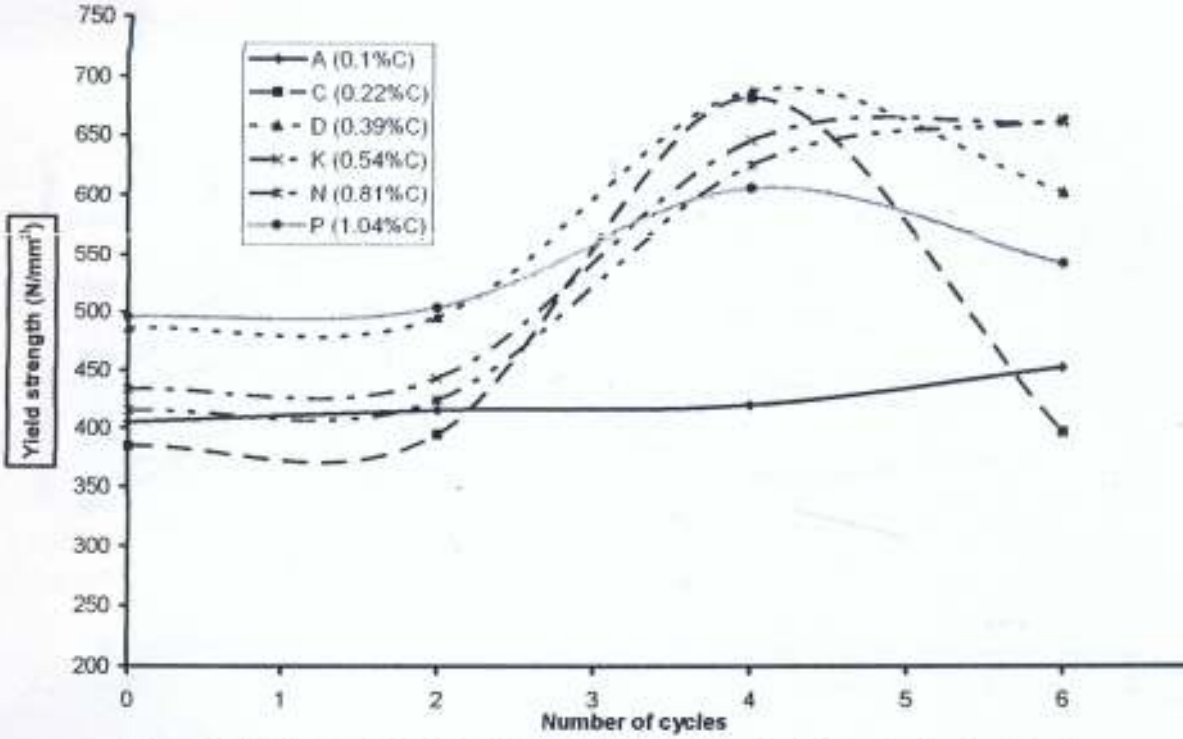


Figure 4.13: Yield strength of the steel samples after six cycles of reaustenitization themal cycling

