EVALUATION OF HOT- BITUMEN BATH AS A QUENCHING MEDIUM FOR AUSTEMPERING OF STEEL AND DUCTILE CAST IRON

BY

AUSE TERVER

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**DEPARTMENT OF METALLURGICAL ENGINEERING
FACULTY OF ENGINEERING
AHMADU BELLO UNIVERSITY
ZARIA
NIGERIA.**

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DECLARATION

I declare that the work in the dissertation entitled: “Evaluation of hot-bitumen bath as a quenching medium for Austempering of steel and Ductile Cast Iron”, has been performed by me in the Department of Metallurgical Engineering under the supervision of. Prof. O.B. Oloche, Dr. S.B Hassan, and Dr. O. Aponbiede.

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

Ause Terver

| --- | --- | --- |
| Name of student | Signature | Date |

CERTIFICATION

This dissertation entitled "EVALUATION OF HOT-BITUMEN BATH AS A QUENCHING MEDIUM FOR AUSTEMPERING OF STEELS AND DUCTILE CAST IRON". By AUSE TERVER, meets the regulations governing the award of the degree of Doctor of Philosophy (Metallurgical Engineering) of Ahmadu Bello University, Zaria, and is approved for its contribution to knowledge and literary presentation.

_____ Date _____

Prof. O. B. Oloche
Chairman, Supervisory Committee

_____ Date _____

Dr. S.B. Hassan
Member, Supervisory Committee

_____ Date _____

Dr. O. Aponbiede
Member, Supervisory Committee

_____ Date _____

Dr. O. Aponbiede
Head of Department

_____ Date _____

Prof. S.A NKOM
Dean, Postgraduate School

DEDICATION

This work is dedicated to my senior daughters miss DOOFAN and LINDA TERVER AUSE.

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It is the wish of Jesus Christ (Mark 12:17) that we should give to Caesar what is Caesar's and give to God what is God's. In that respect, and coupled with the fact that education itself starts right at home and continues immediately thereafter, I would first like to express my sincere appreciation to two of my senior brothers, Mr. Michael Anande Ityav and Sabastine Shior Tegh (DSP) for their combined efforts/assistance in my education right from my youth.

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ABSTRACT

In this research work, the possibility of using hot bitumen as quenching medium for austempering of steel and ductile cast iron was investigated. Standard izod Impact, hardness and tensile samples were machined from 0.55%C-steel, 0.75%C-steel and ductile cast iron bars. The samples were austenitized at 800,850, 900 and 950^{o}C, quenched in hot bitumen at 350^{o}C and austempered for 5minutes, 30minutes, 1, 2, 3, 4 and 5hours respectively. The samples were tested for hardness, impact and tensile strength, microstructures were also examined. The result of the microstructures obtained showed that hot bitumen was able to cause formation of bainite structure in the samples. Austempering in hot bitumen resulted to increase in hardness. The as-received hardness value of 0.55%C-steel increased from 200 to 430HV after austempering, while 0.75%C-steel with as-received hardness value of 260HV increased to 548HV and the as cast hardness value of ductile cast iron increased from 148 to 410HV. Austempering of ductile cast iron in hot bitumen resulted to improved impact strength which increased from 17J (as-cast) to 29J (austempered). Austenitization temperature was found to have influence on the impact strength of ductile cast iron.

The highest impact strength of 29J was obtained at 850^{o}C while the lowest impact energy was 4J at 950^{o}C. At all the austempering times, the impact energy of 0.55%C-steel (21, 22, 37, 33, 29J at 1, 2, 3, 4, 5hrs respectively) was found to be higher than that of 0.75%C-steel (18, 17, 17, 16, 24J at 1, 2, 3, 4, 5hrs respectively). Results of tensile test showed that austempering in hot bitumen resulted to increase in tensile strength, percent elongation and percent reduction in area of ductile cast iron. The investigation carried out showed that hot bitumen can be used as quenching medium in austempering process.

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LIST OF ABBREVIATIONS/SYMBOLS

ADI	-	Autempered Ductile iron
γ	-	Austenite phase
α	-	Ferrite phase
Fe_3C	-	Cementitle pahse
γ_{hc}	-	Carbon Enriced austenite

CHAPTER ONE

1.0 INTRODUCTION

Heat treatment originated as an ancient art in man's attempts to improve the performance of metals in their practical applications. Khanna (2002) pointed out that, in the middle of the 19th century, more and more attention was directed to the art and science of producing, working and treating of metals to suit various areas of applications. In the present day metallurgical practice, heat treatment has come to represent a class of engineering techniques involving thermal, mechanical and chemical treatment of metals/alloys by which the desired microstructures and properties are obtained to suit variety of applications (Khanna. 2002).

