

**EVALUATION OF THE SUITABILITY OF WARM WATER FOR
INTERRUPTED QUENCHING OF DUCTILE CAST IRONS**

BY

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CERTIFICATION

This work has not been presented elsewhere for the award of a degree or any other purpose

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DEDICATION

This work is dedicated to:

1. The only dependable, reliable and faithful God.
2. My late father, Mr. Aliyu Barnabas. and
3. My beloved Mother, Mrs Theresa Barnabas.



ABSTRACT

The Evaluation of the suitability of warm water for interrupted quenching of ductile cast irons was carried out. The interrupted quenching process was carried out by austenitizing at 850°C for 30minutes, quenching in warm water at 80°C temperature for 40seconds and then quickly transferred to another furnace maintained at different predetermined temperatures and holding periods. The selected furnace temperatures were between 175°C, and 450°C, while the holding times varied from 30 to 180 minutes. Final cooling was carried out in air. After heat treatment, the Rockwell hardness (Rc) value of each specimen was measured and microstructural examination carried out. Statistical Package for Social Science (SPSS) was used to develop a computer model and computer software from the hardness values obtained. Results showed that the maximum and minimum hardness values of 53HRc and 19HRc were obtained respectively. Metallographic analysis showed that untempered martensite was obtained at holding temperature below 250 °C, tempered martensite at 250 °C to 325 °C, over-tempered martensite at holding temperature of 375 °C for short holding times, over-tempering occurred beyond the holding temperature of 400 °C for all holding times. For the model developed, the correlation coefficient was found to be between 0.9925 and 0.9595 while the standard error was between 2.162 and 2.823. This new heat treatment process route for ductile iron has provided a cheap, easy and efficient means of producing tempered ductile iron without using the conventional method which is relatively expensive to procure, not readily available and energy consuming.



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NOMENCLATURE

X_1 = Time of tempering

X_2 = Temperature of Tempering

X_{12} = Percentage Iron

d = Pair difference between the Experimental and Numerical Data on each Column.

$\bar{d}$ = Mean Pair difference

$\text{Var}(d)$ = Variance of Pair difference

$\text{Var}(\bar{d})$ = Variance of mean difference

t_v = Theoretical Number of Degrees of Freedom available for the Estimation of the Variance
i.e. $(n-1)$

N = Number of Paired test Samples

α = Confidence Interval (%)

Sd = Standard Deviation

$\text{Cov}(x,y)$ = Covariance of X and Y

σ_x = Variance of X

σ_y = Variance of Y

P_{xy} = Correlation Coefficient

$\bar{x}$ = Mean of X

$\bar{y}$ = Mean of Y

X = Experimental Value

Y = Numerical Value

N = No. of pair Test Samples

Y_C = Predicted Value

Y_a = Experimental Value

$\bar{Y}$ = Average of Y_a

Y = Actual Value (Experimental Value)

Y_r = Predicted value (Numerical Value)

HRC = Rockwell Hardness value (Rockwell – C Scale)

CHAPTER ONE

1.0 INTRODUCTION



Ductile Irons, also known as spheriodised or Nodular Cast Irons, has the structure of graphite nodules in a matrix which may be ferritic, pearlitic or ferritic-pearlitic (ductile grades), bainitic (strong and tough grades) or martensitic (strong and hard grades). The variations in the matrix structure have caused the mechanical properties of ductile cast irons to vary widely and have made them applicable in a wide range of industrial applications, especially in the areas requiring high strength, high toughness and high ductility. It's use will continue to expand as the design engineers continue to improve these properties. Ductile iron and steel are similar metallurgically, the main difference is the high carbon and silicon levels in ductile iron which lead to the formation of graphite eutectic structure. The higher the silicon content, the lower the solubility of carbon in austenite and more readily carbon is precipitated as graphite during slow cooling to produce a ferrite matrix. Ductile iron castings can be heat treated to increase strength, toughness, ductility, wear resistance, corrosion resistance, improve consistency of properties, machinability and relief internal stresses.

Apart from the matrix structure, other microstructural factors that contributes to loss of toughness in ductile irons include nodular count and nodularity of graphite, percentage or continuity of intercellular or interdendritic carbides, and micro porosity.

The best overall mechanical properties of high strength, toughness and hardness are obtained when austenite is caused to transform to bainite structures. Hence, according to (Hemanth, 2001), the major users of ductile iron components include the automobile and machine tool industry.

This study focuses on the use of ductile iron in order to obtain good heat treating properties of martempering structures not involving the traditional martempering process and also to develop a reliable computer model for theoretical determination of these properties. Not much information exists with respect to investigating and modeling of the mechanical properties of ductile irons produced through interrupted cooling processes (Martempering) especially the use of warm water as a quenchant.

1.1 BACKGROUND OF THE RESEARCH

An understanding of the properties of materials is essential in both the design and applications in any engineering project if it is to prove satisfactory for its intended purpose (Neil, and Ravindra, 1996). The ability of a material to adequately perform in service demonstrates several mechanical properties of the material. For instance, the network of bridges and structures, high rise buildings and road ways are illustrations of the capabilities of materials (Vernon, 1977).

Unlike grey and white cast irons, which are brittle, ductile irons can be processed to have high strength, high hardness, great ductility and good toughness. A comparable cast iron is malleable cast iron which has been successfully replaced by ductile iron because of superior mechanical properties part from the ease of production and good economy. Another attraction for ductile iron is its application where medium carbon steels had been earlier favoured.

Conventional heat treatment of ductile iron causes transformation of austenite into structures such as high carbon ferritic, pearlitic, tempered or untempered martensitic structures. Heat treatment by interrupted cooling of steels and ductile iron provide special properties because of the unique structures which are produced. In

addition, the introduction of thermally induced strain and its effects during direct quenching is drastically reduced. Also, heat treatment by interrupted cooling cause's formation of refined structures of fine pearlites, bainites and tempered martensite depending on the transformation temperature.

Norminal chemical composition for the productionof ADI is 3.6%C, 2.5%Si, 0.3%Mn, and 0.05% Mg. Other suitable additions such as Cu, Mn, Mo, and Ni have been identified by several authors including Branka et al (2004) and Hassan et al(2000). These authors reported that the primary purpose of adding Cu,Ni,or Mo to ADI is to increase the hardenability of the matrix sufficiently to ensure that the formation of pearlite is prevented during the austempering process. Certain alloying elements, however, will lead to increase in intercellular segregation and carbide formation hence slowing down the austempering kinetics, increasing the overall processing time required to obtain the optimum properties. However, the austempered ductile irons should be sufficiently alloyed to prevent transformation to pearlite, but not over alloyed to avoid the retardation of the bainite transformation.

Unit cell model (UCM) calculations are widely used to predict macro-scale constitutive behavior of composites and the pioneer model in this area was credited to Gurson (1977),which was later improved upon by Tvergaard and Needleman (1984) and it is usually referenced as GTN model. A number of authors, Dong et al, (1997); Berdin et al, (2001), proposed modeling the mechanical behavior of cast irons using GTN model. The model accounts for the nucleation, growth and coalescence of microvoids by means of the void volume fraction, which is the average measure of a void matrix aggregate. Recent contributions in the field are due to Pirondi et al, (2006), Bonora et al, (2006), Horstemeyer (2007), Zairi et al,(2008) proposed two

different micro mechanical models based on porosity evolution. However, not much work has been carried out on modeling of mechanical properties of ductile irons heat-treated by interrupted cooling over a wide range of temperatures of 175⁰C-450⁰C. Finally, there is also need to replace the conventional salt (nitrate,nitrite and chloride) which is expensive and relatively complex with more health friendly quenchant. It is known that Hassan et al,(2008) studied the possibility of using hot vegetable oils (shear butter and groundnut oils at temperature of 250⁰C) in place of traditional salts, Suitability of Khaha Senegalensis seed oil as a quenchant in austempering heat treatment of ductile iron was investigated (Isah et al, 2009). The investigation of water will add to the number of other materials that can be utilized for interrupted quenching processes minimizing energy consumption that is associated with salt bath because of the elevated temperature of the salt as well as the increased capital equipment cost for the bath and the equipment that operates it.

1.2 JUSTIFICATION FOR THE RESEARCH

The ductile iron has consistently replaced some steel, that is, many cast and forged steel components. This is mainly because of its high toughness in the quenched and tempered condition and the cost of production. There is need to increase this spectrum by producing critical components at cheaper costs and under safer environmental conditions. It is hoped that this will be achieved by some interrupted cooling heat-treatment cycles. In this respect, there is need to replace salt (nitrate, nitrite and chloride) with other quenchant which performs similar work as salt bath but more health friendly and economical.

Modern techniques in component design involve the use of computer, especially the use of computer aided design methods. The same trend is applicable in

metallurgical processes whereby computer models are developed for theoretical determination of mechanical properties of materials. There is therefore also need for modeling the estimation of the mechanical property in order to improve the reliability of the test results and overall efficiency of the production system. There is need to device a simple, flexible and efficient means of estimating this property at a relatively low cost. Hence, this is the rational behind modelling of the mechanical properties and development of computer software to estimate the modelled mechanical properties of ductile iron heat treated by interrupted quenching using water as quenchant.

1.3 AIM AND OBJECTIVES OF THE RESEARCH

The aim of this research is to evaluate the suitability of warm water for interrupted quenching of ductile cast irons and develop a computer model for predicting the hardness values of the ductile irons produced using the process.

The specific objectives of the research are to:

- (i) investigate the use of warm water for interrupted quenching heat treatment process for ductile irons.
- (ii) determine the hardness values of ductile irons subjected to above heat-treatment.
- (iii) develop and validate a computer model, using the data obtained in (ii).

1.4 CONTRIBUTION TO KNOWLEDGE

The study, if successful, is expected to:

- (a) show the suitability and the reliability of warm water as a quenching medium for the interrupted cooling of ductile iron for the production of martempered structures,
- (b) provide an easy, cheap and alternative process route to the current salts, and
- (c) successfully lead to the development of a computer model for predicting the mechanical properties of the ductile irons so produced.

1.5 SCOPE OF THE RESEARCH

The research work will cover the heat treatment of a large number of as-cast ductile iron samples, each of 20mm X 20mm X 15mm size, which were sourced from the University of Wisconsin, Madison, USA. All of the samples, except two, were subjected to a preliminary normalizing heat treatment by heating to 850°C.

The main work was centred on heating the normalized sample to and austenitized at 850°C, quenched in warm water at 80°C for 40, and then reheated to a subcritical temperature of between 175°C and 450°C for specific holding times. After this, final cooling was carried out in air.

Hardness measurements were carried out on the heat treated samples. These hardness values were used to develop and validate a computer model. Finally, metallographic works were carried out to relate the hardness values obtained with the observed microstructures.



CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Cast Iron

According to Yescas-Gonzalez and Bhadeshia (1998), cast irons typically contain 2 to 4 wt% of carbon with a high silicon concentrations and a greater concentration of impurities than steels. The *carbon equivalent* (CE) of a cast iron helps to distinguish the grey and ductile cast irons which cool into a microstructure containing graphite and the white irons where the carbon is present mainly as cementite. The carbon equivalent is defined as:

$$CE(wt\%) = C + \frac{Si + P}{3} \dots\dots\dots(2.1)$$

Where C, Si, and P are the percentages of carbon, silicon and phosphorous respectively present in the cast iron.

A high cooling rate and a low carbon equivalent favour the formation of white cast iron whereas a low cooling rate, a high carbon equivalent and presence of graphitizing agents promote the formation of graphite phase. During solidification, a major proportion of the carbon precipitates in the form of graphite or cementite (Colin and Brandos, 1976). When solidification is just complete, the precipitated phase is embedded in a matrix of austenite which has an equilibrium carbon concentration of about 2 wt%. On further cooling, the carbon concentration of the austenite decreases as more cementite or graphite precipitates from solid solution. The last austenite in the structure decomposes into pearlite at the eutectoid temperature.

2.1.1 White Iron

White Irons are fully carbidic in its final form. The presence of different carbides, produced by alloying, makes white iron extremely hard and abrasion resistant but very brittle.

2.1.2 Grey Iron

Grey iron is by far the oldest and most common form of cast iron, which has been so named because its fracture surface has a grey appearance. Metallurgically, it consists of carbon in the form of flake graphite, which has been caused to occur by the addition of graphitizing agents, in a matrix which may be ferritic, pearlitic or a mixture of both in the as-cast condition.

Grey cast irons contain graphite flakes in the microstructure. The graphite flakes, which are rosettes in three dimensions, have a lower density than liquid iron and hence result in expansion during solidification and compensate for the freezing contraction. This expansion during solidification and inherent fluidity of liquid grey iron, have made this metal ideal for the production of shrinkage-free, intricate castings such as motor blocks.

The graphite flakes have been responsible for the good damping characteristics and good machinability, because they act as chip-breakers and lubricate the cutting tools. However, graphite flakes are stress concentrators, leading to poor toughness, low ultimate tensile strength and brittleness. The recommended maximum applied tensile stress is therefore limited to a quarter of its actual ultimate tensile strength.

2.1.3 Nodular Cast Iron

The graphite can be induced to precipitate in a spheroidal shape by removing sulphur from the melt by means of a small addition of calcium carbide. *In addition, a minute addition of magnesium or cerium, which* poisons the preferred growth directions have been found to lead to isotropic growth resulting in spheroids of graphite. The calcium treatment is necessary before the addition of magnesium because the latter also has an affinity for both sulphur and oxygen, whereas its spheroidising ability depends on its presence in solution in the liquid iron.

For practical conveniences, magnesium is frequently added as an alloy with iron and silicon (Fe-Si-Mg) rather than as pure magnesium. However, magnesium tends to encourage the precipitation of cementite, so silicon is also added (in the form of ferro-silicon) to ensure the precipitation of carbon as graphite. The ferro-silicon is known as an inoculant.

Nodular cast irons, in the as-cast condition, may contain matrix structures, which may be ferritic, pearlitic or both. The mechanical properties of these irons vary widely in respect of strength and toughness. However, they all have good ductility and therefore commonly referred to as ductile cast irons or simply ductile irons. The presence of graphite nodule has been principally responsible for these properties.

The improved mechanical properties of ductile irons are derived from an improved manufacturing process that changes the character of the graphite content of the iron. Ductile Iron's graphite form is spheroidal, or

nodular, instead of the flake form in grey cast Iron. Due to the spheroidal shape of graphite, ductile irons have approximately twice the strength of grey cast iron as determined by tensile, beam, ring bending, and bursting tests. Its impact strength and elongation are many times greater than grey cast iron's.

As-cast ductile irons can be heat treated to produce fully ferritic, pearlitic, martensitic or bainitic matrix structures for the desired mechanical properties has excellent toughness and is used widely, for example in crankshafts.

The latest breakthrough in cast irons is where the matrix structure has been modified to be fully bainitic. This results in a major improvement in toughness and strength. The bainite structures are obtained by isothermal transformation of the austenite at temperatures below that at which pearlite forms. Ductile irons, do not only possess all of grey cast iron's attractive qualities, such as machinability and corrosion resistance, but also have higher strength, toughness, and ductility. They are lighter in weight, stronger, more durable and more cost effective than grey cast Iron (Keith *et al*, 1949).

Although they have chemical compositions similar to those of grey cast Iron, ductile iron incorporates significant casting refinements, additional metallurgical processes, and superior quality control.

2.2 Nodularization of Graphite

Graphite shapes in cast iron are influenced basically by alteration of the bulk melt chemistry which leads to formation of perfect or imperfect nodules (Yamamoto, 1978). Evidences are now available to buttress the role of inoculants: magnesium, cerium, rare-earth elements, lithium, calcium etc. as nodularizers. They include the experimental observations of the enrichment of the elements in the graphite nodules. Lyubenko, *et al* (1961) recorded cerium concentration in the nodules of cerium-magnesium treated cast irons. Ililiert-Liiulblom (1954) and Bolotov *et al* (1957) have also found calcium and cerium to be uniformly distributed in the graphite nodules (Yamamoto, 1978).

In 1982, Fidos used the versatile high resolving power Secondary Ion Mass Spectroscopy (SIMS) to observe the presence of higher concentration of magnesium and calcium in nodules than in the surrounding matrix. His approximate atomic concentrations of both elements were 0.2 and 0.17 weight %, respectively (Franklin *et al*, 1984).

Although there are some theories that explain the nodularization process, but there is not yet a universal one to explain the controlling mechanism in cast iron. However, there is a generally accepted concept that the surface-active elements; sulphur and oxygen hinder the process of nodularization by adsorbing on certain crystallographic planes (Franklin and Stark, 1984). At the onset of graphite formation in gray irons, nuclei have also been found nodular in shape (Kubota *et al*, 1986), even though they later grow into (lakes as a result of the activity of the surface-active elements.

The principal differences that exist between the growth mechanisms of the flake and nodular graphite have been found by Franklin and Stark (1984). According to these authors, flake graphite growth occurs predominantly on the prism plane of the hexagonal graphite lattice and takes place as a coupled eutectic austenite in contact with the melt. Nodular graphite growth occurs on the basal plane by a defect controlled spiral growth mechanism as a divorced eutectic, the graphite nodules being surrounded by liquid iron during the initial part of the growth processes.

Subramanian (1982), Johnson and Smart (1977), Francis (1979) and Sodocha and Gruzeleski (1974) and Kubota *et al*, (1986) have all in their respective research reported the dependence of the role of nodularizing agents on sulphur and oxygen concentrations in iron melt. The nodularizing agents display higher affinity for the two elements and hence, combine with them instead of adsorbing on the prism planes. In this way the basal planes of the graphite lattice acquire a more favorably lower surface energy in contact with liquid iron, a condition that enhances the growth mechanism of nodular graphite. Minkoff and Nixon (1966) based on their SEM study proposed a stage growth mechanism, which has been attributed to the sequential adsorption of nodularizing atoms (Kubota *et al*, 1986).

Yamamoto, *et al* (1978), believes that the severally proposed models of the formation of graphite nodules lack adequate observation. Instead, they proposed the gas bubble theory to explain the same mechanism in the magnesium treated cast iron. The gas bubble theory relates the nodule formation process to the precipitation of carbon into the magnesium bubbles from the supersaturated molten iron. The bubbles

are formed only when external pressure is lower than the equilibrium vapour pressure of Magnesium (Mg).

The work of Ikenzo *et al* (1981) which has led to the formation of nodular graphite in pore containing white cast iron sintered by means of direct electric resistance heating tends to support the gas-bubble theory. But the theory does not seem to be operative in all respects since it does not explain nodulurization in the cerium or other high boiling point nodularizing agents.

Magnesium is the only element of all the nodularizers that has its boiling point (1090°C) below the pouring temperature of about 1400°C. Calcium for instance has boiling points in excess of the pouring temperature. It is therefore not likely to promote nodule formation by the bubble theory mechanism. The work of Dhindaw and Verhoeven does not support the gas theory. In the work, they were able to produce different nodule counts in two melts having identical gas content. Also, cooling high purity melts under vacuum and argon did not show any significant difference in nodule count even when the rest of the conditions were maintained constant. This should not be the case, since gas bubbles stability in the two cases would be quite different.

In order to clarify the mechanism of nodular graphite formation, the interfacial free energy ratio is now regarded as a deciding parameter (Mitsuharu and Yoshisada, 1979). It involves measuring the interfacial energies between molten irons which solidified with different structures of graphite, and specific planes of graphite crystals and thereafter calculating the interfacial free energy ratio. The ratio indicates the relative stability of two geometrical arrangements, such

as flaky single crystal and spheroidal polycrystal. A small value of the ratio corresponds to low free energy and stability of the spheroidal graphite polycrystal.

2.3 Heat treatment of Cast Irons

Heat treatment is an operation that involves heating and cooling of materials at controlled rates in their solid state with the desire to impart properties of interest. Like steels, the matrix of cast irons is amenable to control by a variety of heat treatments. The principles of heat treatment in ferrous metals are similar because of the polymorphic changes exhibited by the base metal, iron, at different temperatures. Common heat treatment processes for cast irons include:

1. Stress-relieving or aging treatments, which aims at removing residual stresses in the iron. It is accomplished by heating cast iron to between 400 and 550°C, holding at the temperature for a period of over 30 minutes. The holding time may be as long as 5 hours depending on the size of the component.
2. Annealing helps to impart improved ductility and facilitate machinability in castings. The temperature range commonly employed in the annealing of cast iron is between 750°C to 850°C even though annealing at temperatures as low as 650°C has been satisfactorily used. Highly alloyed irons are sometimes annealed at temperatures as high as 950°C. There are different types of annealing processes applicable to cast irons, which include:
 - (i) Ferritizing heat treatment, which is carried out principally to convert the pearlite structure in the matrix to ferrite. Complete ferritic structure can be achieved by heating to temperatures as low as 700°C

for 1 to 5 hours depending on the composition and size of casting as well as the amount of pearlite present in the matrix. Final cooling must be very slow, in the furnace, to avoid austenite-pearlite transformation.