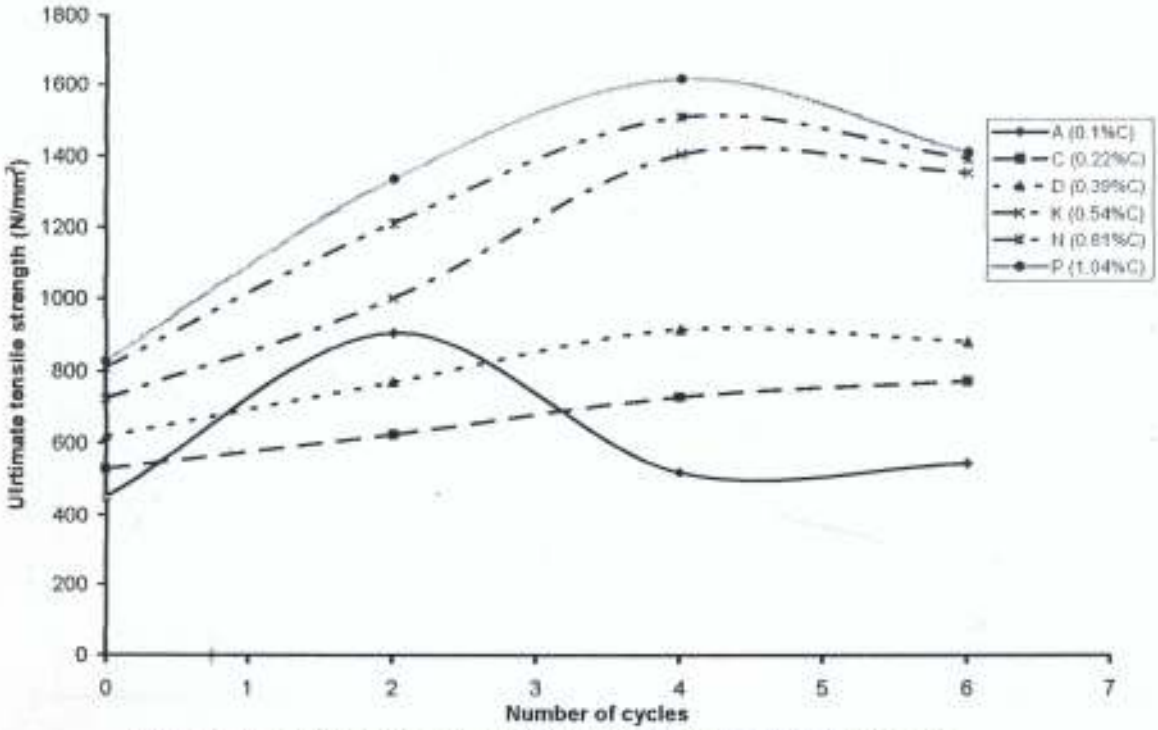


Figure 4.14: Ultimate tensile sttrength of the steel samples after six cycles of re-austenitization thermal cycling

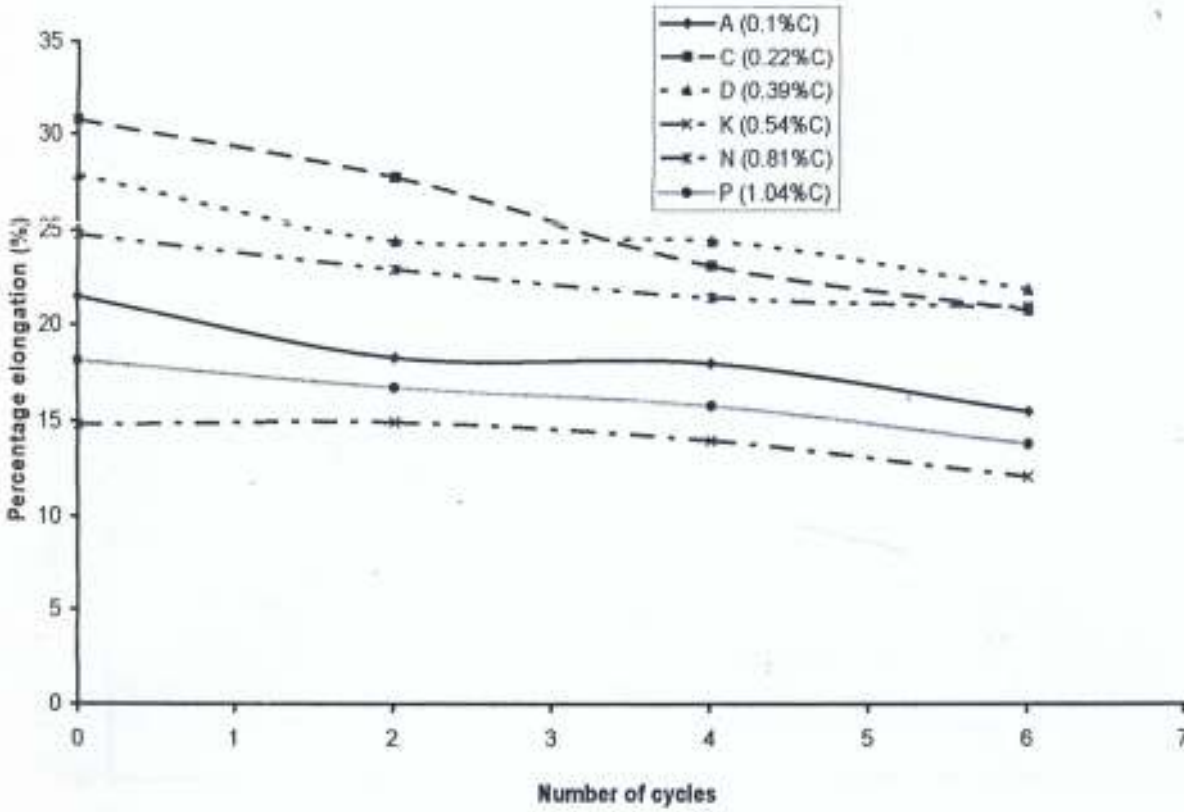


Figure 4.15: Percentage elongation of the steel samples after six cycles of re-austenitization thermal cycling