The mechanical properties of metals generally are related and dependent on their compositional and microstructural make-up (metallurgical properties). A change in microstructure is in practice usually achieved through phase transformation by heat treatment process (Rajan et al, 1988). Therefore, the fundamental objective of heat treatment is to produce a change in microstructure, which eventually results to change in mechanical properties. For steels and cast irons, heat treatment is applied to cause the transformation of austenite to lower temperature products which results to different structure depending on the

composition of the alloy and the rate/type of transformation (whether diffusional or non-diffusional) (Rajan et al, 1988).

Heat treatment is known to be one of the industrial finishing operations just like electroplating, machining, etc, which is usually applied to metal components before they are finally put into service. Rajan et al (1988) defines heat treatment as "heating and cooling operation(s) applied to metals and alloys in solid state so as to obtain the desired microstructures and properties". Among all engineering metals/alloys, steels and cast irons are more versatile. This versatility is based on the fact that their properties can be controlled and changed at will by heat treatment.

Formation of austenite is a preliminary step for any heat treatment process for ferrous alloys. Depending on parameters such as composition of the alloy, cooling rate, section size, etc; austenite may transform on cooling to lower temperature phases such as ferrite, pearlite or martensite. The presence of any of these structures in the alloy determines the properties. Heat treatment could be classified into normalizing, annealing, martempering, flame hardening and austempering (Avner, 1984; Khanna, 2002). These heat treatment processes are used to achieve the followings: refine the grain size, soften the metal, increase the hardness, remove residual stresses, improve machinability, toughness, and ductility, homogenize the structure to remove segregation. Hardening by

quenching is usually done by heating the ferrous component (Steel/cast iron) to austenite phase, soaking for sometime to effect homogenization. Then removing and fast cooling (quenching) in a liquid medium (quenchant). Generally, quenchants are grouped into two categories:

i. Room temperature quenchants

ii. High temperature (150-450^{0}C) quenchants

The room temperature quenchants are the most popular, common and conventional: examples include water, brine, oil (mineral or vegetable), caustic soda solution, solutions of water soluble polymers, etc). Those quenchants maintained at higher temperatures are used for interrupted quenching heat treatment process such as martempering, austempering and ausforming. Examples of this type of quenching media are molten salt bath, molten lead bath and hot oil bath (Imasogie et al, 1991); (Adewuyi and Afonja, 2000).

Sometimes, quenching media such as water, brine and oil may be too drastic. Components quenched in such media would have inferior mechanical properties and defects (cracking and distortion). Such components with defects are eventually rejected and scrapped. Therefore, in order to impart good mechanical properties (especially strength and toughness/ductility) and simultaneously avoid cracking and distortion, many researchers: Donald (1985), Adewuyi and Afonja (2000), Mallia and Grech (1997), Avner (1984) and

Khanna (2002) suggested that other heat treatment methods like martempering and austempering in molten salt bath or hot oil bath could be used. Unfortunately, salt bath furnace is very expensive and scarce. Therefore, there is the need to seek for an alternative.

The major aim of this research work is to use steel and ductile cast iron to evaluate the effectiveness of hot bitumen bath as a medium for austempering, The emphasis was to establish whether or not the use of hot bitumen bath as austempering medium would result to formation of bainite structure and subsequent improvement in hardness and izod impact strength of the steels and ductile cast iron samples investigated.

1.1 PROBLEM STATEMENT AND RESEARCH QUESTIONS

Austempering is known to be one of the quench hardening methods which is applied to ferrous alloys in order to improve toughness and ductility. It is usually carried out in a molten bath. The three molten baths used for austempering are:

salt bath, lead bath and hot oil bath (Mallia and Grech, 1997; Imasogie et al, 1991). Salt bath furnace is the commonest one used for austempering. According to Yazdani and Elliott (1999), Cassel TS220 (K_2NO_3 + $NaNO_3$) is used as the Molten salt for austempering. The salt bath furnace and the salts are usually

imported. Bitumen was identified to be a material that may be suitable as a medium for austempering. The fundamental questions that arose were:

i. To what temperature range can bitumen be heated to cause it boil?

ii. Could this boiling temperature be high enough to maintain the austempering range of 250-450°C?

iii. Could the boiling temperature of bitumen be used to produce bainite (ausferrite) microstructure in steels and ductile cast iron as does the molten salt bath?

iv. Since bitumen stains, how can the component be cleaned? and

v. Will the cleaning method be economical and environmentally friendly?