- (ii) Malleabilizing Heat Treatment, carried out when massive carbides are present in the matrix, they are converted into austenite and graphite by heating to between 820 and 930°C for 1 to 5 hours. This is followed by slow cooling to about 700°C, held for about 3 to 5 hours, depending on the malleabilizing temperature, alloy content and component size to convert the matrix to ferrite (Metal Handbook, 1980)
 - (iii) Malleabilizing treatment for white cast irons are carried at the same temperature range, but takes 2 to 3 days of holding to fully convert the combined carbon to tempered carbon. Subsequent slow cooling through the critical range (10°C/hr) results in a microstructure which consists of ferrite plus temper carbon or products containing various amounts of pearlite and ferrite together with temper carbon.
3. Normalizing Heat Treatment process carried by heating to between 800°C to 850°C for an excess of 30 minutes, the holding period depends on the time needed for full compositional homogenization and full austenization. Higher temperatures of up 900°C or above may be adopted for alloy grades, but this followed by slow cooling to about 800°C to avoid cracks in thin sections. Final cooling is in still or forced air.

4. **Hardening Heat Treatment** process, which is carried out to produce hard martensite structures in cast irons. Heating is in the same way as in the Normalizing heat treatment, but the component is rapidly cooled by quenching in a cold liquid medium
5. **Tempering Heat Treatment**, involving heating of the normalized or quenched structures to a subcritical temperature for a sufficient time to cause removal of internal stresses, transformation of retained austenite and formation of tempered martensite before the final slow cooling down.
6. **Austempering Heat Treatment**, a process whereby austenite is caused to transform to bainite structures. Conventionally, cast irons components are:
 - (i) heated to the austenite temperature of between 800°C and 850°C to effect necessary homogenization of composition and structure.
 - (ii) rapidly quenched in salt bath to a bainite transformation temperature range of between 250°C and 400°C , held at this temperature for a period of time necessary for austenite to fully transform to bainite structure.
 - (iii) final cooling down to room temperature in air.

2.4 Bainite Formation

Bainite, as the product of austenite decomposition was first reported in 1930 by davenport and Bain, and has been so designated in honor of E.G. Bain. (Singh, 2005). TTT diagram of eutectoid steel shows that Bainite, i.e., lath-shaped fine aggregates of ferrite and carbide form isothermally between the nose of the curve and the M_s temperature, i.e., usually in temperature range between 250°C to 550°C . The morphology of bainite is distinctly different from that of fine lamellar pearlite. Bainitic transformation has some of the features which are similar

to pearlitic reaction and some of the characteristics of martensite. Bainite is presumed to have its own 'C' type kinetics, shown clearly by many alloy steels, but pearlite and bainite transformation overlap considerably in plain carbon and many low alloy steels (Singh, 2005). Bainite may be obtained isothermally, or during athermal treatments at cooling rates too fast to form pearlite, yet not rapid enough to produce martensite. In carbon steels, however, the overhanging pearlite nose eclipses the bainite transformation. The result is that austenite on continuous cooling either transforms fully to pearlite, or to martensite or a mixture of both. But bainite forms easily in some alloy steels containing elements like Chromium and Molybdenum. These elements which retard transformation to pearlite help in the formation of bainite on continuous cooling. Ability to form bainite increases with greater hardenability of the steel and cast iron (Singh, 2005).

Transformations in general are classified as either reconstructive or displacive. Reconstructive transformations are diffusional and require the distribution of atoms or individual atomic motion as in austenite -to- pearlite transformation. Displacive transformation, on the other hand, is non-diffusional and is often referred to as athermal or martensitic. It occurs only when austenite is rapidly cooled to temperature at which diffusion processes are impossible. And this is accomplished by the shift type mechanism, which consists of ordered co-operative directional displacement of atoms during the process of lattice rearrangement (Umore, 1990).

Bainite transformation is an intermediary between the principal types mentioned above. It combines both elements of the pearlite and martensite transformations. At its transformation temperature, the rate of self diffusion of iron is

low, the diffusion of the alloying elements is practically impossible while the rate of carbon diffusion is sufficiently high.

Bainitic transformation has its own 'C' curve for most alloy steels and cast irons, with well defined B_s temperature. But there is quite an overlapping between bottom of pearlite reaction curve and top of the bainitic reaction curve, in carbon and low alloy steels, which do not have a well defined B_s temperature. B_s temperature for low alloy steels is given by:

$$B_s \text{ (}^\circ\text{C)} = 830 - 270(\%C) - 37(\%Ni) - 70(\%Cr) - 83(\%Mo) - 90(\%Mn) \dots\dots\dots (2.2).$$

In alloy steels, the amount of Bainite formed increases with time when the steel is held between B_s and B_f temperatures, but transformation does not go to completion (Singh, 2005). As the morphology of lower bainite is very much similar to martensite, it is often difficult to fix the B_f temperature. Moreover, bainite reaction may extend into martensitic region, i.e., B_f temperature may be above, or below M_s temperature. To an approximation, B_f temperature may be obtained by equation;

$$B_f \text{ (}^\circ\text{C)} = B_s - 120^\circ\text{C} \dots\dots\dots (2.3)$$

Growth of bainitic-ferrite plate requires the local diffusion of carbon, which also helps to nucleate finely dispersed carbides. This constitutes one of the differences with the growth of martensite plates, where no diffusion, even of carbon, occurs. But the diffusion of substitutional alloying elements does not occur in bainitic reaction, which constitutes one of the differences with pearlite reaction, where these solute atoms partition between ferrite and cementite when pearlite forms. As said earlier, continuous cooling of carbon steels and cast irons do not yield bainite because of

shielding by ferrite and pearlitic reactions, but substitutional alloying elements retard ferritic and pearlitic reactions to produce bainite by continuous cooling. Low carbon steels with 0.5% Mo and 0.002% B have complete bainitic structure. Bainitic reaction shows two important characteristics (Singh, 2005);

2.6 Martempering (Marquenching)

Martempering, otherwise known as Marquenching, permits the transformation of austenite to tempered martensite to take place at some temperatures within the M_s - M_f temperature range. By using interrupted quenching, the cooling is stopped at a point above the martensite transformation region to allow sufficient time for the center to cool to the same temperature as the surface. Then cooling is continued through the martensite transformation region. The advantage of martempering lies in the reduced thermal gradient between surface and center as the part is quenched to the isothermal temperature and then is air cooled to room temperature. Residual stresses developed during martempering are lower than those developed during conventional quenching because the greatest thermal changes occur throughout the part at approximately the same time. Martempering also reduces or eliminates susceptibility to cracking. It is used primarily to minimize distortion, eliminate cracking, and minimize residual stresses, it also greatly reduces the problems of pollution and fire hazard as long as nitrate-nitrite salts are used rather than martempering oils.



2.7 Austempering of nodular cast iron

Most of the heat treatments, as we have seen above, impart just one type of property or the other on the irons. They are strengthened and hardened by the normalizing and hardening processes while they become softened by any of the annealing or tempering processes. The austempering process is one heat treatment which is capable of imparting high strength, hardness and toughness in steels and their alloys. This exceptional combination has been attributed to the relief of residual stresses, distortion and cracklings associated with rapid cooling in quenched and as cast structures.

Austempering is a four-stage heat treatment process involving:

- (i) heating to the austenite temperature range above the critical temperature.
- (ii) soaking for a sufficient time depending on the size and composition,
- (iii) quenching to the austempering temperature holding at the temperature for isothermal transformation of austenite to bainite.

Austempering temperature is deduced from the Time Temperature Transformation (TTT) diagram, shown in Figure 2.1. In unalloyed cast irons the austempering temperature is the bay of the TTT-diagram between the nose and the M_s temperature.



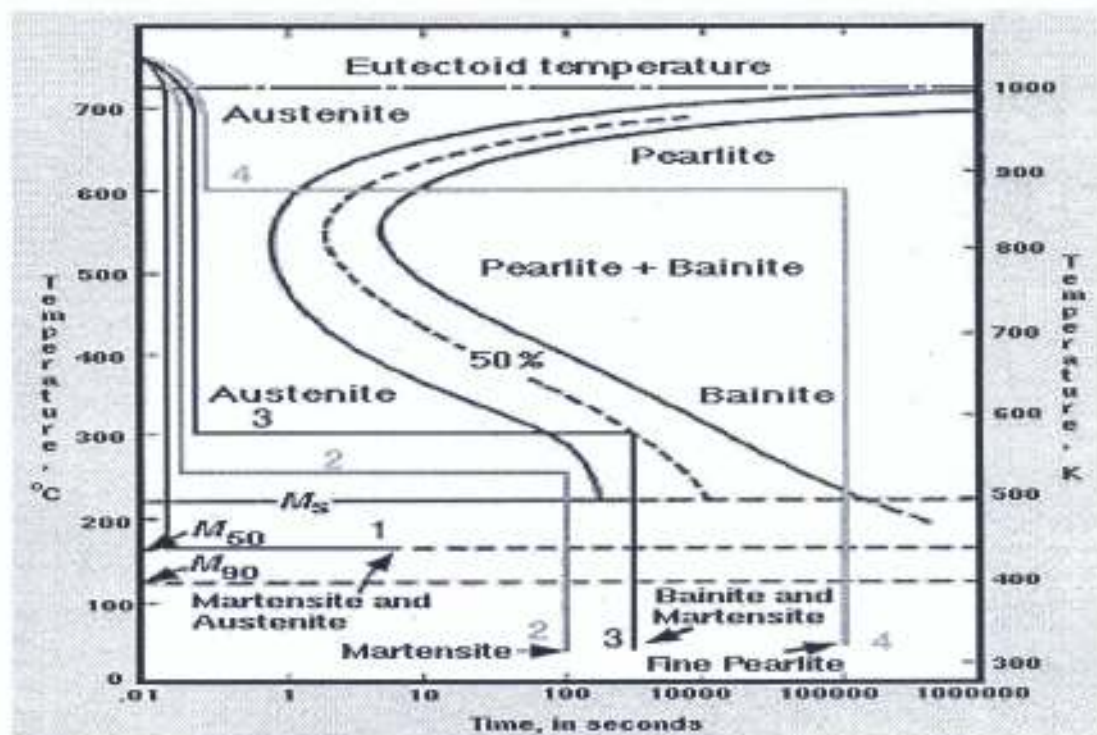


Figure 2.1: Temperature-Time-Transformation Diagram for Ductile Irons

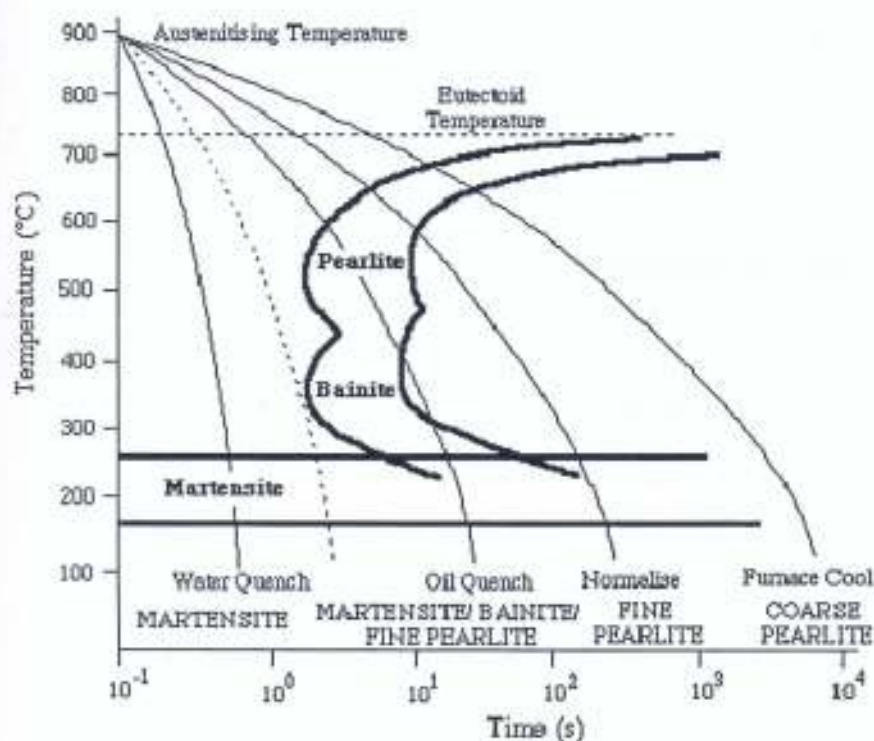


Figure 2.2 Continuous Cooling Transformation Diagram for an alloyed ductile iron

For alloyed cast irons whose pearlite and bainite regions do not overlap, in Figure 2.2, the austempering temperature is selected in the regions of the bainite start transformation curves. As a means of reducing the cost of expensive facility due to the large capital outlay for the furnaces required to produce austempered ductile iron, Janowak et al, (1981) suggested an alternative. That casting be cooled in the mould to a suitable austenitizing temperature (about 860°C), followed by hot shake out and air cooling to the desired austempering temperature. Holding at the temperature is carried out in an insulating to allow the bainitic reaction to occur before the final removal and cooling in air to the room temperature.

2.8 Commercial Salts for Austempering Operations

Table 2.1 presents a number of the salts commonly used with their respective properties. The salt mixture density, ρ_t according to Smithells and Brandes (1976), is calculated from the relationship:

$$\rho_t = A - 10^{-3} B t \dots \dots \dots (2.3)$$

where: A and B are the constants given in table 2.4, and t is the temperature of operation. Other properties considered in the choice of salt mixture at a particular temperature include:

- (i) density, which should increase at a constant rate within the temperature range of treatment,
- (ii) thermal and electrical conductivities which should be sufficiently high to enhance the transfer of heat to and from the specimen, and viscosity which is often kept low as much as possible.

Table 2.1 Typical Composition of Salt Mixture with their working Temperature Ranges and Applications (Umoru, 1990)

Temperature Range	%Composition	User
50°C - 650°C	40/50 NaNO ₂ 50/60 KNO ₃	Tempering of steels, Age hardening of Al alloys
500°C - 750°C	15/25 NaCl, 25/35 BaCl ₂	Tempering of high-speed tool Steel
750°C - 900°	15/25 NaCl, 20/30KCl, 50/60 BaCl ₂	Neutral hardening of steel.
850°C - 950°C	40/50 NaCN, 20/30 BaCO ₃	Cyanide hardening surface
1000°C- 135°C	98 BaCl ₂ 2 NaCl	Hardening high-speed tool steel.

2.9 Application of Austempered Ductile Irons as Steel Substitutes

Since its introduction into the market in 1955, both ductile iron and austempered iron pipes have been recognized as the industry standard for modern water and wastewater systems. More than four decades of field experience have proven their strength, durability, and reliability for transporting raw and potable water, sewage, slurries, and process chemicals.

DI and ADI iron pipes are designed and manufactured to meet industry's most stringent standards, resists damage during shipping and handling, and, once installed, withstands the most demanding operating conditions, including water hammer, frozen ground, deep trenches, areas of high water table and heavy traffic, river crossings, pipe on supports, rocky trenches, and areas of shifting, expansive,

and unstable soils. Additionally, numerous laboratory and field tests have proven that austempered ductile irons possess superior corrosion resistance greater than that of grey cast iron and cast ductile irons, which have service history as pipe materials and, once installed, the pipes are virtually maintenance-free.

This treatment, according to Janowak et al,(1981), results in a high strength, high toughness considerable hardness and good elongation at fracture.

2.10 Mechanism of Bainite Formation

Austempering involves both chemical and transport processes. At isothermal transformation temperature, the chemical process proceeds at a higher rate than the transport process. Hence the transport process determines the transformation rate. The transport rate, which is temperature dependent, is provided by Aaronson and Lee (1975) in equation 2.4.

$$J = ZBN \exp \frac{(-\delta G)}{Kt} \exp \frac{-r}{\tau} \dots\dots\dots (2.4)$$

Where

- J = is the flux through the n-space
- B = is the frequency factor
- N = the number of atomic nucleation site per unit volume
- τ = the incubation period.
- t = the isothermal transformation reaction time
- k = Boltzman constant,
- Z = the Zeldovich non-equilibrium factor typically given as 1/20, and
- δG = the free energy difference.

Generally, transformation is by nucleation and growth mechanisms. The driving force for the reactions is the free energy difference, δG between the parent and the new-phase. δG increases with degree of undercooling according to the expression:

$$\delta G = \frac{(T_0 - T)\delta H}{T_0} \dots \dots \dots (2.5)$$

where:

T_0 = the transition temperature

δH = the heat of transformation, which is determined by the isothermal transformation temperature (T)

and $(T_0 - T)$ = the degree of undercooling.

The rates of isothermal nucleation(N) and growth(G) are also dependent on the transformation temperature in addition to other metallurgical factors such as; degree of oxidation, the presence of undissolved particles, homogeneity of austenite and the size of austenite grains (Lakhtin, 1983). On the other hand, nucleation rate, N depends mainly on composition and for a particular composition on the degree of undercooling (Gulaev, 1980).

Usually, transformation results in interfaces between the parent and product phases. The nucleation site density, N_s has been found (Onuoha, 1986) to show direct relationship with the grain boundary width, X and the length of grain edge, d according to the equation.

$$N_s = \left(\frac{x}{d}\right)^{1-n} \left(\frac{N}{v_s}\right) \dots \dots \dots 2.10$$

Where:

x/d is usually of the order of 10 and N/V_0 ranges from 10^2 to 10^3 for $m=2$ to 10^3 for $m=0$.

Upon quenching to the isothermal transformation temperature, the sufficiently diffusive carbon is first redistributed throughout the austenite leading to the formation of various volumes of austenite, some enriched in carbon and others depleted of it (Singh, 2005).

The austenite with low carbon content and M_s point in the temperature range required for bainite formation, nucleates bainitic ferrite by shear displacive mechanism. Bainitic-ferrite plate possesses habit plane that are characteristically irrational in nature just as in martensite.

After nucleation, acicular ferrite grows from the austenite by rejecting carbon into the surrounding austenite. The formation of Fe_3C , associated with characteristic bainite transformations is suppressed by the high silicon content of the ductile iron resulting in bainite-like structure with a substantial amount of high carbon content retained austenite stable to very low temperatures. Upon extended holding at the austempering temperature, the austenite enriched in carbon then decomposes into ferrite plus carbide (Higgins, 1980).

One of the fundamental characteristics of bainitic reaction is that both upper and lower bainite exhibit a definite surface relief, which suggests some similarity with martensitic transformation. Lower bainite exhibits a single uniform surface relief across each plate, while multiple surface in upper bainite is because each relief is associated with the formation of individual lath. But Bainite plates grow with time, and new plates are also nucleated (Singh, 2005).

Bainitic formation takes place only when the temperature of isothermal reaction falls below a definite temperature designated as B_s , which falls for alloy steels, is given by:

$$B_s (^{\circ}\text{C}) = 830 - 270\% (\%C) - 37(\%Ni) - 70 (\%Cr) - 83 (\%Mo) - 90(\%Mn) \dots 2.7$$

In alloy steels, the amount of bainite formed increases with time when the steel is held between B_s and B_f temperatures, but transformation does not go to completion (Singh, 2005). As the morphology of lower bainite is very much similar to martensite, it is often difficult to fix the B_f temperature. Bainite reaction may extend into martensitic region, that is below the M_s temperature. To an approximation, the lowest bainite formation temperature (B_f) may be obtained by equation;

$$B_f (^{\circ}\text{C}) = B_s - 120^{\circ}\text{C} \dots \dots \dots (2.8)$$

The growth of bainitic-ferrite plate requires the local diffusion of carbon, which also helps to nucleate finely dispersed carbides. This constitutes of the differences with the growth of martensite plates, where no diffusion of carbon occurs. No diffusion of substitutional alloying elements occurs in the bainitic reaction, which constitutes one of the differences with pearlite reaction, where these solute atoms partition between ferrite and cementite when pearlite forms.