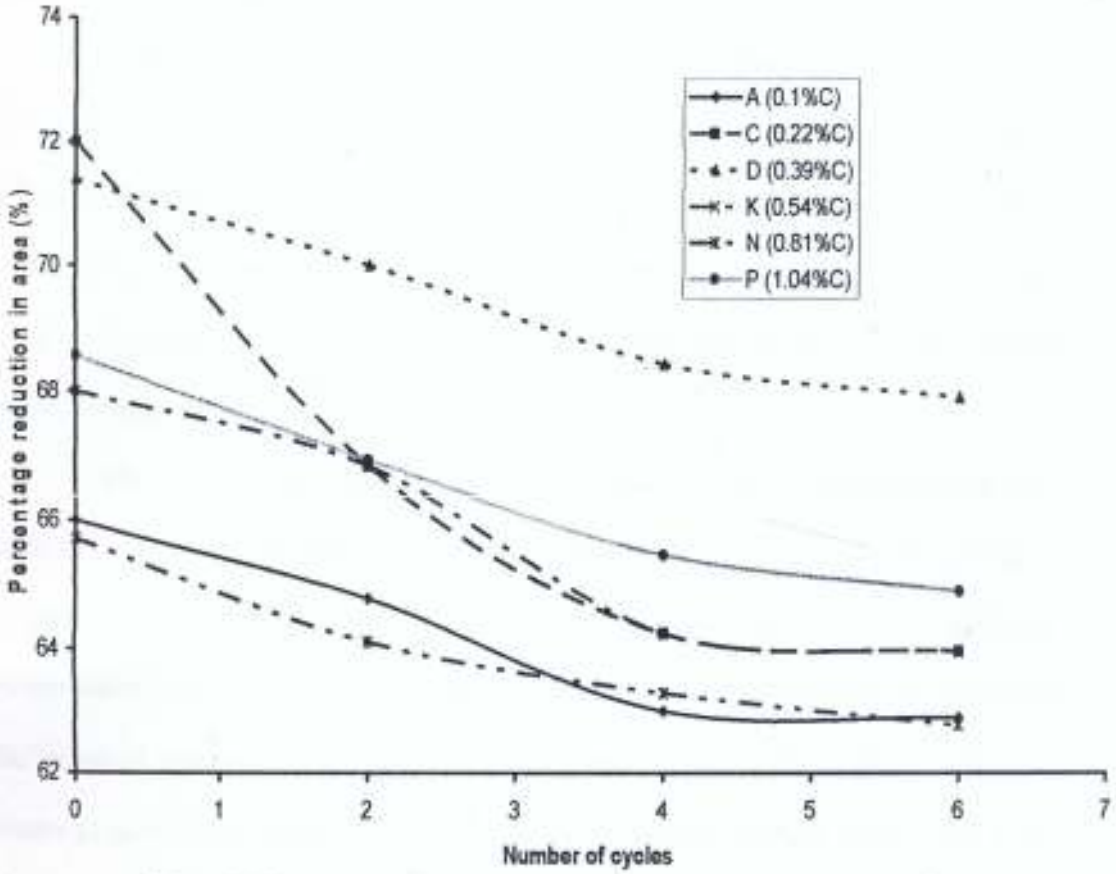


Figure 4.16: Percentage reduction in area of the steel samples after six cycles of re-austenitization thermal cycling

4.2.6 Microstructure

Hypoeutectoid steels have low hardenability, thus proeutectoid ferrite followed by precipitation of (α + carbide), according to the $\gamma = \alpha$+ carbide reaction, should be expected at high transformation temperature upon quenching. The observed microstructures of samples A and C agreed with the above stated fact. As shown in the optical micrograph displayed in plate 4.1 to 4.6 ferrite and pearlite were the notable structures in these samples

Martensite is observed to be present in the micrographs of hypereutectoid steel samples (D, K, N and P). They were found to contain ferrite, martensite and carbide, in varying amounts which probably depend on the type of thermal cycling treatment, carbon content and the number of cycles. Ferrite phases were observed to increase with the increased number of cycles up to a point when further cycling did not affect the phases proportion any longer. It was also observed that the changes were found to be more pronounced with the re- austenitization thermal cycling than the alternate thermal cycling process.