1.2 AIMS AND OBJECTIVES OF THE STUDY

The most common austempering equipment (salt bath furnace) is costly thus the main aim of this research work is to use steel and ductile cast iron to evaluate the effectiveness of hot bitumen bath as an alternative medium to molten salt bath. Specific objectives targeted to be achieved in this research include:

i. Determine and establish the boiling point of bitumen and its suitability for the austempering range (250-450°C).

ii. Determine the possibility of austempering steel and ductile cast iron in hot bitumen bath.
iii. Evaluate the mechanical properties of austempered samples
iv. Compare obtained results with those from molten salt bath
v. Draw conclusions and recommendations based on the analysis and comparism of the results.

1.3 JUSTIFICATION/SIGNIFICANCE OF THE STUDY

Bitumen is a residue from fractional distillation of crude oil. Nigeria is one of the largest producers of crude oil in the world. Bitumen also occurs naturally in Ondo and Ogun State in large quantity in mixture with sand. According to Adegoke (1980), estimate of recoverable bitumen from the sands vary from 35-50 billion barrels per year. Therefore concerted effort should be made towards exploitation and use of this abundant natural resources (bitumen). Numerous studies; Yadzani and Elliott (1999), Kazerooni et al (1997), Imagsogie et al (2000) were carried out on austempering of steels and ductile cast iron using Cassel TS220 molten salt (K_2NO_3 and $NaNO_3$). At present, to the best of my knowledge, the use of hot bitumen bath as a medium for austempering has not been investigated. Therefore, the present research is aimed at using hot bitumen as an alternative to molten salt bath for austempering of Steel and ductile Iron.

This will go a long way in assisting many local heat treatment industries in the area of austempering and conserve the limited foreign exchange due to the importation of salt bath furnace and associated consumables.

1.4 SCOPE/COVERAGE OF THE STUDY

Three ferrous alloys: Medium carbon steel (0.55%C), high carbon steel (0.75%C) and unalloyed ductile cast irons were used to evaluate the suitability of hot bitumen bath as austempering medium. After austempering, the major parameters evaluated were:

i. Microstructure using optical metallurgical microscope

ii. Hardness using Vickers hardness tester

iii. Izod impact toughness using Avery Denison impact testing machine.

1.5 LIMITATIONS/SHORT COMINGS OF THE STUDY

There was no suitable furnace (salt bath equipment) into which bitumen could be poured, heated and the temperature controlled and fixed within the austempering range. Therefore, in alternative, a four corner kerosene stove was used for heating the bitumen to its boiling temperature. A laboratory thermometer (380^{O}C max.) was used to measure and ascertain the boiling temperature of bitumen which was used as the austempering temperature.

Optical metallurgical microscope was used to obtain the microstructures. Emphasis was laid on analysis and comparison of microstructure formed using hot bitumen bath as against molten salt bath. This was based on the already established fact that microstructure is one of the factors that determine the subsequent mechanical properties of metals/alloys.

1.6 ASSUMPTIONS UNDERLYING THE STUDY

The source of heat used was from a four-corner kerosene stove. The shortcoming was that, the temperature of hot bitumen bath could not be regulated and controlled at a desired level. The next available alternative was to heat bitumen to the boiling point. It is a known fact that the temperature at the boiling point of any liquid remains constant irrespective of how long the liquid is heated thereafter, or how much more heat is supplied. This boiling temperature range of 350±5°C was considered and used as the austempering temperature in this research work.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1.0 Steel and cast irons

Based on the iron- carbon phase diagram, ferrous metals are classified as steel or cast irons. Steels in general are alloys of iron and carbon that contain 2% carbon, less than 1% manganese, controlled amounts of phosphorous and sulphur (Allen, 1969). Of all the metals steels are by far the most widely used materials for engineering constructions. They are available as castings, forgings rolled products, and other engineering fabricated products based on their high level of mechanical and environmental properties.