Continuous cooling of carbon steel and cast irons enhances formation of bainites because of the shielding of the ferrite and pearlitic reactions. In the same way, substitution alloying elements retard ferritic and pearlitic reactions to further promote bainite formation during continuous cooling. In this way, low carbon steels with 0.5% Mo and 0.002% B have complete bainitic structure.

Ferrite is the nucleating phase in both types of bainite. It is confirmed that lower bainitic ferrite appears to form by shear. Surface relief produced by individual's laths of upper bainite also indicates shear mechanism to have produced them. The ferrite laths formed for the lower bainite is more supersaturated with carbon than those for the upper bainite.

Carbon diffusion takes place in carbon enriched surrounding the bainite plate in upper bainite to precipitate cementite at austenite-ferrite interface. The carbon-depleted austenite now transforms to ferrite by shear mechanism. Thus, growth of existing bainite plates and nucleation of new plates takes place simultaneously.

In lower bainite, ferrite plates, formed by shear mechanism, are highly supersaturated with carbon, diffusion of carbon occurs in this ferrite, which is also the controlling step of formation of lower bainite, and the carbides are precipitated in the ferrite itself. The nature of carbide formed is dependent on the composition of the steel and cast iron and the temperature of transformation (Singh, 2005).

2.11 The Morphology of Bainite Structures

Austempering temperature influences bainite morphology, the proportion of retained austenite and martensite formation. Austempering time is important when the processing window is reduced or closed (Singh, 2005). Martensite formation is favoured by low austempering temperature and short holding time. There are two different types of bainite in cast irons based on structural differences.

1. Upper bainite

The upper bainite, usually produced at temperatures above about 370°C, have a coarse structure of acicular ferrite in austenite. Also called feathery bainite,

due to its resemblance of a bird's feather, upper bainite forms in the temperature range of between 400°C to 550°C. The fine plates of carbide are parallel to the direction of growth of Bainite.

Transmission electron microscopy shows upper bainite to have ferrite laths running parallel to the longer axis with carbide precipitation mainly at lath boundaries. These ferrite laths have still finer structure consisting of smaller sub-laths (called sheaves) about 5µm wide with high dislocation density. With decrease in the temperature of transformation, the laths become finer and closer to cause smaller spacing of the carbide plates between laths and there is an increase in dislocation density (Singh, 2005).

The essential feature of bainite is that diffusivity of carbon is high enough to cause the partition of carbon between ferrite and austenite, i.e., when bainitic laths of ferrite ($C < 0.03\%$) grow, enrichment of carbon occurs in austenite, and thus cementite does not precipitate within the laths, but in austenite at lath boundaries, when carbon becomes critical enough to cause their precipitation. These carbides are present as discontinuous stringers in low carbon steels but as continuous stringers when the carbon content is high in steels and cast irons (Singh, 2005).

2. Lower Bainites

Also called plate bainite, has martensite-like acicular appearance as it forms as individual plates adopting a lenticular shape, and forms in the temperature range of between 250°C to 400°C. The bainitic ferrite plates have smaller sub-plates with low angle boundaries between them. These have higher dislocation

density than in upper bainite, but lesser than in martensite. The carbides in lower bainite are finer and precipitate within the ferrite plates at an angle of 55 to 60° to the bainite plate axis. The carbides have shapes of rods or blades and either cementite, ϵ -carbide, or a mixture of both depending on the temperature of transformation and the composition of the iron (Singh, 2005).

Alloying elements, if present, do not diffuse in bainite structures and do not form their carbides. Silicon as usual encourages the formation of ϵ -carbide as it retards the nucleation of cementite, but other alloying elements allow cementite to form.

The lower bainites, produced at temperatures between M_s point and below 300°C, exhibit finer structures. The structure of the lower bainite resembles that of the typical upper bainite in that there is lath formation but is different because cementite plates are formed at the lath boundary.

Depending on the holding time of the austempering temperature, the micro structural combinations differ. Too short a holding time results in the formation of martensite on quenching while extended holding results in carbide precipitation both in combination with the acicular ferrites. Moderate holding fields the acicular ferrite and retaining austenite combination (Singh, 2005).

2.12 The Processing Window

The austempering is conventionally defined in two stages. The end of the first stage corresponds to the maximization of the fraction of bainitic ferrite and the enrichment of the austenite, the second with the onset of carbide precipitation. The time interval between these two stages is the heat treatment window. During this

interval, only minor changes in the morphology and composition of the ferrite and stabilized-austenite microstructure take place. The extent of this heat-treatment window depends on many factors, including the chemical composition, segregation patterns and austempering temperature. Manganese effect and high contents of this element can effectively shrink or even eliminate the heat-treatment window due to overlap of stage I and II. A closed processing window means that the optimum mechanical properties cannot be achieved due to the severity of the overlap. Martensite occurs in region with less carbon-enrichment of austenite, and carbides in those regions where II has started prematurely because of chemical segregation.

2.13 Effect of Structures on Mechanical Properties of Austempered Nodular Irons

Austempering provides nodular irons with a wide range of mechanical properties characterized mainly by tensile strength and toughness. The properties achieved depend principally on the austenitizing and austempering conditions.

2.13.1 Austenitizing Temperature and Time

Austenitizing as a first step in any austempering treatment is usually carried out in the irons ((Harding, 1986), Rossi (1981)). The equilibrium carbon content of austenite increases as temperature increases between 0.60%C, at 770°C, to about 1.4wt%C at 1000°C (Rimdan and King, 1982). When the carbon content of austenite is raised it will lower the martensite start, M_s point thereby lowering the bainite transformation temperature and delaying the B_s times.

From the work of Harding (1986) austenitizing time does not significantly affect the mechanical properties of austempered irons austenitized at higher temperatures.

2.13.2 Austempering Temperature and Time

During austempering, ductile iron undergoes two-stage transformation process. In stage I, the austenite (γ) decomposes into ferrite (α) and high carbon austenite (γ_{hc})



If the sample is held at the austempering temperature for too long, then a next reaction (stage II) takes place where high carbon austenite further decomposes into ferrite and carbide:



The product of this second reaction is not the most desirable because the carbide embrittles the iron and degrades the mechanical properties.

The mechanical properties of austempered ductile iron are related to three microstructural variables: bainite morphology, proportion of retained austenite and martensite formation. The formation of martensite is favoured by low austempering temperature and short holding times (Harding, 1986). The bainite structure in which lower bainite predominates have higher strength and lower ductility than those in which upper bainite is the major constituent.

Some austenite is usually retained in irons having either upper or lower bainite structure after austempering for 0.5 to 3 hours. The amount of retained

austenite in lower bainite structures does not generally exceed 10% (Barr and Janowak, 1983) while in upper bainitic structures much larger contents of austenite is retained and may even be up to between 40 and 60% (Harding, 1986)

2.14 Modeling And Simulation

2.14.1 Modeling

This is a simplified representation of a system or part of a system in a physical mathematical form (Dilwyn and Mike, 1993) that is suitable for demonstrating the behaviour of the system. It is an act of making a simple description of a system or process that can be used to illustrate or explain the system.

2.14.2 Simulation

This is the process of subjecting model to various input or environmental conditions in order to observe the behaviour and subsequently explore the nature of the results obtained from the real world system. It involves the manipulation of the model to create a real life using computer models to study the behaviour that could exist in reality.

2.14.3 Mathematical modeling

This consists of applying mathematical skills to obtain useful answers to real life problems. (Dilwyn and Mike, 1993), that is, the activity of translating real life problem into mathematics for subsequent analysis. A mathematical model will be created and its solution will usually provide information useful in dealing with the original problem. The components of the system are represented by idealized

element that have essential characteristics of the real component and whose behaviour can be described by mathematical equations which are in turn refer to a model and they are very useful in making predictions when solved and interpreted. The activity of modelling is a process which involves a number of clearly identifiable stages. The most helpful way of representing these stages is by means of a flowchart.(Dilwyn and Mike, 1993) and this is as shown below.

1. Model Formulation

Here we denote the significant variables by assigning units and state all assumptions made on the system. The goal to be achieved is quantified by translating it into mathematical equations using relevant mathematical tool such as statistics, numerical methods and operaional research among others.. Each factor is given a sense of value by assigning a state which is usually low case alphabets. In the course of this project, multiple regression analysis as a tool will be used to develop the models.

2. Model Solution

Here, the mathematical model of the problem formulated in the above stage is solved using algebraic and/or numerical methods, calculus, statistics and graphs. (Dilwyn and Mike, 1993) among other tools. The selection of a particular tool to be used depend on the nature of problem to be solved, parameters involved, factors to be considered and the aim to be achieved. Write computer program or use a prepared package if available and use a simulation package if necessary.

Extract values for the variables that we want either in tabular or graphical form. (Dilwyn and Mike, 1993).

3. Model Validation

Once the model equations are formed and solved, the next stage of the model building is that of testing if the solution obtained is reasonable in serving the purpose which it was meant i.e. whether it conforms to reality. This is done by interpreting the obtained result followed by its validation. This is achieved by ensuring that the model solution answer the following questions. Does the prediction agree with real data?; Has the model fulfil its purpose when it is evaluated?; Can the model be significantly improved by greater mathematical sophistication? and do the interim results suggest that more accuracy is needed by rerunning with an improved model? (Dilwyn and Mike, 1993). If the first two questions are yes, go on to box 6 of figure 2.4, but if otherwise, go back to box 1. This is important; very often the modelling cycle is traversed a number of times before the results are satisfactory. The Statistical methods of testing will be used to validate the model to be developed in this research work.

4. Report

The last stage is the writing of the report in such a way that the important features are clear and the result that is required be read stand out. Who is the report for and what do the readers want to know? Also, how much detail is required in the report are important.

2.14.4 Model Development/Formulation

Multiple regression analysis techniques have been used in this work for the models development and is based on building a probabilistic model that relates a dependent variable Y to more than one independent or predictor variable denoted by $X_1, X_2, \dots, X_n$; (Oyetunji, 2007 and Anderson, et. al 1981).

A general additive statistical multiple regression model, which relate independent variable Y to predictors variables $X_1, X_2, X_3, \dots, X_n$ is of the form

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \dots + \beta_n X_n + \varepsilon \dots \dots \dots (2.1)$$

Where:

$\beta_0, \beta_1, \beta_2, \dots, \beta_n$ are constant parameters, and ε is a random variable to take care of error (Azeez, et. al. 2004).

Statistically, the random variation ε is assumed to be normally distributed with mean value 0 and normal distribution for various values of $X_1, X_2, X_3, \dots, X_n$.

According to Azeez, et. al. 2004 and Mendenhall, et. al. 1981, $E(\varepsilon) = 0$.

then

$$E(Y) = \beta_0 + \beta_1 X_1 + \dots + \beta_2 X_2 + \dots + \beta_n X_n \dots \dots \dots (2.12).$$

From a practical point of view, ε acknowledge the inability to provide an exact model for nature. (Mendenhall, et. al. 1981).

Thus, just as $\beta_0 + \beta_1 X$ describe the mean value Y as a function of X in simple linear regression, the $E(Y)$ is often referred to as true or population regression function $E(Y)$ while the β 's are the population regression coefficients. The regression coefficient β_1 is interpreted as the expected change in Y associated with a

1-unit increase in X_1 while $X_2, X_3, \dots, X_N$ are held constant. Analogous interpretations hold for $\beta_2, \dots, \beta_n$ (Oyetunji, 2007).

2.14.5 Estimation of Parameters

From the simple linear regression equation,

$$Y = \beta_0 + \beta_1 X_1 + \dots + \beta_2 X_2 + \dots + \beta_n X_n \quad (\text{Wilfrid and Frank J., 1969}), \dots \quad (2.13)$$

the coefficients are determined from the above linear regression equation whereby Y is a dependent variable and $X_1, \dots, X_N$. Are independent variables (Wilfrid and Frank J, 1969).

2.14.6 Least Square Method

This is the best, most accurate and reliable method of solving a regression function (Azeez, et. al. 2004). It is an attempt to minimize the sum of squares of the value difference between the observed values (Y) and the theoretical values $E(Y)$ obtained by solving the regression equations.

Mathematically, least square method utilizes the relationship below:

$$M_{in} = \sum (Y - E(Y))^2 \quad \dots \quad (2.14)$$

Where

Y = Observed value of dependent variable for i th observation

$E(Y)$ = Theoretical value obtained from the regression analysis formula of:

$$E(Y) = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \dots + \beta_n X_n + \varepsilon \quad (\text{Anderson, et. al 1981})$$

Hence the following $Y_1, X_{11}, X_{21}, X_{31}, X_{41}, \dots, X_{n1}$ values form data to be used for the exercise, e.g.

(a) the first set of values will be:

$$Y_1, X_{11}, X_{21}, X_{31}, \dots, X_{N1}$$

(b) the second set of values:

$$Y_2, X_{12}, X_{22}, X_{32}, \dots, X_{N2}$$

(c) kth set of values:

$$Y_N, X_{1k}, X_{2k}, X_{3k}, \dots, X_{Nk}$$

Then the required values for $\beta_0, \beta_1, \dots, \beta_k$ are obtained by solving the equations:

$$n\beta_0 + \beta_1 \sum X_{ij} + \beta_2 \sum X_{2j} + \dots + \beta_N \sum X_{Nj} = \sum Y_j$$

$$\beta_0 \sum X_{ij} + \beta_1 EX_{ij} + \beta_2 EX_{ij}X_{2j} + \beta_N EX_{ij}X_{Nj} = EX_{ij}Y_j$$

$$\dots\dots\dots$$

$$\dots\dots\dots$$

$$\dots\dots\dots$$

$$\beta_0 EX_{ij} + \beta_1 EX_{ij}X_{2j} + \beta_k EX_{ij}X_{kj} + \beta_k EX^2(k) = EX_{ij}Y_j, \dots\dots(2.15)$$

Solving those equations yield the least square estimates $\beta_0, \beta_1, \dots, \beta_k$. In general, the equation (2.15) can be solved manually by first computing all coefficients of β_i s and then using matrices particularly, Gaussian elimination method. (Oyetunji, 2007)

The computations, although not theoretically complex, are difficult even for N as small as 4 or 5. The arithmetic is not only tedious but it is hazardous. Since it is difficult to detect error or to be sure that errors resulting from the use of a limited

number of significant digits are not building up and destroy the accuracy of the results.
(Wilfrid and Frank J, 1969)

2.15. Computer Aided Solution

Generally, multiple regression analysis is solved using computer programs that can routinely generate the values of the least square estimates. In fact, several computer packages have been developed especially to handle a variety of statistical computations, include multiple regression analysis. Three of the more common packages available are BMDP (Biomedical Computer Programs: P series), SAS (Statistical Analysis system) and SPSS (Statistical Package for the Social Sciences). (Anderson, et. al. 1981).

The general procedure that is followed in using such packages is shown below in figure 2.5. However, once data have been inputted into the computer, the regression program takes over and computes the least square estimators together with a variety of other information that is printed out on a report of the user.

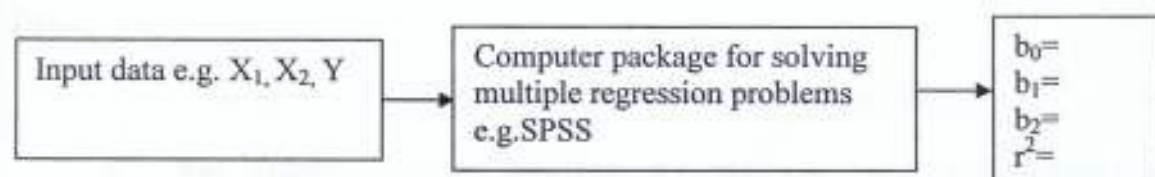


Figure 2.5: General procedure followed in using Computer package.

The same equation;

$$Y = E(Y) + E = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \dots + \beta_n X_n + \varepsilon \dots \dots \dots (2.16)$$

is used for the model and the following assumptions are made in order to justify the use of the models:

1. For any given set of values of $X_1, X_2, \dots, X_n$, E has a normal distribution with mean equal to 0 and variance equal to σ^2 and is the same for each set of Y values
2. The random errors are independent (in a probabilistic sense)
3. The observed values Y are based on a random sample from the assumed normal probability distribution and that each observation is independent of all other observations.

2.16 Model Validation

Model validation involves testing the level of agreement between the theoretical values and the observed or measures values. For this purpose, statistical tests are commonly used and the following tests are popular:

- i. Paired t-test
- ii. Correlation Coefficient
- iii. Standard Error of Prediction

2.16.1 Pair t-tests

The pair t- test technique uses the following nomenclatures:

d = Pair difference between the experimental and numerical data on each column.

$\bar{d}$ = Mean Pair difference

$\text{Var}(d)$ = Variance of Pair difference

$\text{Var}(\bar{d})$ = Variance of mean difference

t_v = Theoretical number of degrees of freedom available for the estimation of the variance i.e. $(n-1)$

N = Number of paired test samples

α = Confidence interval (%)

Sd = Standard deviation

where according to Oyetunji (2007):

$$(\bar{d}) = \sum \frac{(numerical\ data - Experimental\ data)}{No\ of\ paired\ test\ samples} \dots\dots\dots(2.17)$$

$$Var(d) = \frac{\sum d^2 - \frac{(Numerical\ data - Experimental\ data)^2}{No.\ of\ Paired\ test\ samples}}{No\ of\ paired\ test\ samples - 1} \dots\dots\dots(2.18)$$

$$Var(\bar{d}) = \frac{Var(d)}{No\ of\ paired\ test\ samples} \dots\dots\dots(2.19)$$

$$Sd = \sqrt{Var(\bar{d})} \dots\dots\dots(2.20)$$

Acceptable tolerance interval for a given confidence interval and numerical

$$data = \bar{d} + t_{v(1-\alpha)} sd \dots\dots\dots(2.21)$$

2.16.2 Correlation Analysis and Coefficient of Correlation

This is a statistical technique that is used to test for the relationship between two variables. The variables are said to be correlated when an increase or decrease in one variable is accompanied by an increase or decrease in the other. It is commonly used along with the regression analysis to measure how well the regression lines explain the variations of the dependent variable. If an increase in one variable corresponds to an increase in the other, the correlation is said to be positive. But if increase in one corresponds to decrease in the other, the correlation is referring to as

negative. If there is no relationship between the two variables, they are said to be independent (Dass, 2005).

The following relations are methods used to determine simple correlation:

(1) Karl Pearson’s coefficient of correlation ($P_{x,y}$)

$$P_{x,y} = \frac{C_{ov}(X,Y)}{\partial x \partial y} \dots\dots\dots(2.22)$$

where:

$$-1 \leq P_{xy} \leq 1$$

$$C_{ov}(x,y) = \frac{1}{n} \sum_{j=1}^n (x - \bar{x})(y - \bar{y}) \dots\dots\dots(2.23)$$

$$Cov(x,y) = \text{Covariance of X and Y} \dots\dots\dots(2.24)$$

∂x = Variance of X

∂y = Variance of Y

P_{xy} = Correlation Coefficient

$\bar{x}$ = Mean of X

$\bar{Y}$ = Mean of Y

X = Experimental value

Y = Numerical value

N = No. of pair test samples

(2) Pearson’s rank correlation coefficient (R_p)

$$R_p = 1 - \frac{6 \sum d^2}{n(n^2 - 1)} \dots\dots\dots (2.25)$$

(3) Coefficient correlation (Rc)

$$R_c = \sqrt{1 - \frac{\sum (Y_d - Y)^2}{\sum (Y_d - Y)^2}} \dots\dots\dots (2.26)$$

Table 2.1 Rule of Thumb for Interpreting Correlation Coefficient (R) (Aderoba, 1995)

Value of R	Interpretation
0.90 – 1.00	Very high correlation
0.70 – 0.90	High correlation
0.40 – 0.70	Moderate correlation
0.20 – 0.40	Low correlation
0 – 0.20	Slight correlation

2.17 Standard Error of Prediction

This is the deviation of the predicted value from the observed value. It is given by:

$$E_{YX} = \sqrt{\frac{\sum (Y - Y_r)^2}{n}} \dots\dots\dots (2.27)$$

Where:

Y = the actual value (observed)

Y_r = the predicted value (Dass, 2005)

The Karl Pearson's equation for calculating the standard error of the predicted numerical data (Y) is given as:

$$SE_{y-x} = \sqrt{\left[\frac{1}{n(n-2)} \right] n \sum y^2 - \frac{[nExy - (Ex)(Ey)]^2}{nEx^2 - (Ex)^2}} \dots\dots\dots (2.28)$$

where:

Y = Numerical data

X = Experimental data

N = No. of pair test samples

2.18 Software Development

A software consists of series of computer activities designed to perform specific functions. To effectively do this, the computer requires a program, which is a list of instructions for the computer to follow to accomplish the task of processing data into information. A program is written in codes to form a high level specialized programming language. Hundreds of programming languages are available and have been designed for different purposes. These include BASIC (Beginner All purpose Symbolic Instruction Code) Fuzzy logic, FORTRAN, COBALT, PASCAL, DBASE, C-Language, and JAVA. Each of these has various versions for different environments.