The increased fraction of ferrite with increased number of cycle is explained by the fact that cycling refines grain sizes and the number of austenite nuclei formed above AC_1 increases, as the austenite grain size after each preceding cycle is smaller. Austenite grains transforms to several ferrite grains on cooling, therefore the finer the prior austenite grains the higher the rate of ferrite transformation at high transformation temperatures upon quenching. Thus, the observed increase in proportion of ferrite as the size of austenite decreased with increased number of cycles.

Higher proportion of ferrite was observed with increase in the carbon content. This is because martensite increases with increase in the carbon content and with

number of cycles. The observed changes in tensile properties of the steel samples as earlier reported confirmed the above observation.

Increase in tensile strength with increased number of cycles was not only due to decreasing grain size but also due to some other morphological changes in the constituent phases of the steel samples.

The higher the carbon contents of the steel, the higher the carbon contents of the austenite and the higher the tendency of martensite formation. The diffusion of carbon from the carbon-enriched constituents into the austenite at the austenitizing temperature is time dependent and the homogenization of carbon compositions may not be possible in a single circle for the short holding times. The higher the carbon content of the steel samples the higher the carbon composition of the austenite and the higher the proportion of martensite formed upon quenching.

Plates 4.1 to 4.6 show the microstructure of the various steel samples in the as-received form and after thermal cycling. It was observed that the grain size was slightly smaller the higher the carbon content. Moreover, the size is larger the higher the austenitizing temperature due to increased grain growth tendency as the temperature is raised. The higher the carbon content, the greater this tendency. It was generally observed that the microstructures became finer with increasing number of cycles with the first few austenitization cycles producing microstructures with uneven grain size distribution and finer structures with a good degree of homogeneity after further cycling.

For the samples subjected to alternate thermal cycling treatment, unequal grain size distribution was observed. This was suspected to be as a result of the undissolved proeutectoid ferrite grains in the two-phase region, which remain after quenching. The

austenite grain in the same region transformed to finer ferrite grains. The final structure thus, consisted of fine newly transformed ferrite grains and coarse undissolved ferrite.

4.2.7 Hardness properties of the thermally cycled steel samples

Tables 4.4 and 4.5 present the evolution of Brinell Hardness number of the steel samples after various numbers of alternate thermal cycling and re-austenitization thermal cycling. The changes in hardness values of the steel samples with the number of cycle of alternate thermal cycling are shown in Figures 4.15 and 4.16. It is observed that the hardness increased in both cases with increasing number of cycles. Also the higher the carbon contents the more the increase in the hardness value of the steel samples. This was probably due to higher volume fraction of harder martensile the higher the carbon content of the samples.

The changes in hardness were also consistent with the changes in the grain size. As would be expected, fine-grained structures were harder than coarse-grained structures.

Pasco, (1978) has pointed out that since deformation under the indenter is plastic, it should be possible to relate the hardness to the plastic properties of the material. Therefore the increase in hardness with decreasing grain size could be explained from the point of view of plastic deformation. Grain boundaries constitute barriers to the motion of dislocation and this impediment leads to increasing strength and hardness. Refinement of grain amounts to multiplication of grain boundaries, so that finer grain size gives higher hardness. It was however, observed that the higher the carbon content the greater the hardness. The observation could be explained through the diffusionless transformation resulting from the quenching treatment

Conventionally, quenching from austenite range hardens steel. The transformation of austenite to martensite by a diffusionless shear-type transformation in

quenching is responsible for the higher hardness obtained and this property is attributed to the effectiveness of the interstitial carbon in hindering the dislocation motion (Callister, 1999).