Cast iron is an iron-carbon alloy in which the percentage carbon is greater than 2.1 and silicon content between 1-3% with eutectic solidification (Walton,1971). Cast irons belongs to a family of ferrous metals which are intended to be subjected to subsequent heat treatment but which cannot be mechanically worked. Unlike steels, cast irons has a substantial amount of eutectic structure. They are very important ferrous materials that give excellent casting because they have combined advantages of low cost, low melting point and high fluidity (Smith, 1981).

2.1.1 Classes of steels

Steels are classified based on the chemical composition (especially % carbon) as: plain carbon steels and alloy steels. Plain carbon steels are further sub- classified into:

(i) Dead mild steel

(ii) Mild steels

(iii) Medium carbon steels

(iv) High carbon steels (Smith, 1981; Rajan et al, 1988, and Khanna, 2002).

2.1. 2. Dead mild steels

These are steels with very low carbon content (less than 0.15%), and contain basically ferrite structures hence they are very soft and ductile. They are not hardenable by quenching but only respond mildly to strengthening by cold work. General areas of application concern areas of extreme ductility such as in wires, chains, wire meshes, sheet metals for deep drawing and press-out shapes (carbodies), nails and binding wires.

2.1.2 Mild steels

These steels (0.15- 0.30%C) are also essentially ferritic, with scattered pearlite in the ferrite matrix. They are stronger than dead mild steels, they do not also respond appreciably to hardening by quenching. They are usually used in the normalized condition after hot rolling. They are used for making rivets, wood nails, screw, bolts and nuts, carburizing steels, civil and building engineering construction steels, e.t.c.

2.1.3 Medium carbon steels

These steels (0.35-0.65%C) are hardenable steels and are known to posses, after hardening (by quenching), high strength, toughness and considerable hardness. They are commonly used in the quenched and tempered condition.

These steels are used for: Machine elements such as; gears, shafts, connecting rods, wheel spokes, spanners, e.t.c.

2.1.4 High carbon steels

These steels (>0.65% C) are also hardenable, and are used in the quenched and tempered condition. They are used for springs (coiled and laminated),

hammers, wood saws. Due to their high wear resistance, they are also used for making cutting tools such as; drills, chisels, shear blades, knives, e.t.c.

2.1.5 Alloy steels

According to Khanna (2002), steel is considered to be alloy steel when the maximum of the range given for the content of alloying elements exceeds one or more of the following limits: Mn-1.65%, Si-0.60%, Cu-0.60%. Alloying elements alter the properties of steel and put it into a slightly different class from ordinary plain carbon steels.

Alloy steel are subdivided into

(i) Low alloy steels

(ii) Medium alloy steels

(iii) High alloy steels. The high alloy steels are also subdivided into (a) ultra-high strength steels (b) tool steels (c) stainless steels (Smith, 1981). Stainless steels owe their properties to the formation of adherent chromium oxide film on the surface of the steels, and this prevent further chemical attacks. The different types of stainless steels include: Ferritic stainless steels, Austenitic stainless steels, martensitic stainless steel, and precipitation hardening stainless steels.

2.2.0 Classes of cast iron

Cast iron are alloys of iron, carbon and silicon in which more carbon is present than can be retained in solution in solid metal. In common (unalloyed) cast iron, the carbon that exceeds the solubility limit precipitates either as graphite carbon or as iron carbide. The classification of cast iron is based on the shape, distribution and form of carbon phase (s) in their microstructure.

There are six basic types of cast iron which are classified based on how the excess carbon occurs in the microstructure:

(i) White cast iron

(ii) Malleable cast iron

(iii) Grey cast iron

(iv) Nodular cast iron

(v) Austempered ductile iron

(vi) Compacted graphite iron

(http://www.mechanicalenginer.de/latest/examples.news-items-3-php):

Additional class is the high-alloy iron which contains over three percent alloy content. High alloy irons may be a type of white iron, gray iron, or ductile iron; the Matrix may be ferritic or austenitic.