2.19 Past Works On Modelling Of Mechanical Properties

Quite a number of researches have been carried out on modelling of mechanical properties of various types of engineering materials, steels inclusive. However, a vast majority of processes are yet to be assessed for study.

Oyetunji (2009) modelled the impact properties of grey cast iron produced from Steel and CO₂ moulds. Oyetunji and Adebisi (2008) developed software for rapid estimation of corrosion rate of austempered ductile iron in corrosive media. Oyetunji (2007) studied extensively on modeling mechanical properties of grey cast

iron by using a statistical method of multiple regression analysis to develop the models. He then use pair t-test, correlation coefficient and standard error principles to validate the model developed before developing a software that can be used to rapidly estimate the properties. MATHLAB programming package was used to develop the software.

Ndaliman, (2006) carried out an assesment of mechanical properties of medium carbon steel under different quenching media. Lalam, et al (2000) worked on the estimation of mechanical properties of ferritic steel welds. The first part of the study focused on yield and tensile strength. He claimed that the trends predicted by the model appeared to be consistent with those expected metallurgically. However, he emphasised that only the simplest trends have been examined since the number of variables involved is very large. The second part of the study, which was on prediction of elongation and Charpy-impact toughness, was found useful. However, much more systematic experimental data are needed before the predictability can be assured.

Chopra, (1994) studied the estimation of fracture toughness of cast stainless steel during thermal aging in LWR systems. In his work, he determined the procedures and correlation for predicting Charpy-impact energy and fracture toughness of the steel used. He estimated the mechanical properties of a specific cast stainless steel from the extent and kinetics of thermal embrittlement, and correlated the aged and unaged impact energy for the estimation. He then concluded that the correlation could be used to assess thermal embrittlement of cast stainless steel component..

Simecek and Hajduk, (2007) used 3D finite elements method to prediction of mechanical properties of hot rolled steel products. The calculated and the measured process datas show satisfactory correspondence for use in control of the process. However, in the case of Myllykoski, et al (1996), the development of a model for predicting mechanical properties of batch annealed thin steel strip by using artificial neural network modelling was found to be sufficiently accurate for use to evaluate the effect of process parameters.

Other works on modeling mechanical properties concern a phase-field modelling of the thermo-mechanical properties of carbon steels, by Jin Seol, et al (2000), microstructures and mechanical properties of Fe-Mn-(Al,Si) TRIP/TWIP steels by Hua DING, et al (2006), statistical modelling of mechanical tensile properties of steels by using neural network and multivariate data analysis by Dumortier, and Leher, (1999).

Sterjovski, et al (2005) used artificial neural networks for modelling the mechanical properties of steels in various applications. He used the model developed to predict:

- (i) Impact toughness of quenched and tempered pressure vessel steel;
- (ii) Hardness of the simulated Heat Affected Zone (HAZ) in pipeline and tap fitting steels; and
- (iii) Hot ductility and hot strength of various alloys steels over temperature range for strand and slab straightening in continous casting process.

It was concluded that the developed model have the capacity to eliminate the need for expensive experimental investigation in areas like testing, manufacturing process, inspectioning and repair.

CHAPTER 3

3.0 RESEARCH METHODOLOGY

3.1 Materials

All ductile iron test specimens, used for this research work, were produced from castings sourced from Madison in USA. The test specimens were cut into 20mm by 20 mm size. Normal materials for metallographic specimen preparation, such as abrasive papers, polishing cloth, and diamond paste, water as coolant, etchant, cotton wool, tissue paper and desiccators, were adopted.

3.2 Equipment

A mass spectrometric analyzer was used to determine the chemical composition of the samples. Two heat treatment furnaces (Vecstar and Carbolite) were used, one for the austenitization of specimens at 850⁰C and the other for austenite transformation at selected lower temperatures. An electric oven was used for austenite transformation at very low temperatures.

An electric heater was used to heat water, for carrying out interrupted cooling, to temperature of 80⁰C while a mercury thermometer was used for measuring the temperature of the water. Stop watch was used for all time keeping.

Hardness test specimens were cut from the as-received ductile iron castings using ordinary hack-saw. After this, one the surfaces was ground flat for subsequent hardness measurements and metallographic works, which involved the use of:

- i) a digital Rockwell hardness tester in accordance with (Oyetunji, 2007), (Dibiezue 1997).
- ii) grinding and polishing machines.



Micrographic works were carried out using an optical microscope attached with a digital camera for taking micrographs of microstructures. Finally, computer system and laser jet printer were equally made used for processing the obtained data and report typing and printing

3.3 Heat Treatment of the As-Cast Ductile Iron

All heat treatment operations were carried out in the Department of Metallurgical and Materials Engineering, Federal University of Technology, Akure.

3.3.1 Normalising

A total number of 103 hardness specimens were normalized by austenitizing at 850°C for 60minutes before the final air-cooling. This was a preliminary heat treatment to achieve the necessary structural and compositional homogenization of the as-cast condition.

3.3.2 Production of Martempered Ductile Iron (MDI) by Interrupted Quenching.

The normalized samples were again austenized at 850°C for 30minutes after which they were quenched in warm water, which had been maintained at 80°C , for a holding period of 40seconds. After this, the samples were quickly transferred to another furnace (or oven), which had been maintained at a constant temperature of between 250°C and 450°C , for tempering process. The holding time for tempering in the furnace or oven was varied between 30 and 180 minutes after which the specimen was air cooled down to room temperature.

3.3.3 Hardness Test

The hardness tests were performed with the aid of a digital Rockwell hardness tester in accordance with ASTM. A diamond indenter under the application of 150 KN was pressed into the surface according to the machine operation. For each test specimen, three hardness measurements were taken and the averages of the three readings were calculated. Table 4.1 contains the result of all the hardness measurements and figures 4.1 for the plots of hardness against holding time at various tempering temperatures.

3.3.4 Metallographic Analysis

This stage involved the usual procedure of cutting, grinding, polishing, etching, micro-examination and photo-microscopy (micrograph) of the tests specimens. All necessary procedure and precautions were considered during the processes. Samples size was adequate for manual grinding and polishing.

The specimens were thereafter ground on a series of silicon carbide papers of increasing fineness beginning from 200-grit, 300-grit, and 400-grit and finally 600-grit papers. The usual standardized procedure of rotating through 90° when changing from one grade of paper to the next was observed. Running water was used for cooling and lubricating the specimens during grinding. The specimens were washed under running water in readiness for the next stage.

Polishing was carried out using a nylon cloth attached to a rotating disc and a diamond paste as polishing agent. Polishing was continued until a mirror-finish appearance was obtained.

Etching was done with the aid of cotton wool using Nital (i.e. 2% of Nitric acid and 98% ethanol). Swabbing was done for 3 seconds after which the surface was rinsed under running water, then methylated spirit to remove water stain from the surface and finally dried in warm air.

3.3.5 Microscopic Examination

The specimen was viewed using a metallurgical microscope digital camera and computer system attached. Photomicrographs of microstructures and are in Plates 1-15.

3.4.0 Model Development and Validation

The computer modeling exercise was based on the hardness data generated for the heat treatment operations by the interrupted quenching method as described above. Hardness data had been chosen because it is easy to generate large data this way.

3.4.1 Model Development

Statistical analysis was used to develop the models. The results of the hardness tests were analyzed statistically by using multiple linear regression analysis. The computation of the coefficients for the multiple regression equation was obtained with the aid of computer in a stepwise technique. The package used was statistical package for social science (SPSS). The independent variables were temperature and time. The equation of the model so developed is shown as Equation 4.1.

3.4.2 Validation of the Developed Models

Statistical tests were used to validate the developed model. This is done by comparing the experimental hardness values with the theoretical data generated using the model Equation 4.1. The following statistical test methods were accordingly employed:

- i) Paired t-tests
- ii) Correlation Coefficient
- iii) Standard Error of Prediction

1. Paired t-tests

The calculated values of the variance, standard variation and the intervals, as well as the coefficient of correlation and standard error, are shown in Table 4.4.

2. Correlation Coefficient

The interpretation of the correlation coefficient test was by using the rule of thumb given by Aderoba (1995) and Okoro (2000). The results obtained for the tests are summarized in Table 4.5

3. Standard Error of Prediction

This measures the amount of error in predicting the hardness values of the ductile iron heat treated by interrupted quenching in warm water. The results are contained in Table 4.5.

3.5 Development of Computer Software

The multiple linear regression equation obtained was used to develop the flow chart and algorithm, which were then used to develop software that facilitated the rapid implementation of the developed models.

3.5.1 Development of Algorithm and Flowchart

The flowchart and algorithm were developed for the model. The flowchart developed is as shown in Appendix II; while the user friendly VISUAL BASIC 6.0 package was used to develop the algorithm in a familiar manner is contained in Appendix II.

3.5.2. Computer Software Development

The flowchart in Figure 4.3 and the algorithm in Appendix II developed for the two-type comprehensive models were both made used for the computer software design/coding of the system shown in Appendix III. The following are the keys to the flowchart developed.



4.0 RESULTS

The results of the experiment are shown in table 4.1 below.

4.1 HARDNESS DATA

Table in Appendix A shows the hardness values for the ductile iron samples which had been subjected to interrupted quenching in warm water prior to heating to and holding at various times and various elevated temperatures. It is seen that the hardness values varied between 52.9Rc, at the lowest temperature, and 18.3Rc at the highest temperature.

From this result, it is expected that a wide range of matrix structures will be obtained. This expectation is based on the fact that specific hardness (R_c) values are commonly associated with different matrix structures in ductile irons as contained in the ASTM A-536-70 Standard, and are given as follows:

- Ferrite/Pearlite - 18 – 25Rc.
- Pearlites - 25 – 30Rc.
- Bainites - 30 – 40Rc.
- Tempered Martensite - 30 – 40Rc.
- Martensite - Above 40Rc.

These hardness values are plotted, Figure 4.1, against the holding temperatures at constant holding times.



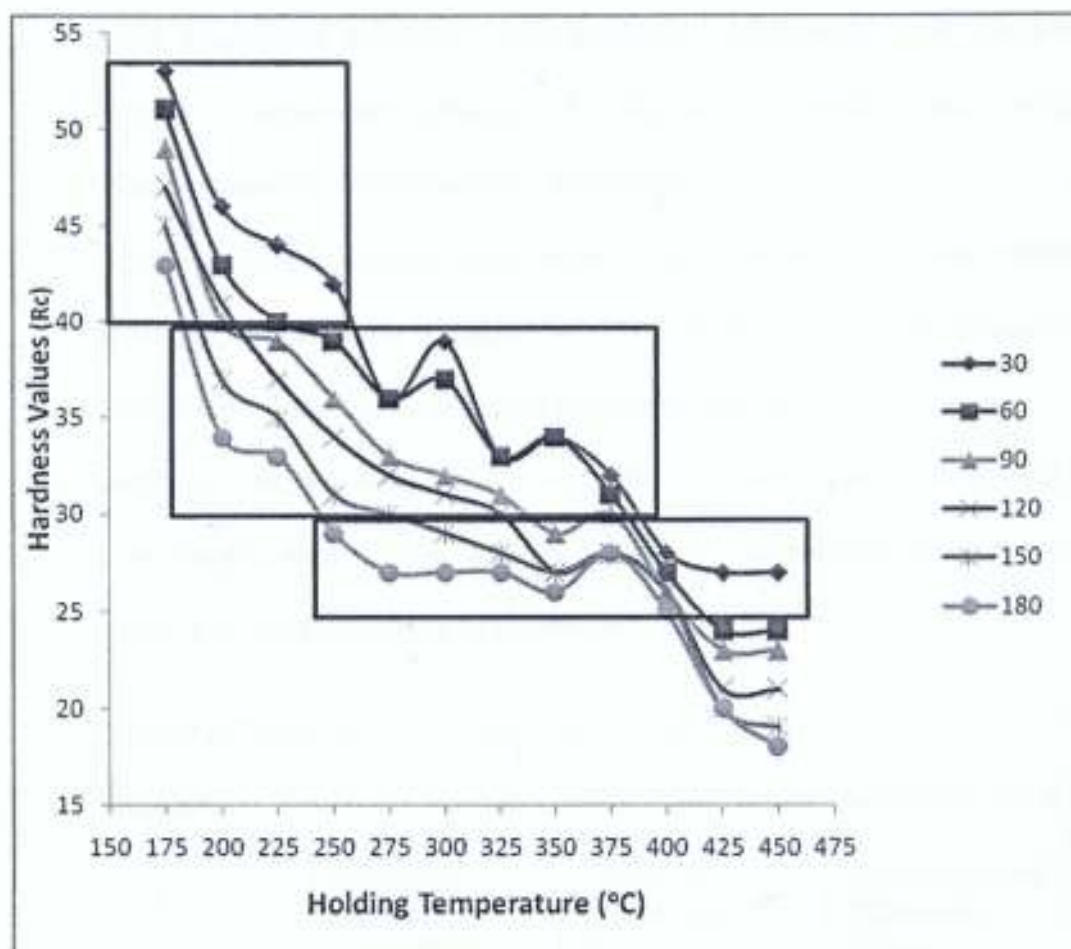


Figure 4.1 Variation of hardness values with different holding temperature.

From this figure and based on hardness values associated with different matrix structures, it is to be expected that:

1. Martensite structures, with hardness values between 40 R_c and 52,9 R_c , would be obtained at holding temperatures below 250°C. This has shown that martensite structures were formed immediately after interrupted quenching in warm water, prior to transfer into the holding furnace, and that the M_s temperature for the ductile iron was above 80°C as confirmed by the CCT diagram in Figure 2.2.

2. Since martensite structures were produced immediately after interrupted cooling in warm water, subsequent heating will only result in transformation of any retained austenite and prior martensite.
3. Matrix structures, with hardness values between 30 and 40 R_c , were produced at holding temperatures between 275°C and 375°C. These hardness values are typical for bainite and or tempered martensite structures.
4. Hardness values between 25 and 30 R_c were produced at holding temperatures above 375°C and are typical for unhardened structures which result from over tempering of martensite.

Table 4.2: Prediction of Microstructures from Hardness Values

Holding Temperatures (°C)	Holding Times for which structures were obtained (min.)		
	Untempered Martensite (Type A)	Tempered Martensite (Type B)	Over-tempered Martensite (Type C)
175	0 - 180	----	----
200	0 - 120	120 - 180	----
225	0 - 60	60 - 180	----
250	0 - 60	60 - 180	----
275	----	0 - 120	150 - 180
300	----	0 - 150	150 - 180
325	----	0 - 120	120 - 180
350	----	0 - 60	60 - 180
375	----	0 - 60	90 - 180
400	----	0 - 30	60 - 180
425	----	----	0 - 180
450	----	----	0 - 180

The formation of tempered martensite structures, with mechanical properties comparable to those of bainites, has provided another process route for producing strong, hard and tough ductile irons with mechanical properties comparable to austempered ductile irons.

4.2 CHARACTERISTIC HARDNESS-TIME PROFILE

Figure 4.1 show the plots of hardness values against holding times at a fixed tempering temperature. It is seen that the hardness of the specimens reduced with holding time irrespective of structure formed. This is because of relief of internal stresses and continued carbon diffusion, especially in the martensite structures. Both of these reduce the internal energy in the structures.

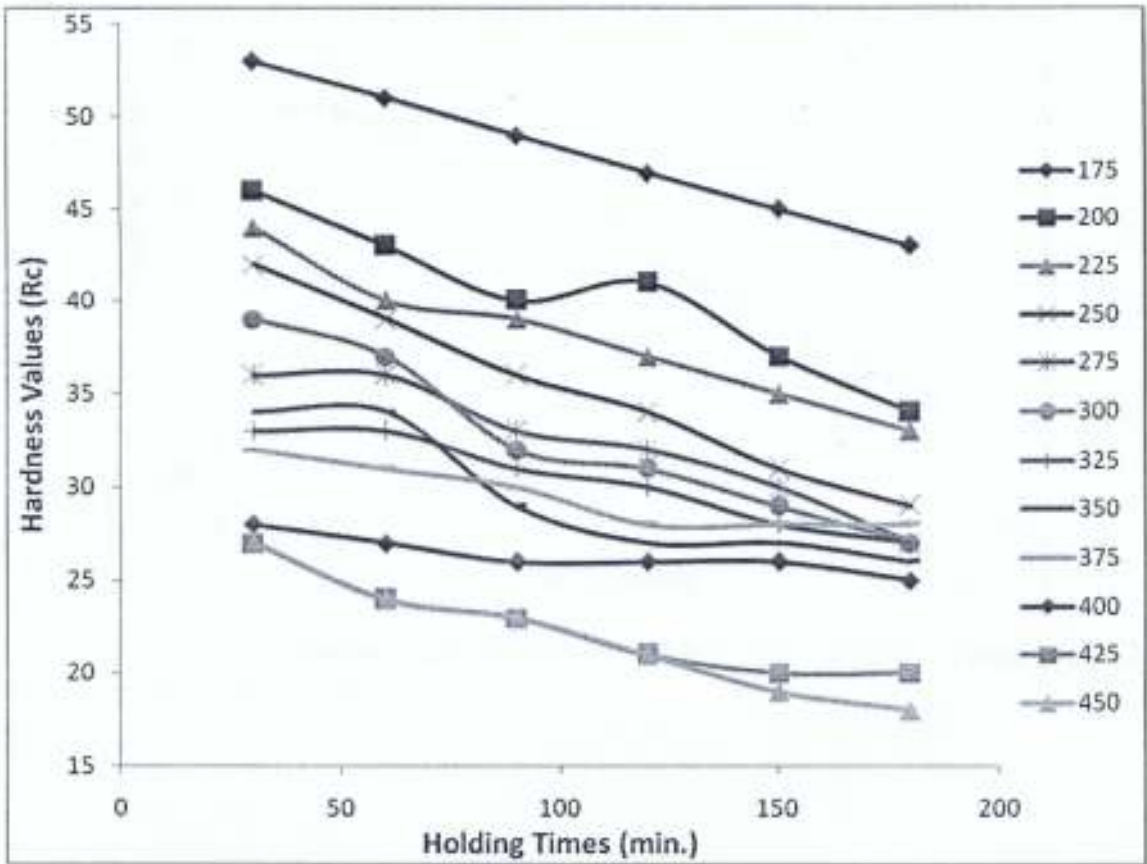


Fig. 4.2: Hardness Values against holding times for different Tempering Temperatures.

The relationships are presented in Figures 4.3(a) to 4.3(l). Expanded plots show that the plots can be classified into two main groups in accordance to the shape or profile.

1. Group-A:

This is the group for which the hardness profile is essentially a continuously decreasing hardness, as in Figures 4.3(a) to 4.3(d), and at holding temperatures of 250°C or less, when only limited tempering has occurred.