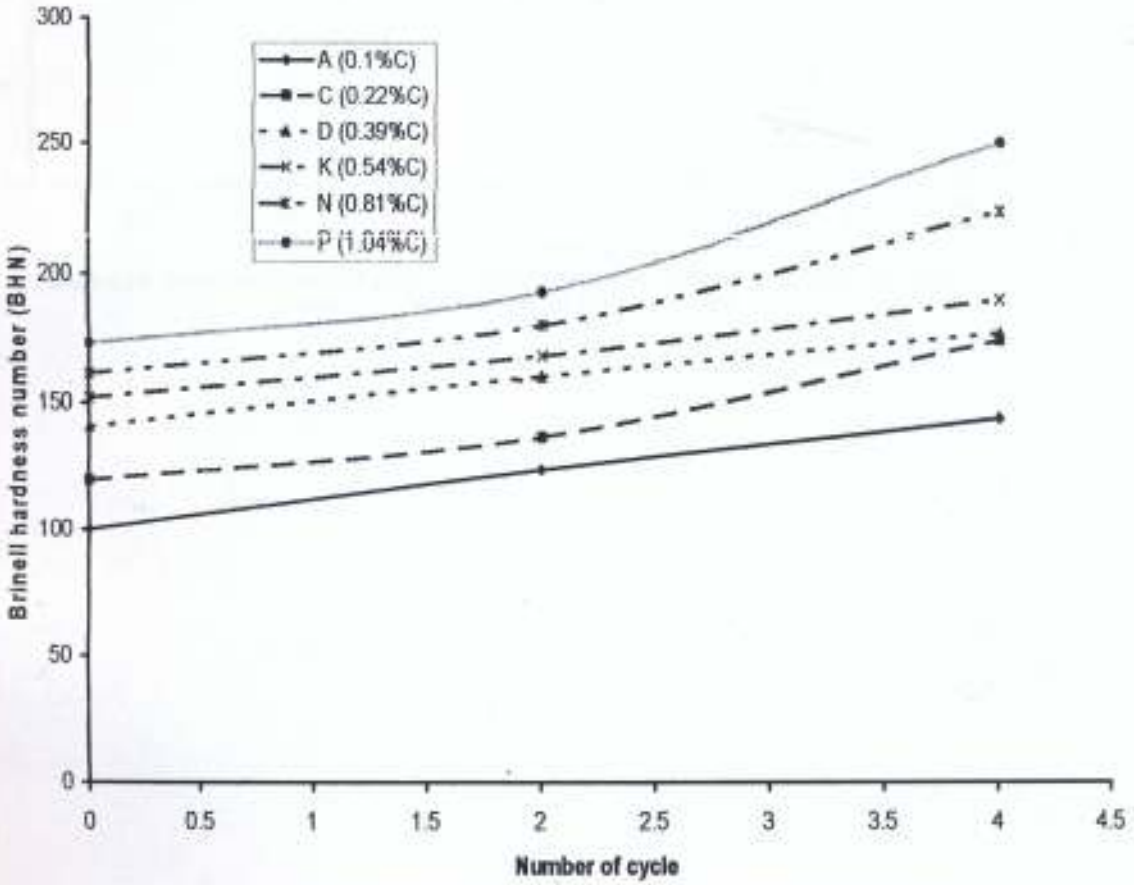


Figure 4.17: Brinell hardness number after various number of re-austenitization thermal cycling at constant holding time and peak temperature