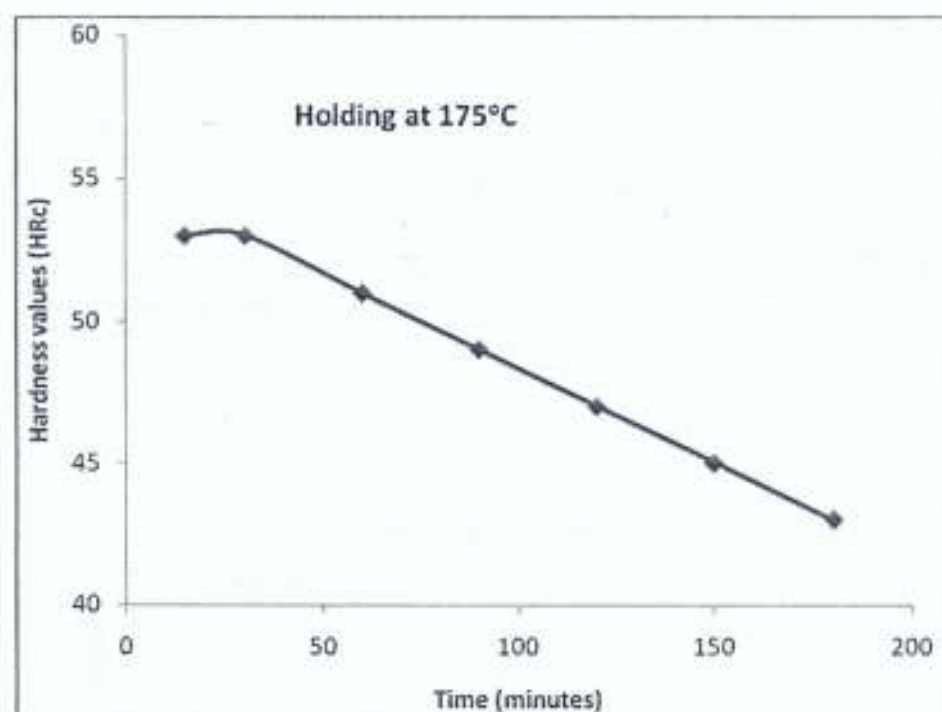


Fig. 4.3(a): Variation of Hardness Values with holding Times at a holding Temperature of 175°C .

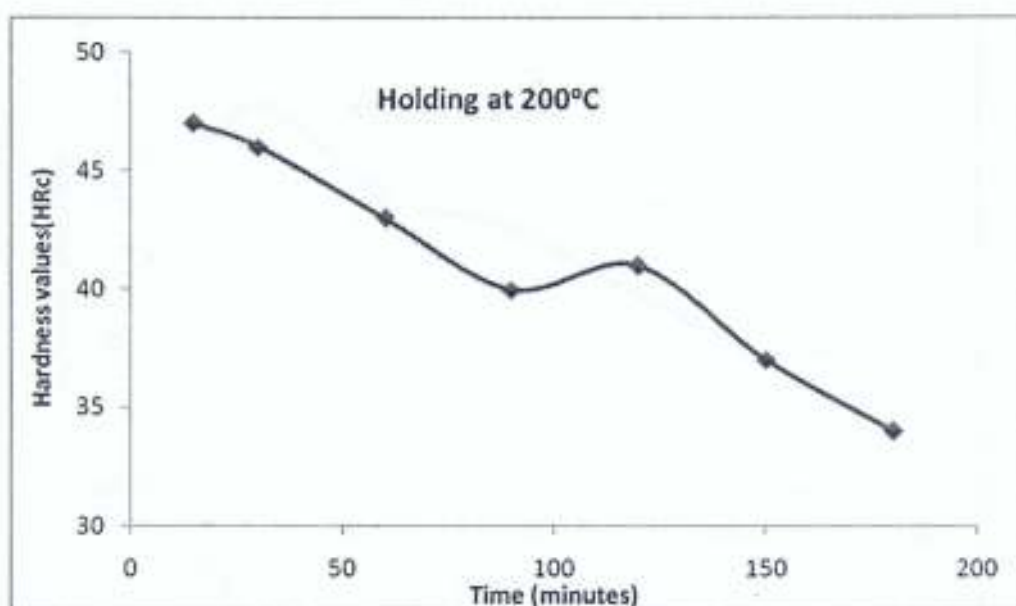


Fig. 4.3(b): Variation of Hardness Values with holding Times at a holding Temperature of 200°C.

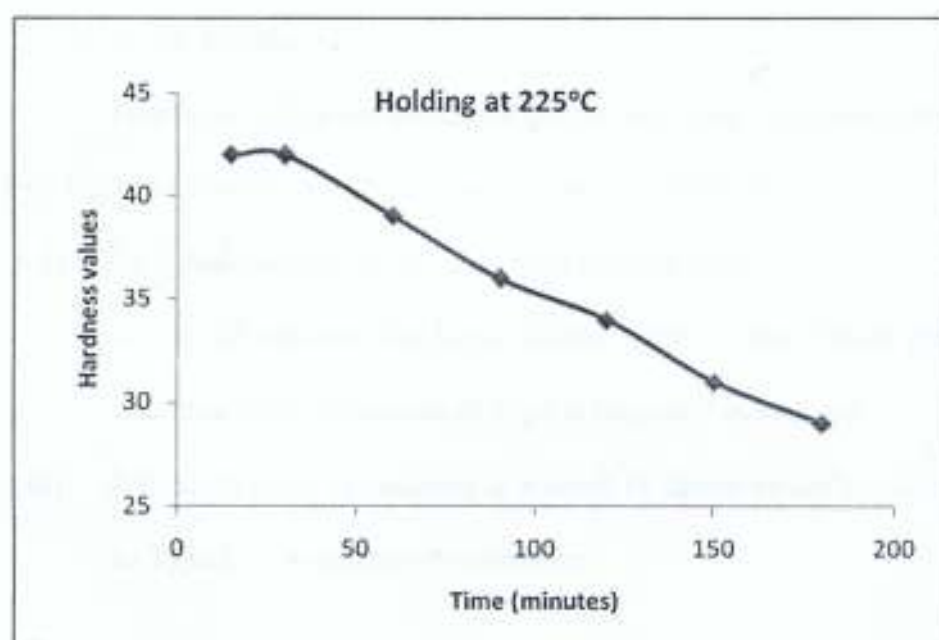


Fig. 4.3(c): Variation of Hardness Values with holding Times at a holding Temperature of 225°C.

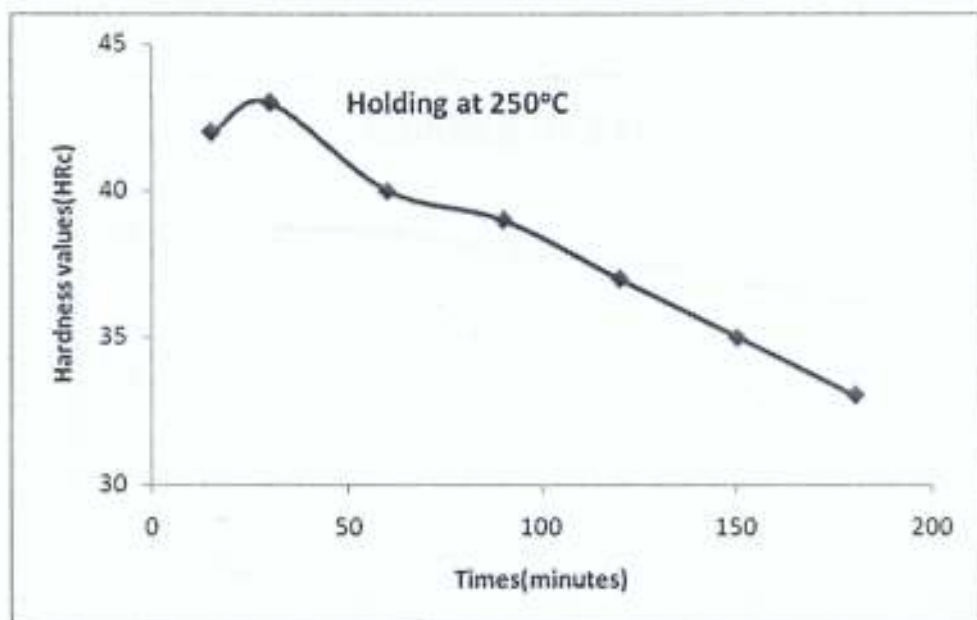


Fig. 4.3(d): Variation of Hardness Values with holding Times at a holding Temperature of 250°C.

2. Group-B: Profile

This is the group for which the profile has three main sections as represented by Figures 4.3 (e) to (h). The curves are characterized by:

- i) An initial decrease in hardness with holding time.
- ii) A bay of constant hardness, where there is short range carbon diffusion resulting in the formation of Type B tempered martensite.
- iii) A final decrease in hardness as a result of extensive carbon diffusion resulting in Type C over-tempered martensite.

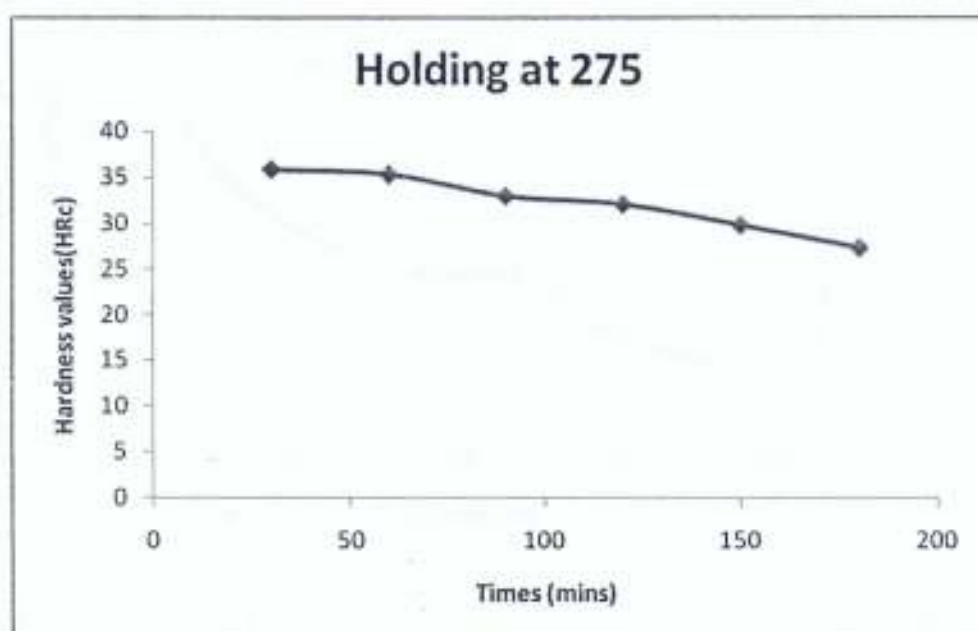


Fig. 4.3(e): Variation of Hardness Values with holding Times at a holding Temperature of 275°C

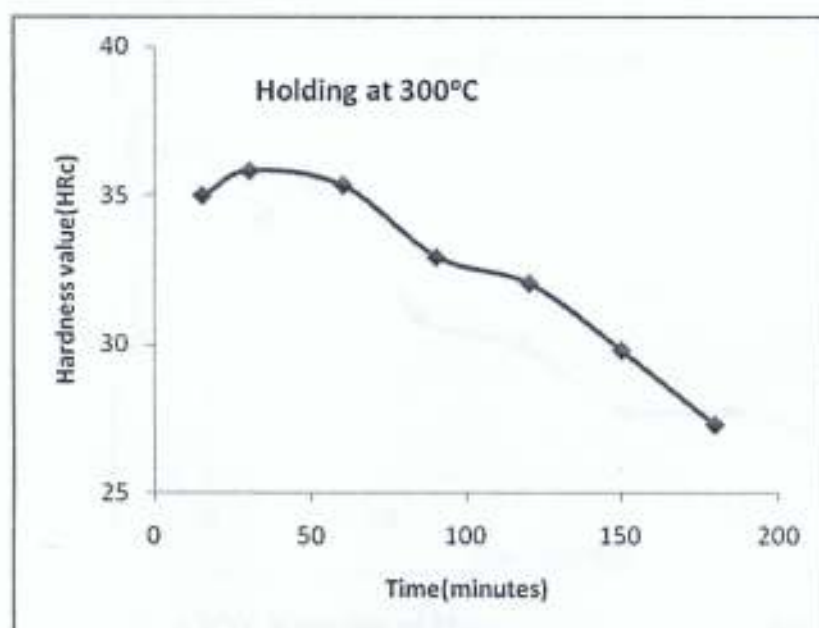


Fig. 4.3(f): Variation of Hardness Values with holding Times at a holding Temperature of 300°C

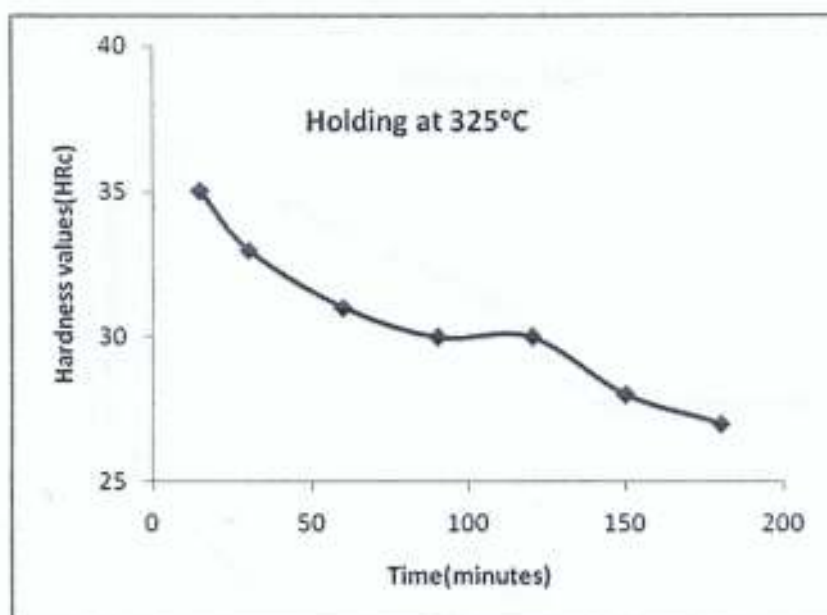


Fig. 4.3(g): Variation of Hardness Values with holding Times at a holding Temperature of 325°C

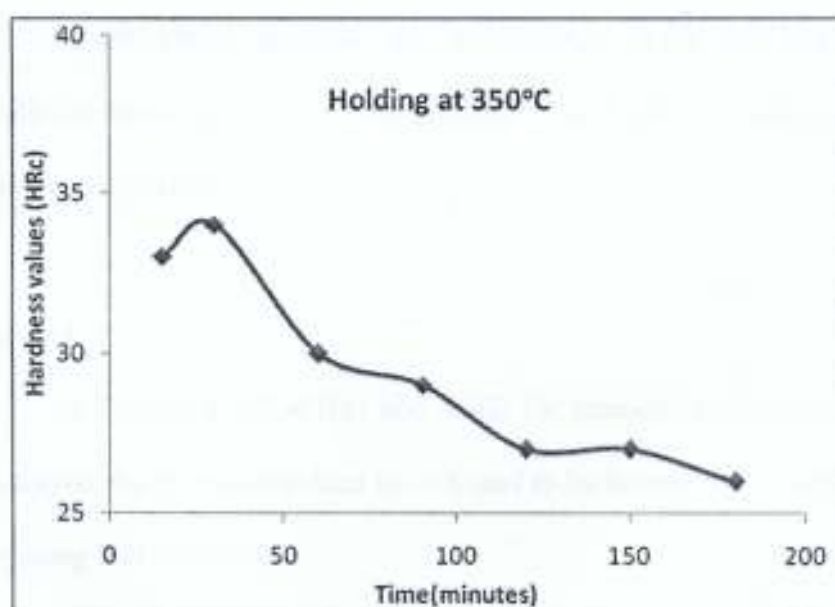


Fig. 4.3(h): Variation of Hardness Values with holding Times at holding Temperature of 350°C

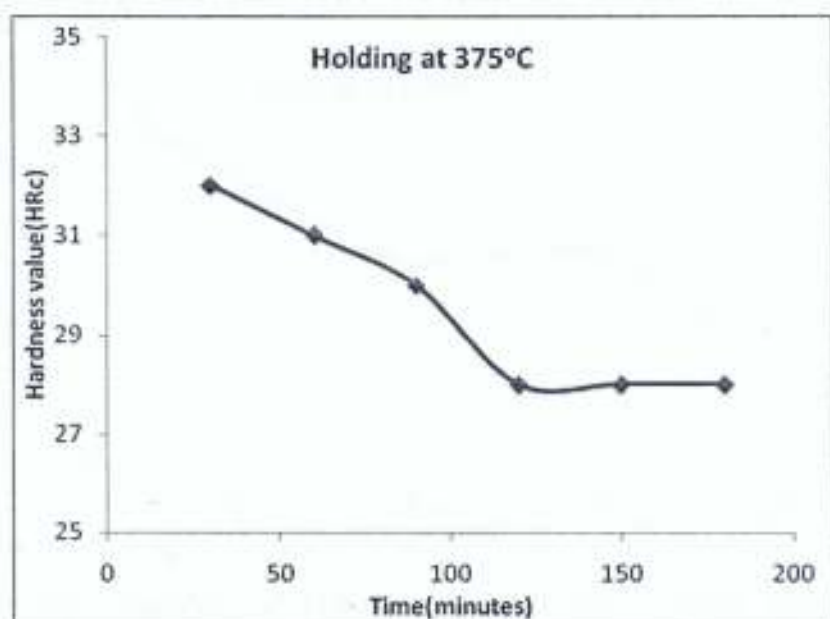


Fig. 4.3(i): Variation of Hardness Values with holding Times at a holding Temperature of 375°C

From the above analysis, the temperature range for Type B tempered martensite has been found to be between 250 °C and 375 °C, which is in agreement with the results in Table 4.1.

3. Group C

In Figures 4.3(j) 4.3(k) and 4.3(l) for tempering temperatures of 400 °C and above, the hardness values were found to be below 30Rc indicating that over tempering had occurred.



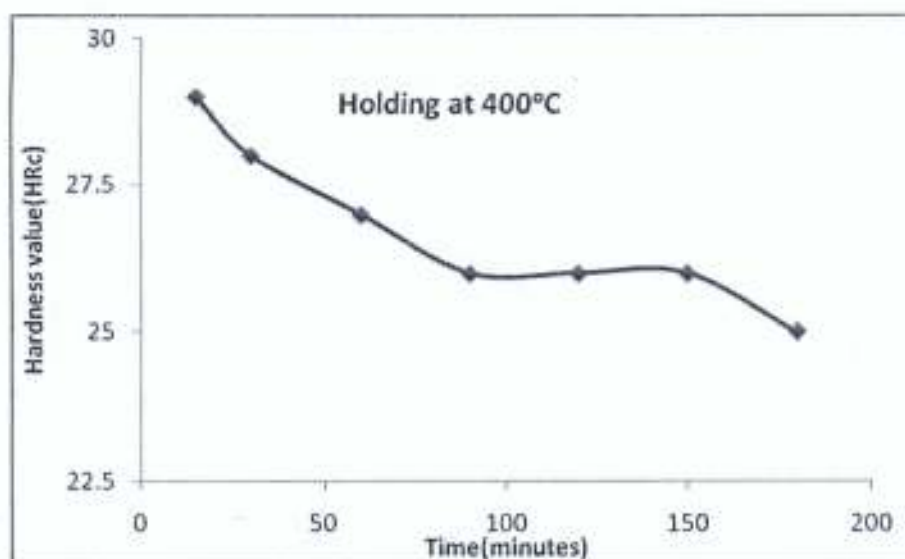


Fig. 4.3(j): Variation of Hardness Values with holding Times at a holding Temperature of 400°C

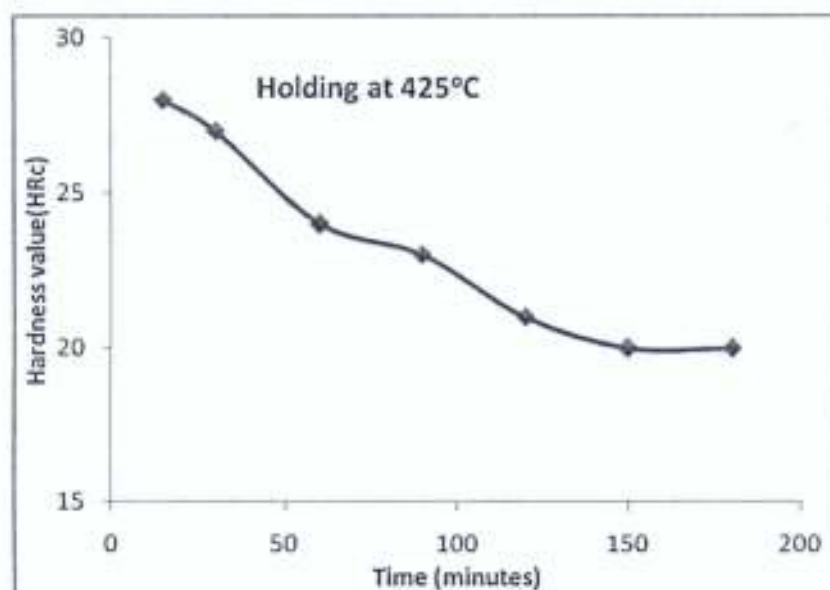


Fig. 4.3(k): Variation of Hardness Values with holding Times at a holding Temperature of 425°C

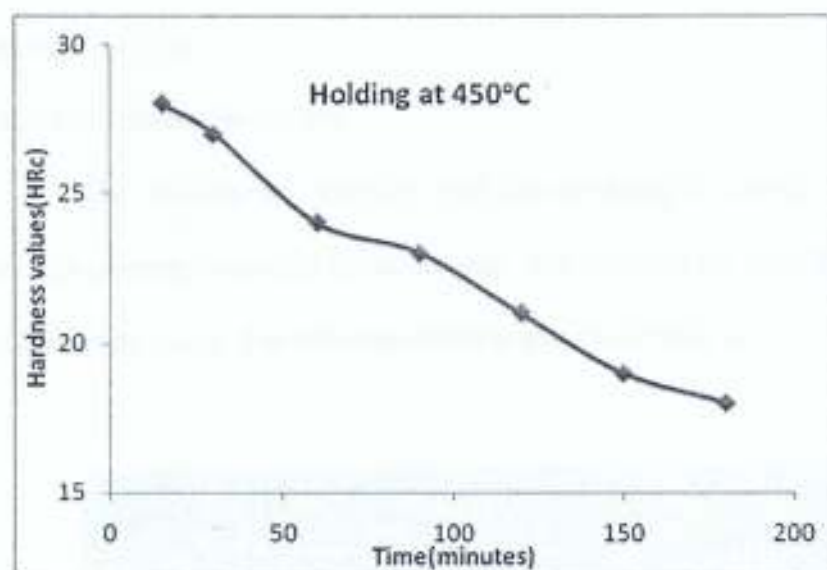


Fig. 4.3(l): Variation of Hardness Values with holding Times at a holding Temperature of 450°C

4.4 Microstructures

4.4.1 The As-received Structure

The as-received samples had ferritic-pearlitic matrix structure, with few pearlite colonies scattered in the matrix. The nodules are well formed and contained in the ferrite phase. The microstructure is shown in Plate 1.

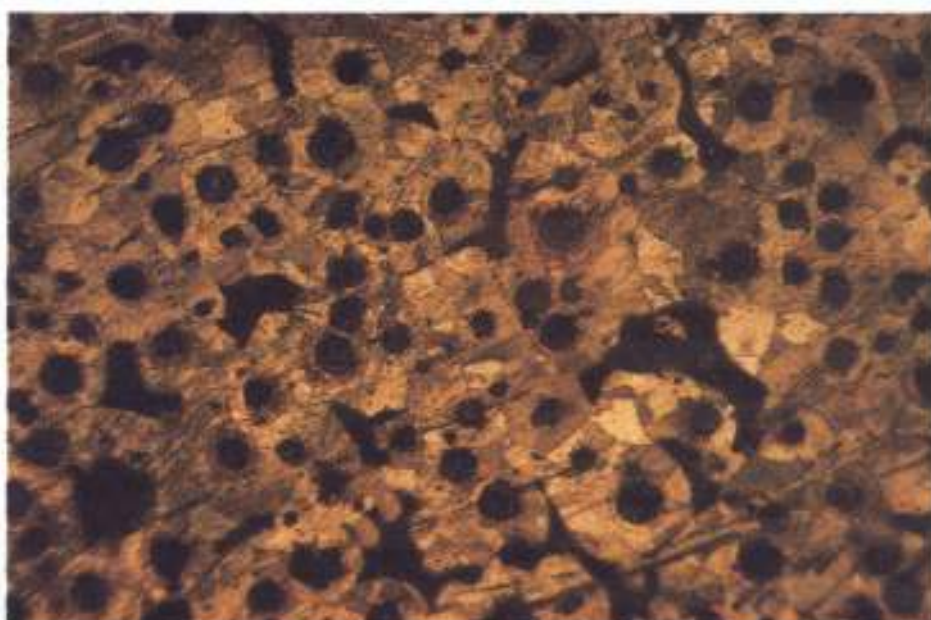


Plate 1: The microstructure of the as-received samples ((etched with 2% nital and at 100X)

4.4.2 Heat Treated Structures

As has been pointed out earlier the structures to be expected have been grouped into three classes:

- i) TYPE A: Martensitic structure, with or without retained austenite.
- ii) TYPE B: Predominantly tempered martensite.
- iii) TYPE C: Principally over-tempered martensite.

4.4.1 Type-A Structures

Plates 2 to 5 contain microstructures obtained after holding between 175°C and 225°C. It is very clear that the structures obtained are essentially martensitic, which confirms the experimental expectation.

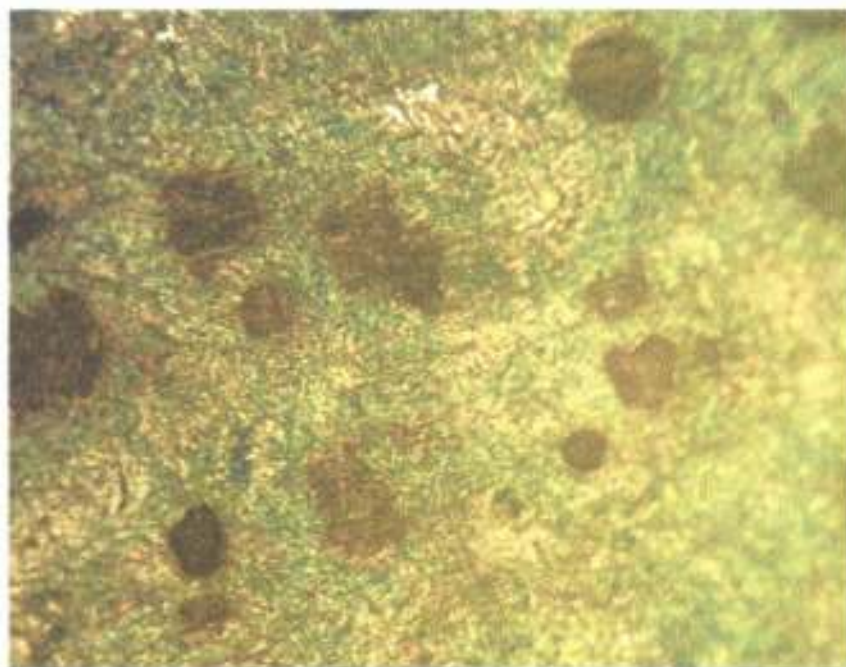


Plate 2: Ductile Iron held at 175°C for 30minutes showing clearly the untempered martensite plates (etched with 2% nital and at 200X)

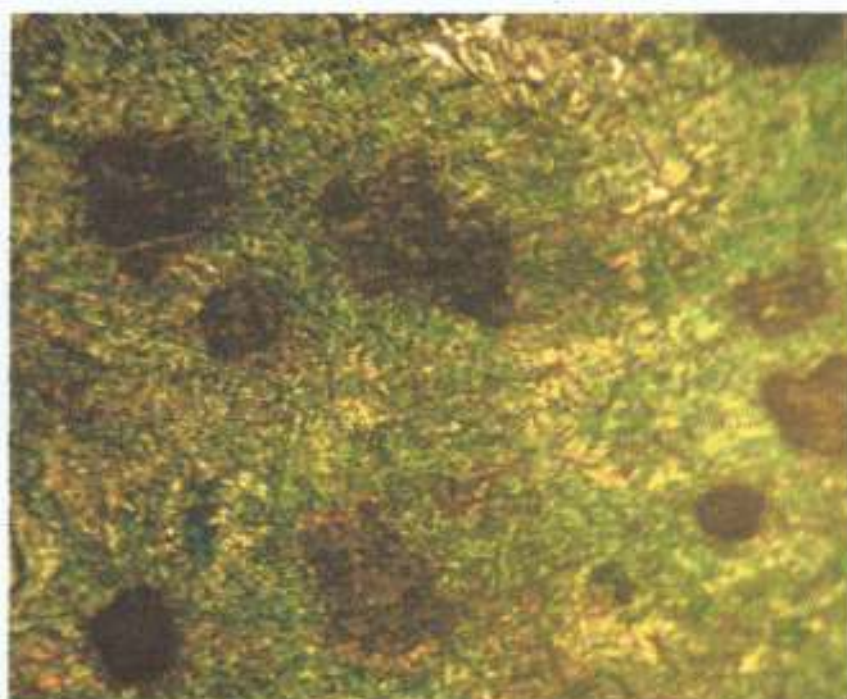


Plate 3: The same structure as in Plate 1 but at a higher magnification (etched with 2% nital and at 400X)

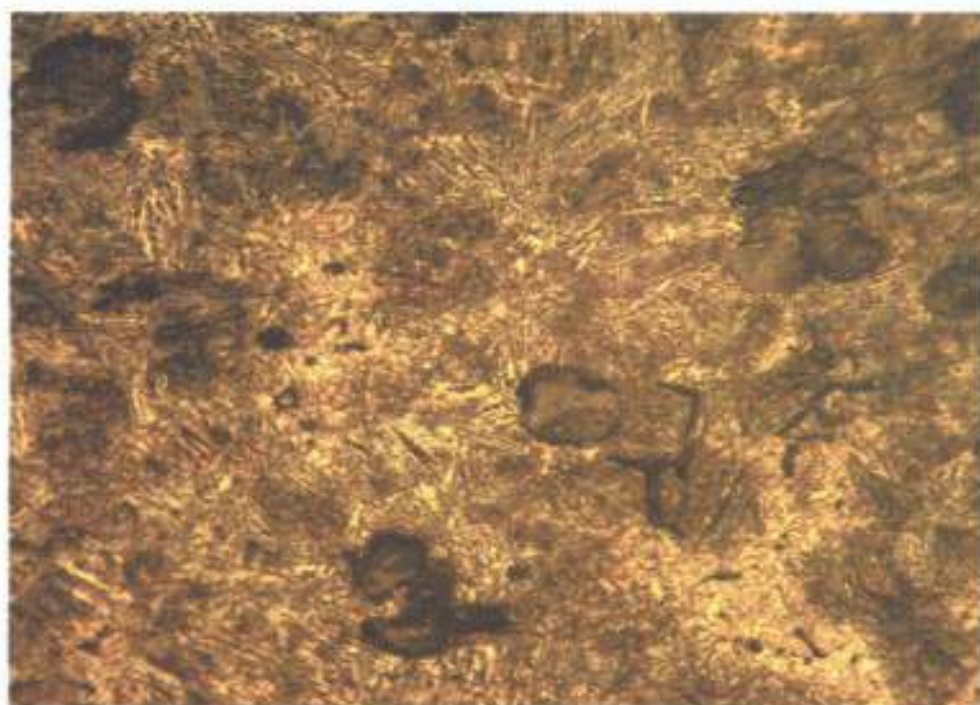


Plate 4: Ductile Iron held at 225°C for 30minutes clearly showing coarse untempered martensite plates (etched with 2% nital and at 200X)

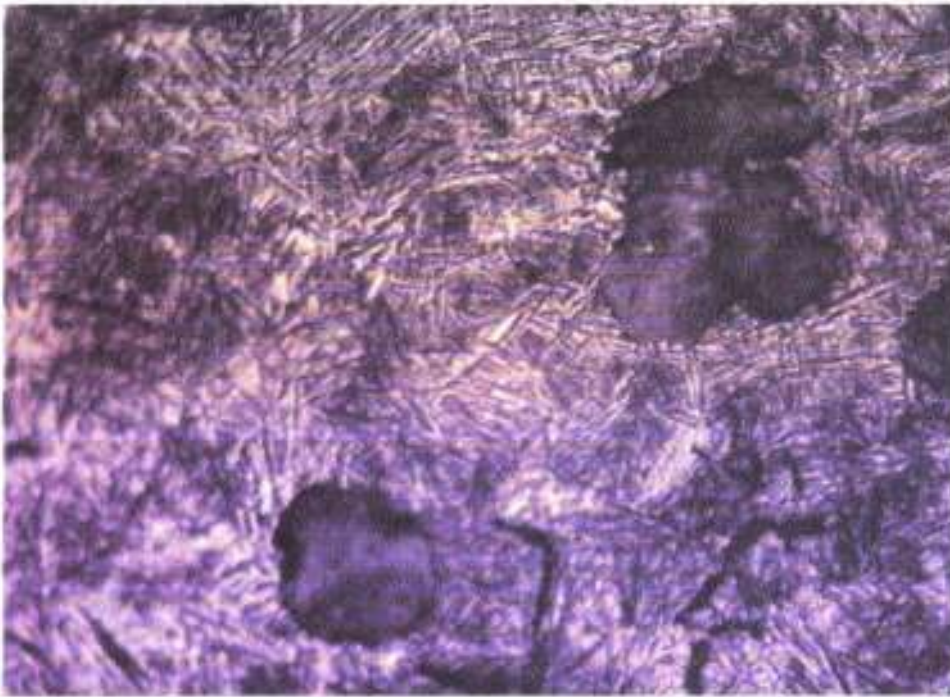


Plate 5: The same as Plate 3 but at a higher magnification (etched with 2% nital and 400X)

4.4.1.1 TYPE-B Structures

This class is the typical tempered martensite structure, as shown in:

- i) Plates 6 and 7 for holding at 250°C for 90 minutes.
- ii) Plates 8 and 9 for holding at 350°C for 60 minutes.
- iii) Plates 10 to 12 for holding at 375°C for 60 minutes.

Plates 6 and 7, containing microstructures for ductile cast irons held at 250°C, clearly show structures which are typical of fine tempered martensite plates classified as Type B. The same structure is observed in Plates 8 to 12 for higher tempering temperatures of 350°C and 375°C.

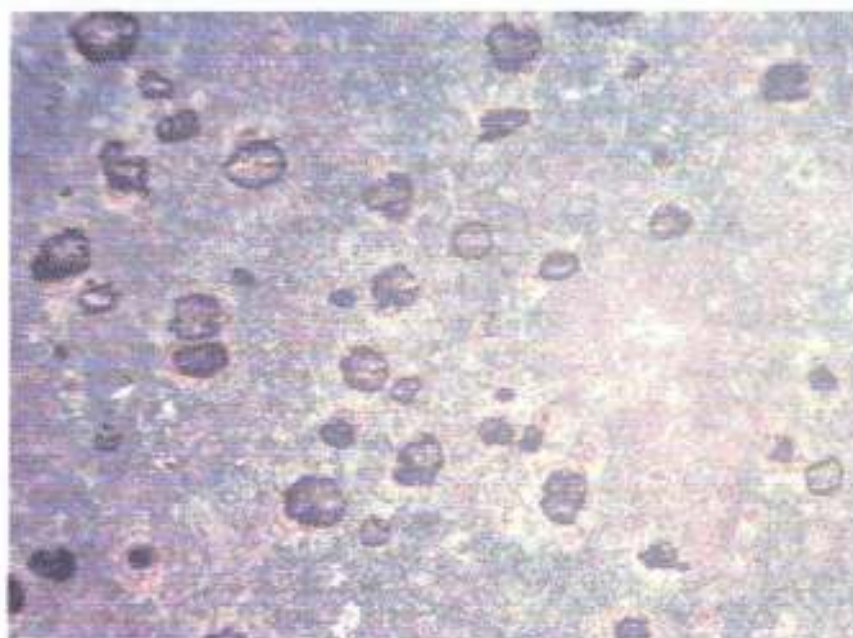


Plate 6: Ductile Iron held at 250°C for 90minutes showing tempered martensite structure (etched with 2% nital and at 100X)

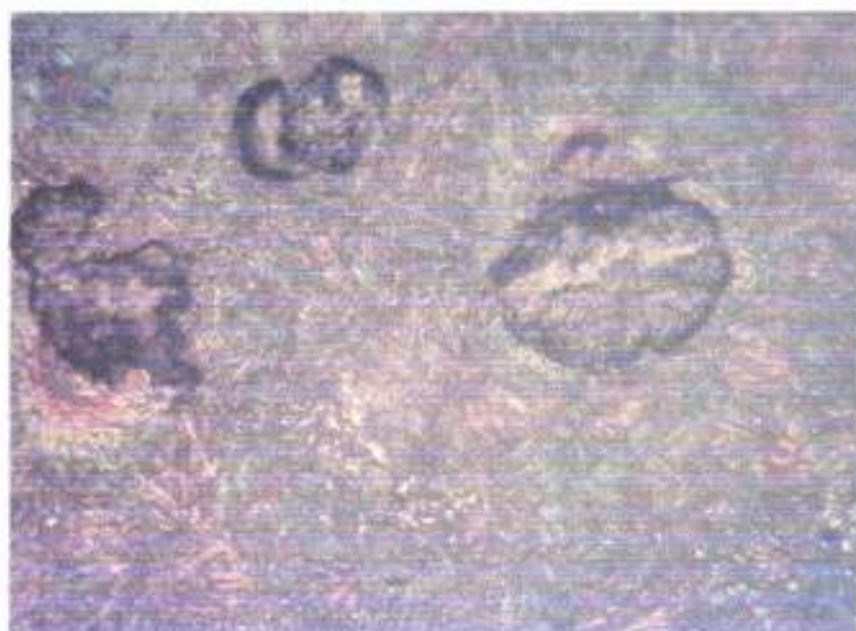


Plate 7: Same as Plate 6, at a higher magnification, showing the martensite plates (etched with 2% nital and at 400X)

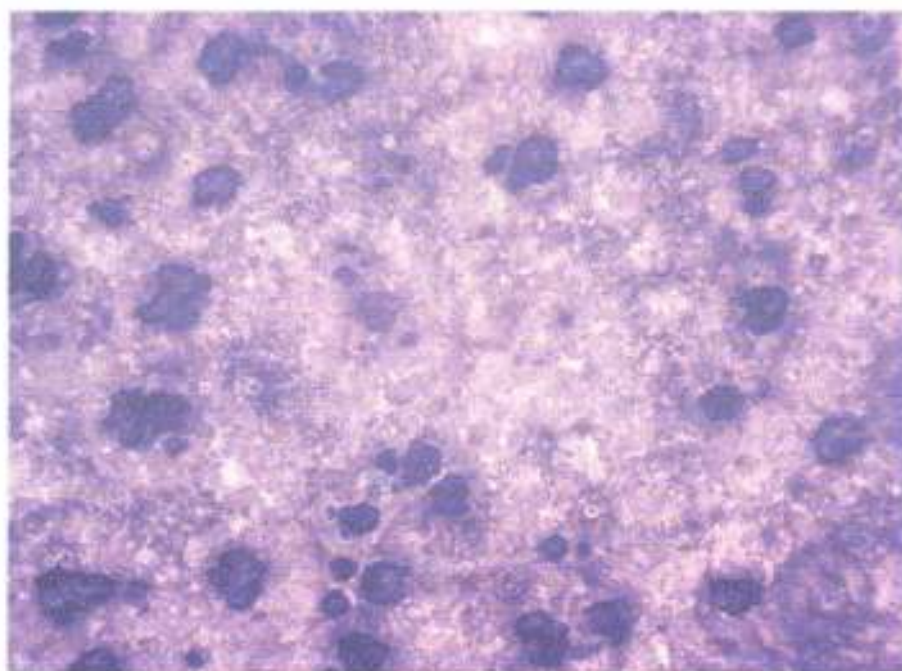


Plate 8: Ductile Iron held at 350°C for 60minutes showing a tempered martensite structure (etched with 2% nital and at 100X)