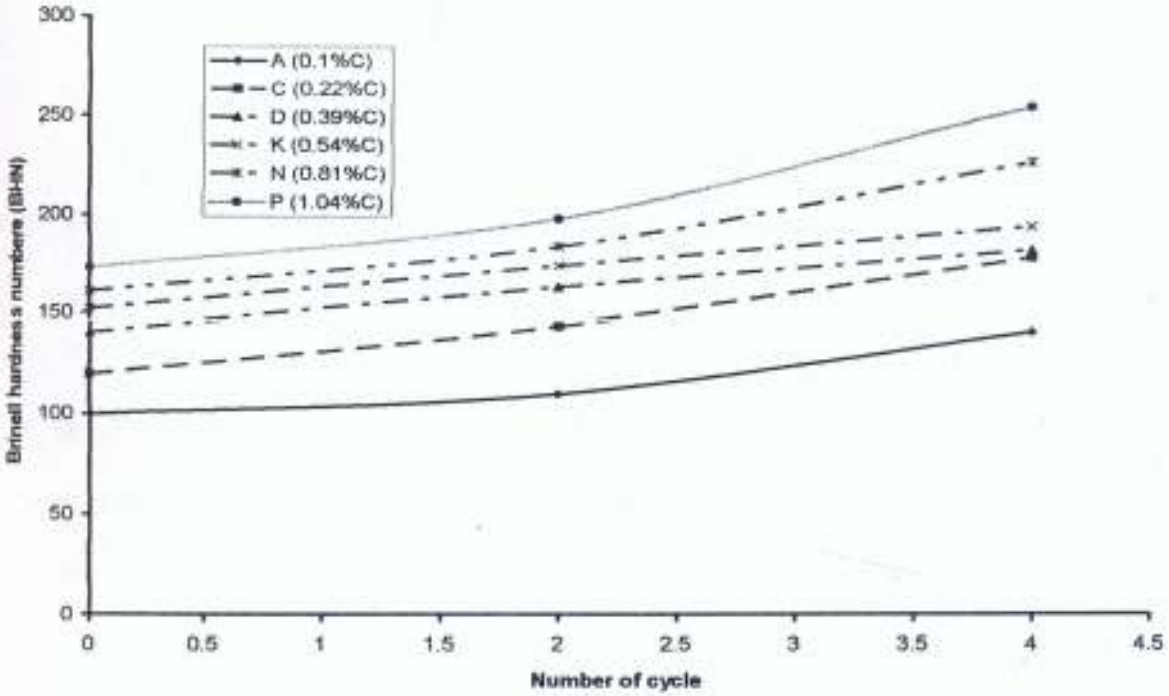


Figure 4.18: Brinell hardness number after various number of alternate thermal cycling at constant holding time and peak temperature

The solubility of carbon in iron is greatly reduced as a result of rapid cooling. The carbon remains in solution and strain the ferrite lattice giving a phase called martensite. One result is that a strong binding is set up between dislocations and carbon atoms and the motion of dislocation is restricted. Another result is the formation of carbon atom cluster on (100) planes.

The contribution to strength from the barriers in the martensite structure is essentially independent of carbon content, while the strengthening due to carbon atoms clustering and dislocation interaction increases approximately linearly with carbon content.

The increase in hardness with the number of cycles may be due to the possible incompleteness of the diffusion process in the austenite range as a result of the rapidity of the process. In heating, carbon diffuses from ferrite into the austenite and the attainment of equilibrium is time dependent. The higher the carbon content in the austenite solution, the higher the hardness obtained after quenching. Increase in carbon content of the austenite solution as the diffusion moves to completion in multiple cycling is probably responsible for increasing hardness as the number of cycle is increased.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The following conclusions can be made from the results presented so far:

(1) Thermal cycling treatments were employed in carbon steels, and an appreciable grain refinement and improved mechanical properties were obtained

(2) The two methods of thermal cycling i.e. re-austenitization thermal cycling and alternate thermal cycling under optimum condition of temperature and time are both effective means of grain refinement in carbon steels.

(3) Cyclic re-austenitization produced better grain refining effect and hence higher strength than alternate thermal cycling but the latter gave better combination of strength and ductility.

(4) Cycling beyond four cycles did not provide noticeable additional grain refinement but rather leads to improved uniform grain size distribution.

(5) Grain size reduction was more pronounced the higher the carbon content for samples A (0.1%C), C (0.22%C), and D (0.39%C), while for samples K (0.54%C), N (0.81%C), and P (1, 04%C) the reduction is less pronounced the higher the carbon content.

5.2 Recommendation

To better comprehend and fully exploit the strengthening potentials of carbon steel by thermal cycling, a more detailed study of the following areas is recommended:

(1) Determination of the separate contributions of other chemical and micro-structural constituents to the properties developed by thermal cycling treatments.

(2) Determination of the optimum levels of other chemical contents for grain refinement by the two thermal cycling methods- alternate thermal cycling repeated austenitization thermal cycling.

(3) A more detailed study of the effect of various thermal cycling treatments on the properties of carbon steels.

A detailed study of the aforementioned areas was not possible due to inadequate facilities and time constraints. It is therefore recommended that future work should be done to include these areas, which are certain to provide necessary knowledge for determining the usefulness of carbon steels as engineering materials.

CHAPTER SIX

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