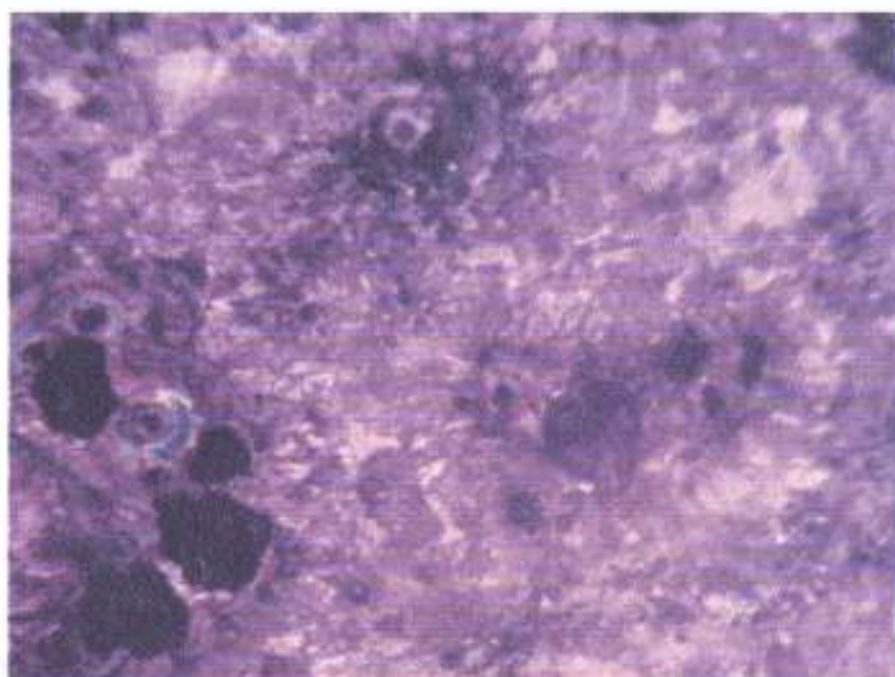


Plate 9: The same as Plate 8 but at higher magnification causing the tempered martensite plates to be resolved (300X)

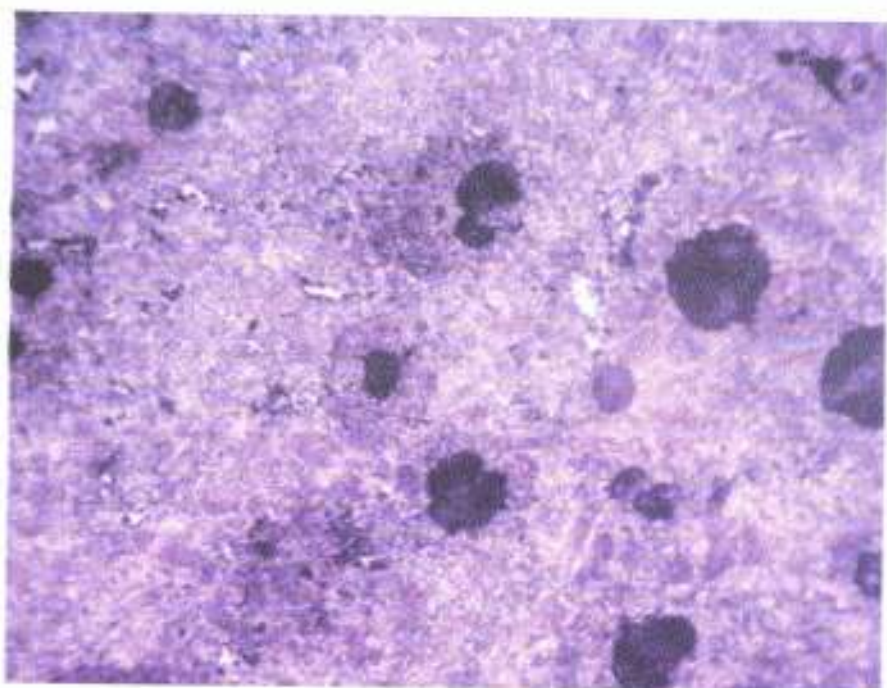


Plate 10: Ductile Iron held at 375°C for 60minutes still showing tempered martensite structure (etched with 2% nital and at 200X)

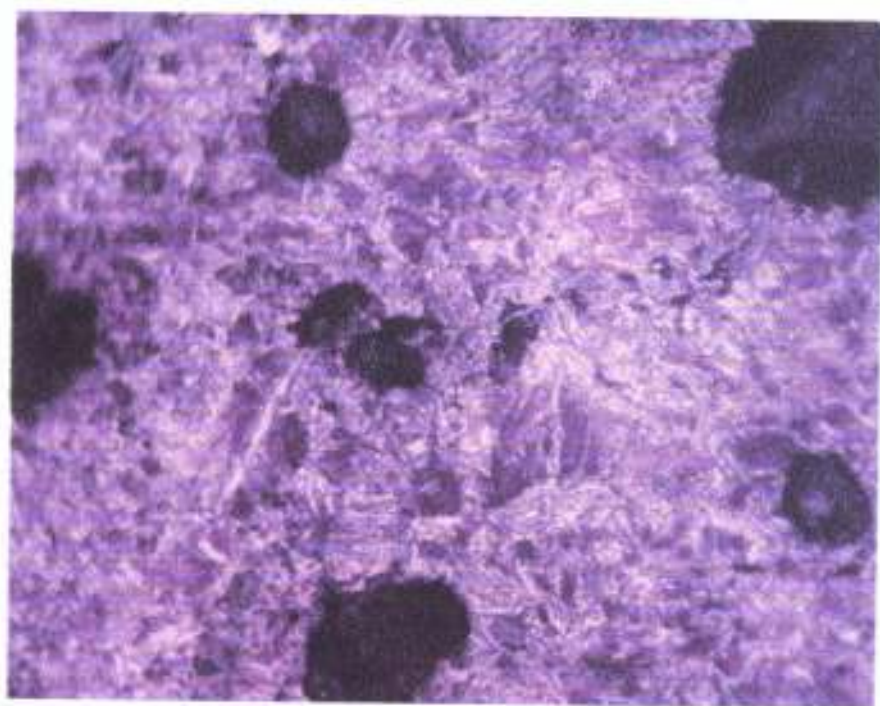


Plate 11: Same tempered martensite as in Plate 10 (400X)

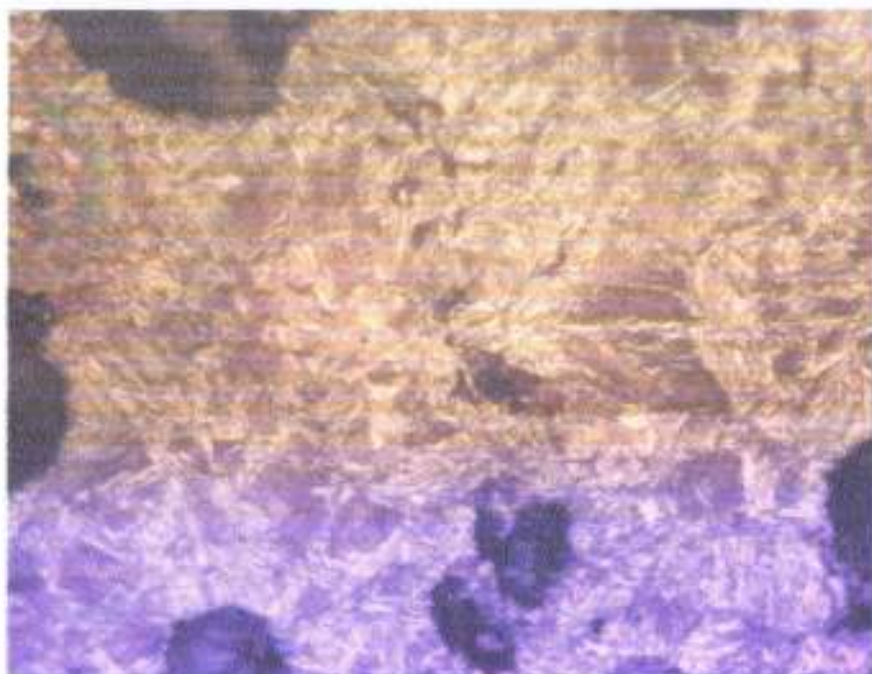


Plate 12: Same tempered martensite as in Plates 10 and 11, but at a higher magnification clearly showing the martensite plates (800X)

4.4.2 TYPE-C STRUCTURE

This consists of over tempered martensite structure consisting of non-martensitic matrix structure with or without ferrite shells surrounding the graphite nodules. According to Table 4.2, these were expected to be formed at high temperatures above 400°C. Plates 13 and 14 show matrix structures without any ferrite shell, which occur short holding periods of less 60 minutes at 425°C holding temperatures.



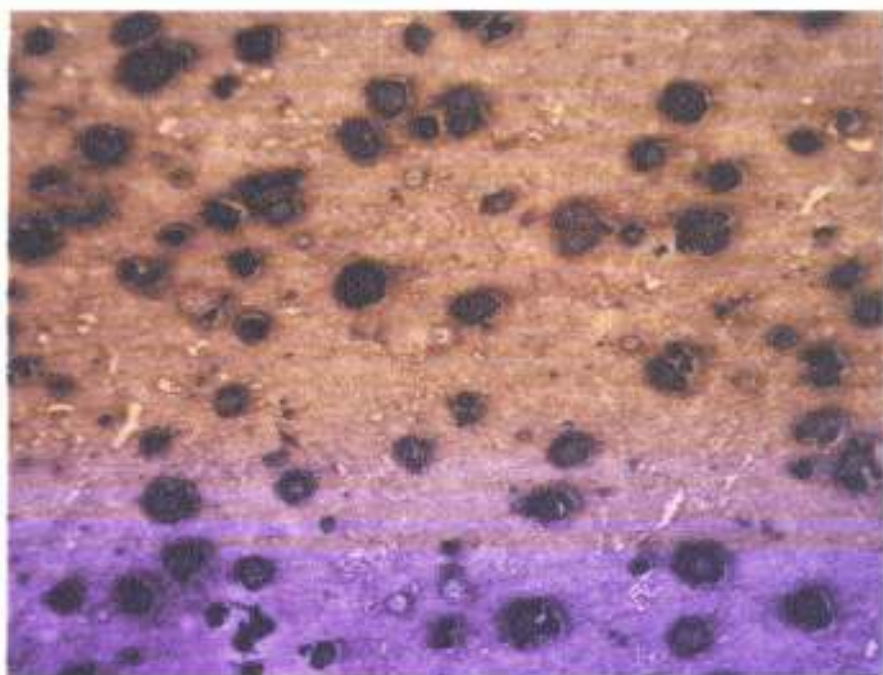


Plate 13: Ductile Iron held at 425°C for 60minutes showing over tempered martensitic structure and some free cementite (white), (etched with 2% nital and at 100X).

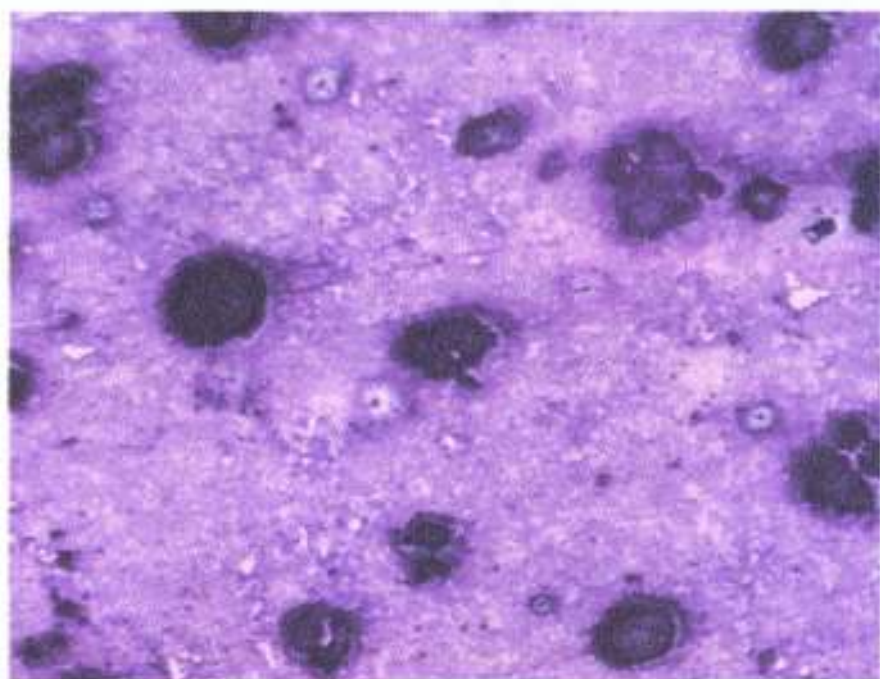


Plate 14: Same as Plate 13, but at a higher magnification, where martensite plates are not observed (200X).

However, prolonged heating for 120 minutes at temperatures, as low as 350°C in Plate 15, has been found to cause formation of thin ferrite shells around the graphite nodules. However Plate 16 has shown that tempering at both high temperatures and for prolonged periods, such as 120 minutes at 425°C, had resulted in formation of a spheroidized structure and extensive ferrite shells around the graphite nodules. The ferrite appears as small seedlings of ferrite or as the bull-eye structure.

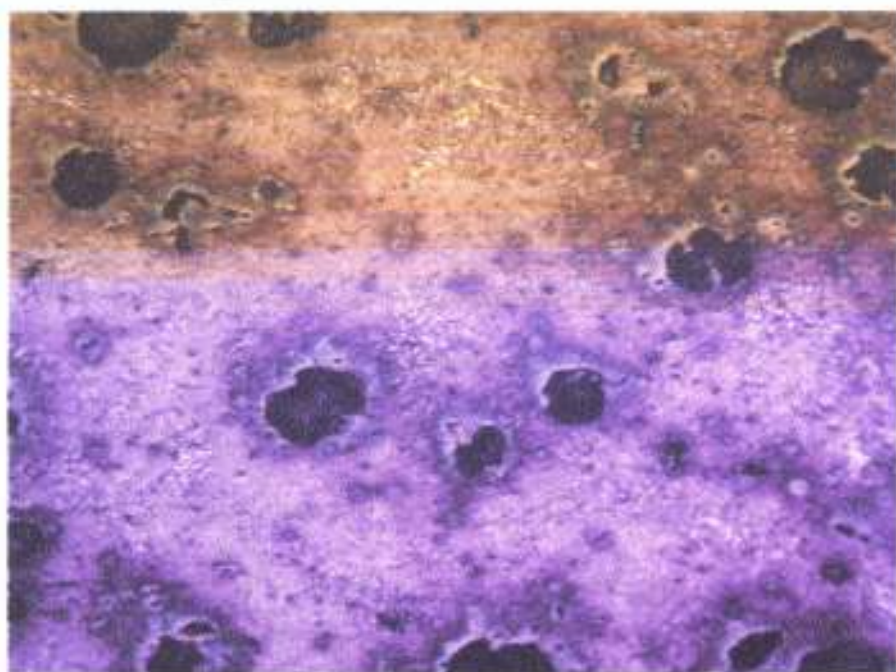


Plate 15: Ductile Iron held at 350°C for 120 minutes. Note that prolonged holding at this temperature has resulted in formation of a spheroidized matrix structure and thin free ferrite shell around the graphite nodules (etched with 2% natal, 200X)



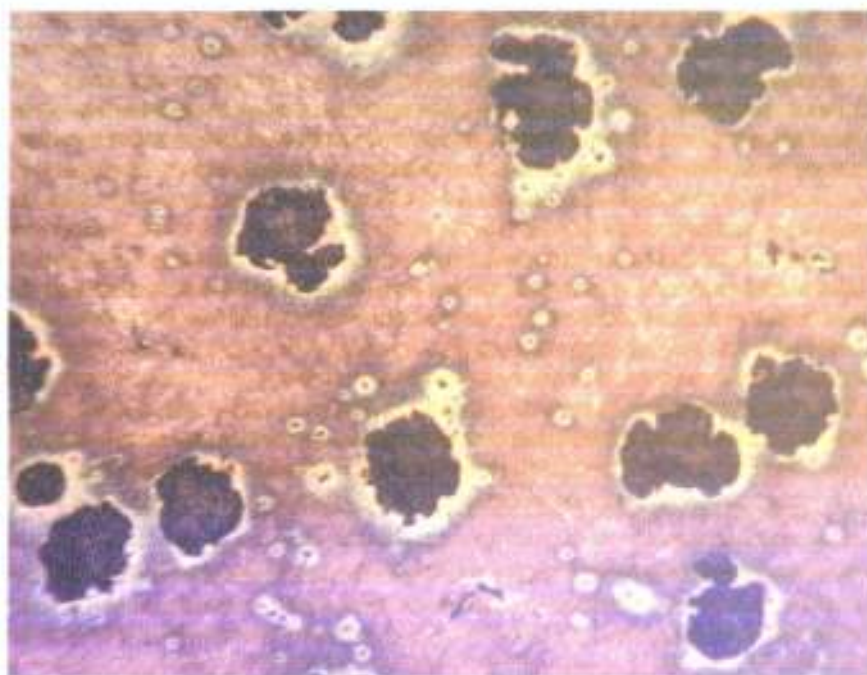


Plate 16: Ductile Iron tempered at 425°C for 120 minutes further showing spheroidized matrix structure and extensive ferrite shells (white) around the graphite nodules. (Etched with 2% nital and at 200X)

Finally, the structures obtained from the practical microstructural investigation are in total agreement with those predicted by the hardness study. It has also confirmed that the above heat treatment process circle is realistic and reliable for the production of structures which have mechanical properties that are comparable to those of austempered ductile irons.

Also, the structures obtained from the microstructural investigation are in total agreement with those predicted by the hardness study. It has also confirmed that the above heat treatment process circle is realistic and reliable for the production of structures which have mechanical properties that are comparable to those of austempered ductile irons.



4.5.0 DEVELOPMENT OF AN APPROPRIATE COMPUTER MODEL

4.5.1 DEVELOPMENT OF MODEL EQUATION

A Computer Model equation was derived from the experimental data generated using Statistical Package for Social Sciences(SPSS) and from equation

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \dots + \beta_n X_n + \varepsilon \dots\dots\dots$$

where $\beta_0, \beta_1, \beta_2, \dots, \beta_n$ are constants (coefficients),
and $X_0, X_1, X_2, \dots, X_n$ are variables.

From the application of the package, standardized coefficients and constant were determined. The appropriate model equation was found to be:

$$Y = 62.504 - 0.079T(^{\circ}C) - 0.054t(\text{minutes});$$

where Y = Hardness value (Rc),
 X_1 = Temperature ($^{\circ}C$),
 X_2 = time (minutes).

The model equation was adopted to generate a new set of theoretical data contained in Appendix B.

4.5.2 Model Validation

Model validation was carried out by applying three different validation tools:

- 1. Pair-t Test,
- 2. Correlation Coefficient Test
- 3. Standard Error of Prediction Test

4.5.2.1 Pair-t Test Analysis

A Pair-t test was carried out by initially calculating the parir difference which finally leads to the determination of the confidence intervals and acceptable interval. It is seen that the:

- i) confidence interval was found to be 94.5%, which shows a very high correlation
- ii) acceptance interval was found to be between 1.05 and 2.14, which is withing the acceptable limit.

4.5.2.2 Correlation Coefficient And Standard Error Tests

The results for the Correlation Coefficient and Standard Error tests are contained in Appendix - C from where the:

- i) correlation coefficient ranged from 0.9595 to 0.9925, which has been found to be in the very high correlation range of 0.9 to 1.0 (Aderoba 1995).
- ii) standard error test was found to be between 2.16 and 2.82, which is very low and which signifies very correlation.

Table 4.2: Correlation Coefficient and Standard Error Analysis Tests for the Model

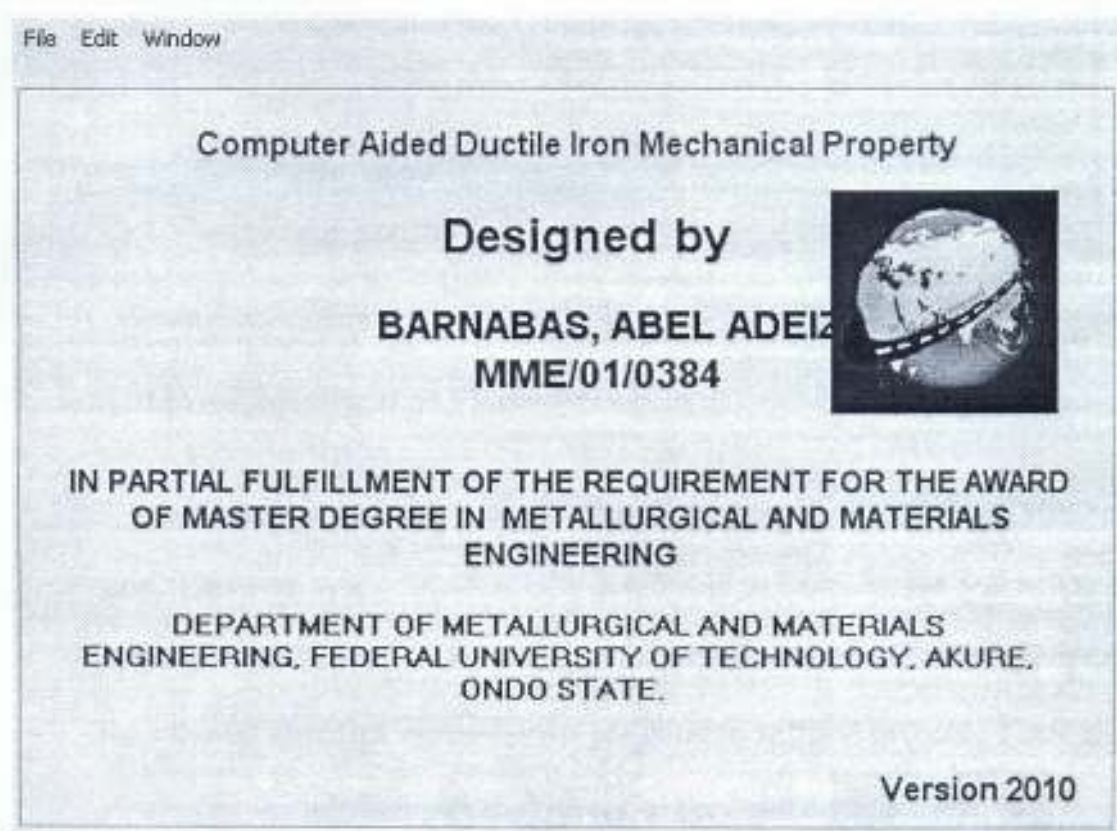
Holding Periods (Minutes)	Correlation coefficient of the Model	Standard error of the Model
30	0.987809	2.162
60	0.9925	2.193
90	0.9595	2.501
120	0.98296	2.823
150	0.98296	2.823

4.6 COMPUTER AID SOFTWARE DEVELOPMENT

The computer software developed for predicting hardness values of heat treated ductile irons was designed according to the following phases:

1. Phase 1 : Title Phase Menu

This phase welcomes the user to the system as shown Phase 1 Menu. It contains information about the name of the software, the developer, the supervisor, the essence of the study and the department in which the work was carried.

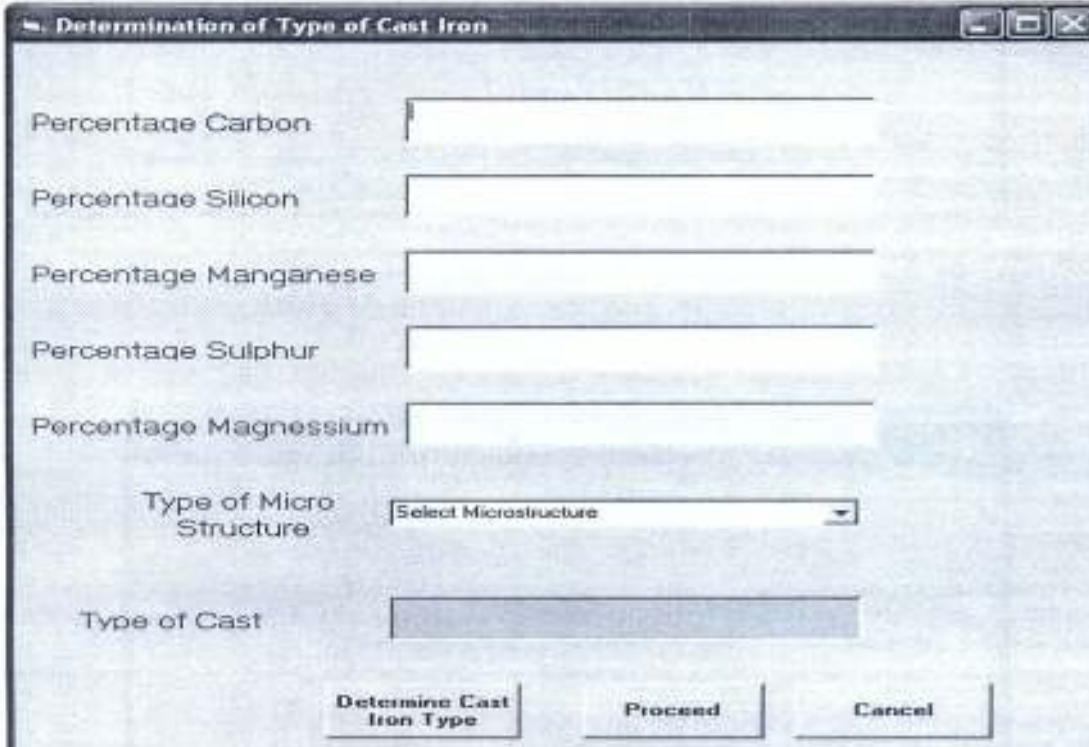


Phase 1: Title Phase Menu for the Developed Software



2. Phase 2: Data Entry Menu

In this phase, data necessary for computation or determination of an output is entered. Such data will include material specification and chemical composition as in menu.



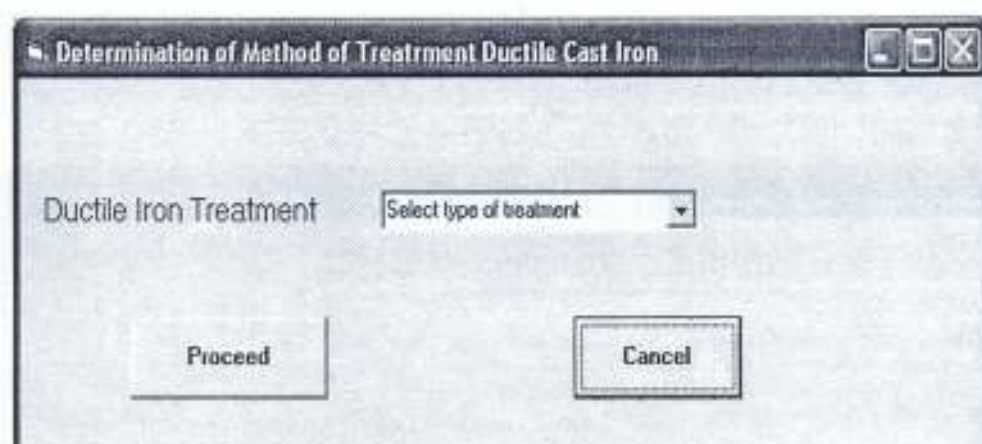
The screenshot shows a software window titled "Determination of Type of Cast Iron". It contains several input fields for chemical composition: "Percentage Carbon", "Percentage Silicon", "Percentage Manganese", "Percentage Sulphur", and "Percentage Magnesium". Below these is a dropdown menu for "Type of Micro Structure" with the text "Select Microstructure". At the bottom left is a text field for "Type of Cast". At the bottom right are three buttons: "Determine Cast Iron Type", "Proceed", and "Cancel".

Percentage Carbon	
Percentage Silicon	
Percentage Manganese	
Percentage Sulphur	
Percentage Magnesium	
Type of Micro Structure	Select Microstructure
Type of Cast	
Determine Cast Iron Type Proceed Cancel	

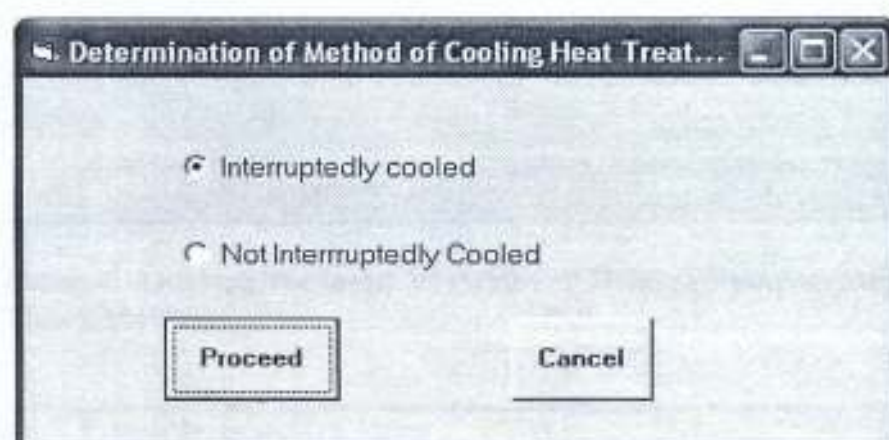
Phase 2: Material Identification Phase

This also includes material identification such as gray cast iron, or steel. However this software is only applicable to ductile irons and will run only for these. In addition the chemical composition input must match that for the material specified otherwise the programme will not proceed. There are some other intermediate phases to be run before reaching this menu.

3. Phase 3: Heat Treatment Menu



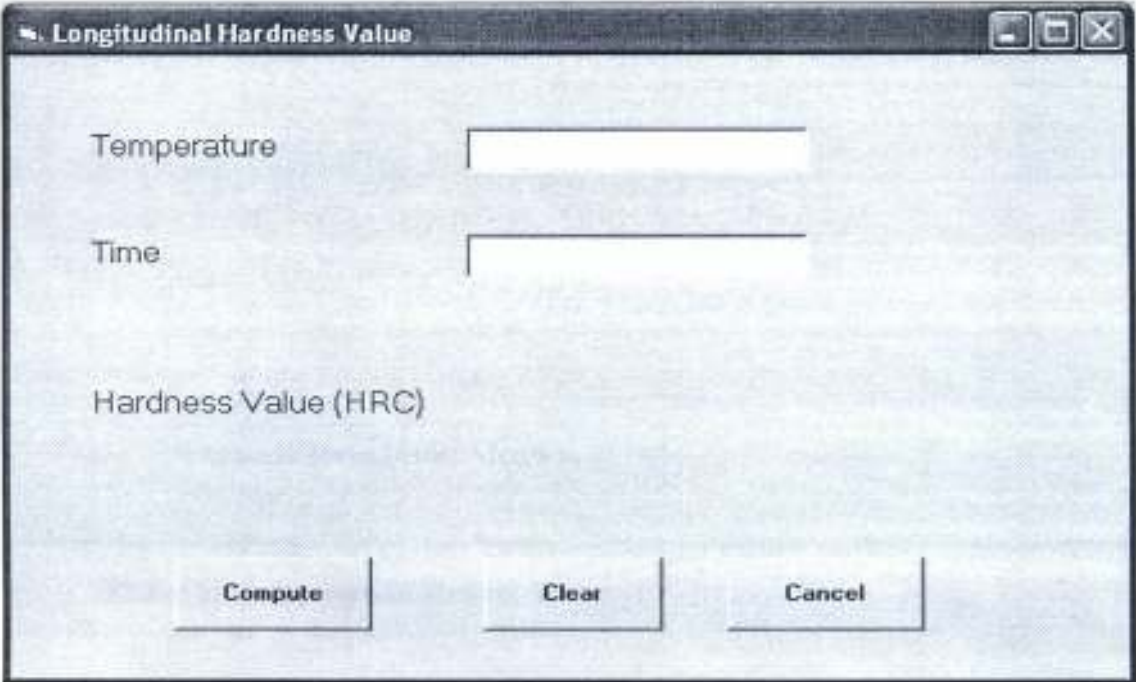
Phase 3(a): Heat treatment Menu – I



Phase 3(b): Heat treatment Menu – II

After this, the property to be evaluated is selected by entering the variable inputs for the determination of the hardness values of ductile iron of known composition after being subjected to a given heat treatment.

4. Phase 4: Variable Data Input Menu



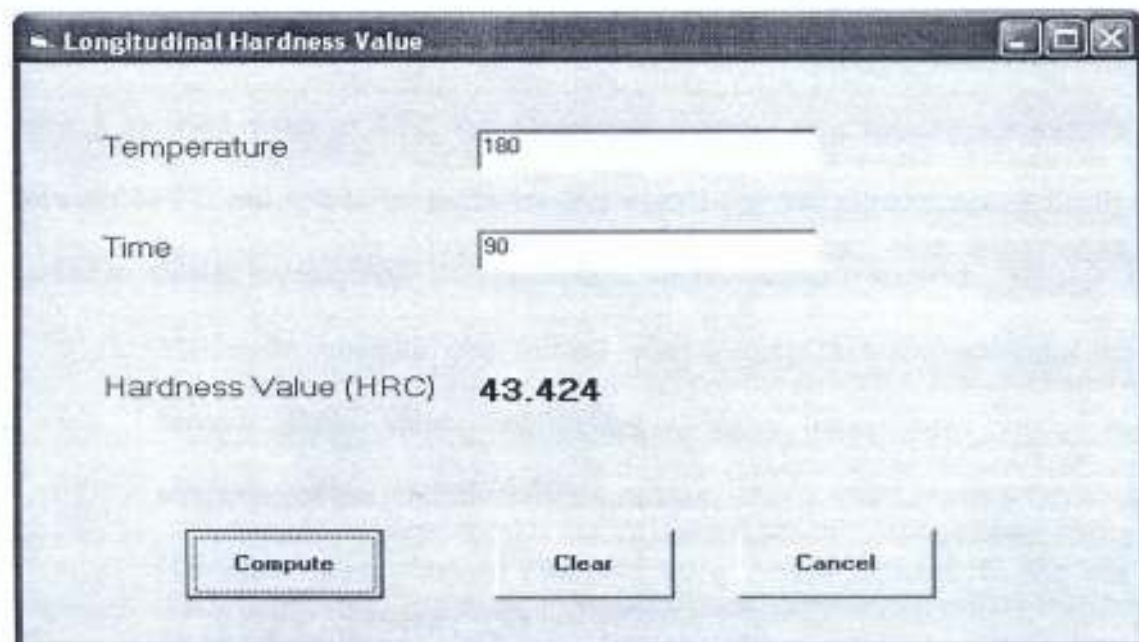
The screenshot shows a software window titled "Longitudinal Hardness Value". Inside the window, there are three input fields: "Temperature", "Time", and "Hardness Value (HRC)". Each field is represented by a text label followed by a rectangular input box. At the bottom of the window, there are three buttons: "Compute", "Clear", and "Cancel". The window has a standard Windows-style title bar with minimize, maximize, and close buttons.

Phase 4: Entering the input Variables of Holding Temperature ($^{\circ}\text{C}$) and Time (minutes)

5. Phase 5: Property Selection Menu

These concerns the output property desired to be computed using the model, which is hardness value in this exercise. Once selected, the output or report phase is reached.

6. Phase 6: Computation/Report Menu.



Longitudinal Hardness Value

Temperature

Time

Hardness Value (HRC) **43.424**

Phase 6: Computation/Report Menu.

This menu gives the final answer, which is the required hardness value for a ductile iron subjected to a holding temperature of 180°C for 90 minutes after being quenched in warm water for 40 seconds. In this computation the hardness value is 43.424HRC, and the structure will be martensite.

4.4.7 Computer Requirements

The computer hardware used for the execution of the software had the following specifications:

1. Type: Personal Computer
2. Pentium 2 and above or Laptop with at least 64 MB RAM,
3. Microsoft Windows 98/NT/2000/XP and
4. 64 MB and 512 MB hard disk free space.

It can also run on computer systems of higher grade like Pentium-3 and Pentium-4.

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion



The heat treatment of ductile cast irons by austenization at 850°C , interrupted cooling in warm water at 80°C for 40 seconds, heating to a holding temperature between 175°C and 450°C for times varying from 30 to 180 minutes before finally cooled in air was investigated. The results from the investigation showed:

1. Martensite structure was formed after cooling in warm water for 40 seconds. Hence subsequent holding at higher temperatures resulted in tempering of the prior martensite structure.
2. Hardness values obtained after tempering ranged between 52.9Rc and 19.6Rc depending on the holding temperature and time.
3. The following matrix structures were obtained after holding at:
 - i) $175^{\circ}\text{C} - 225^{\circ}\text{C}$; virtually untempered martensitic structure, possibly with retained austenite.
 - ii) $200^{\circ}\text{C} - 250^{\circ}\text{C}$; untempered martensite at holding times of less than 60 minutes but tempered martensite at longer holding times.
 - iii) $250^{\circ}\text{C} - 325^{\circ}\text{C}$; tempered martensite form at holding periods less than 120 minutes, but over tempered martensite with low hardness values was obtained at longer holding periods.
 - vii) $325^{\circ}\text{C} - 375^{\circ}\text{C}$; tempered martensite structure for less than 60 minutes holding times but over tempering occurred at prolonged holding.

- viii) Above 375°C; over tempering of martensite in the matrix occurred leading to spheroidized matrix structure. However, extensive ferrite shells formed around the graphite nodules for prolonged heating or holding at temperature above 400°C.
4. The above heat treatment process has been found to be:
- i) a veritable and successful method for producing tempered structures in ductile irons, which have mechanical properties that are characteristic of the austempered ductile iron.
 - ii) cheap, convenient and save to operate when compared with the use of traditional salt baths.
5. A computer model was developed to predict hardness values of ductile irons subjected to heat treatment by this process. This resulted in:
- i) establishment of a model equation for estimating hardness values;
$$Y = 62.504 - 0.079T (^{\circ}\text{C}) \cdot 0.054t (\text{minutes}),$$

There is a good degree of correlation between the experimental and generated values .
 - ii) development of a computer software for theoretical evaluation of hardness values.



5.2 Recommendation for Further Work

This work has been based on the use of warm water at 80°C for the production of tempered martensite structure. Other works can be carried out using other quenching media. In addition, a computer model and software have been developed for the determination of hardness values of ductile irons subjected to the above heat treatment, further work should be carried out to extend this to cover the determination of other mechanical properties such as tensile strength and toughness.



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APPENDIX - A: Hardness values of ductile cast irons subjected to interrupted quenching in warm water prior to holding at various elevated temperatures and times.

TRANSFORMATION TEMPERATURES (°C)	HARDNESS VALUES (Rc) FOR VARIOUS HOLDING TIMES (Minutes) TRANSFORMATION TEMPERATURES					
	30	60	90	120	150	180
175	52.9	51	48.8	47.2	45.5	42.9
200	46.0	42.9	39.8	40.7	37.3	33.6
225	43.8	40.1	39.0	36.7	34.5	32.9
250	42.2	38.7	35.9	33.6	30.7	28.9
275	35.9	35.4	33.0	32.1	29.8	27.3
300	38.7	36.5	31.7	30.8	28.6	26.9
325	32.7	31.0	30.1	29.9	28.0	27.2
350	33.6	29.7	28.6	27.4	26.7	26.2
375	31.8	31.0	29.7	28.2	27.6	27.7
400	37.8	27.2	26.1	26.0	25.9	25.2
425	26.6	23.6	23.1	21.2	20.5	19.6

APPENDIX - B: Generated Values

TIME TEMP	HARDNESS VALUES AT DIFFERENT HOLDING TIMES(Min.)					
	30	60	90	120	150	180
175	47.059	45.439	43.819	42.199	40.579	38.959
200	45.084	43.464	41.844	40.224	38.604	36.984
225	43.109	41.489	39.869	38.249	36.629	35.009
250	41.134	39.514	37.894	36.274	34.654	33.034
275	39.159	37.539	35.919	34.299	32.679	31.059
300	37.184	35.564	33.944	32.324	30.704	29.084
325	35.209	33.589	31.969	30.349	28.729	27.109
350	33.234	31.614	29.994	28.374	26.754	25.134
375	31.259	29.639	28.019	26.399	24.779	23.159
400	29.284	27.664	26.044	24.424	22.804	21.184
425	27.309	25.689	24.069	22.449	20.829	19.209
450	25.334	23.714	22.094	20.474	18.854	17.234

APPENDIX - C: Pair-t Test Analysis to Determine the Acceptable Confidence Interval of the model

Time (Min.)	Pair difference	Square of pair difference	Mean of pair difference	Variance of pair difference	Mean of variance of pair difference	Standard deviation	Confidence Interval	Degree of freedom	Acceptable Interval ($\bar{d} + t_{\alpha/2, \nu}$ Sd)
	(d)	(d ²)	($\bar{d}$)	Var (d)	Var ($\bar{d}$)	(Sd)	(α)	t _v	
30	-4.341	56.089	-0.3617	4.95631	0.41304	0.64267	94.5%	2.20	1.05 - 1.78
60	3.278	57.717	0.2732	5.1656	0.4305	0.6561	94.5%	2.20	1.71 - 1.2
90	7.028	75.06648	0.5856667	6.4500	0.5375	0.73314	94.5%	2.20	2.2 - 0.9
120	2.088	67.50206	0.174	6.10352	0.5086	0.7132	94.5%	2.20	1.74 - 1.4
150	3.098	95.663	0.25816	8.6239	0.7186	0.8477	94.5%	2.20	2.14 - 1.6