2.2.1 White cast iron.

White cast iron derives its name from the fact that its freshly broken surface shows a bright white fracture. White cast iron has almost all its carbon chemically combined in form of cementite, which is a very hard and brittle constituent (Khanna, 2002). The properties of white cast iron include: high hardness, excellent abrasive wear resistance hence not machinable, high strength in compression, very brittle. White cast iron is the first step in the production of malleable iron castings. Other uses include: crusher jaws and hammers, crusher balls, liners for ball mills, rolls for crushing ores and rolling mills e.t.c

2.2.2 Malleable cast iron.

Malleable cast irons are similar structurally to grey and nodular cast irons because they can have ferrite or pearlitic matrix, the only difference is that malleable cast iron is obtained from the hard and brittle white iron through controlled malleabilizing heat treatment. The properties of malleable cast iron include: high toughness/ductility, good machinability, strong in tension and compression, good wear resistance and vibration damping capacity. Areas of applications include: for tough farm implements e.g ploughs, tractor rakes and harrows, automobile parts such as gears, brake drums, cams, etc (Millis, 1972).

2.2.3. Grey cast iron.

Grey cast irons are irons in which some or all of the excess carbon precipitates as graphite flakes (free carbon) in a steel matrix. Grey cast iron exhibits a grey fracture surface because fracture occurs along the graphite flakes (http://www.mechanicalenginer.de/latest/examples.news-items-3-php).

According to Allen (1969), the matrix structure of grey cast iron can be readily changed by heat treatment but the flakes once formed, cannot be changed by heat treatment. However, the quantity, shape and size of the graphite flakes may vary within a wide range. Based on the type of matrix, this iron could be grouped structurally into.

(i) Ferritic grey cast iron

(ii) Pearlitic grey cast iron

(iii) Ferritic – pearlitic grey cast iron

(iv) Pearlitic – ferritic grey cast iron (Smith 1981) and Allen (1969).

The properties of grey cast iron include: brittleness, low strength in tension (but high in compression), excellent machinability, good wear resistance by the lubricative action of graphite, excellent capability for absorbing vibrations, etc. Grey cast iron is used for engine blocks, machine beds, gear cases, brake drums, flywheels, etc.

2.2.4 Nodular cast iron.

Nodular cast iron or ductile cast iron have free carbon in form of graphite nodules well dispersed in a steel Matrix (Allen, 1969). The mechanical properties are greatly influenced by the steel matrix surrounding the graphite nodules. Typical structures found in nodular cast iron are ferrite, pearlite, and matersite. In the as-cast condition, the microstructure consists of graphite nodules which are surrounded by free ferrite in a matrix of pearlite, giving what is known as the Bull's eye structure (Smith, 1981).

The basic properties of nodular cast iron include good castability, wear resistance, high nodules of elasticity, high tensile strength, high ductility and good machinabliltiy. Nodular cast iron is used to make machine parts such as gears, connecting rods, crankshafts, dies, brake drums, flywheel, etc. Ductile cast iron is used as a raw material to be subjected to austempering process to produce Austempered Ductile Iron (ADI)

(http:/www.ductile.org/didata/section4/4intro.htm). Austempered Ductile iron is an engineering material that is gaining wide acceptance by designers of automobile components, agricultural equipment, rail road parts, mining tools, transportation equipment and general machinery. The success of austempered ductile iron as an engineering material lies in its unique microstructure after heat

treatment which consists of acicular ferrite mixed with high carbon content stable austenite (http:www.sae.org)

2.2.5 Compacted Graphite Iron

Compacted graphite iron contains graphite in the form of thick, stubby flakes. Its mechanical properties are between those of gray and ductile iron. http://www.mechanicalenginer.de/latest/examples.news-items-3-php):

2.2.6 Austempered Ductile Iron (Adi)

Austempered Ductile Iron is a type of cast iron produced by subjecting Ductile cast iron to austempering heat treatment. The heat treating of the ductile cast iron produces the ADI with outstanding mechanical properties that can be varied over a wide range by changing the heat treatment parameters (Elliot, 1988; Branka and Branko, 2004).

The microstructure of ADI is different from austempered steel. During the austempering process, ADI undergoes a two-staged transformation (Branka and Branko, 2004).

2.3.0 HEAT TREATMENT PROCESSES OF STEELS AND DUCTILE CAST IRONS

The most important heat treatment processes usually applied to carbon steels and ductile cast irons are:- Stress relieving, annealing, normalizing, quench hardening and tempering, martempeing, austempering and surface hardening, (http:www.key-to-steel.com/view Article asp?ID = 204)

Properties of steels and cast irons can be controlled and varied over a wide range by heat treatment/ hardening and softening of their matrix structure. However, in cast irons, the shape and distribution of the graphite cannot be changed by heat treatment (Walton, 1971). Heat treatment processes are used to change the following properties of steel:

(i) Improve the toughness

(ii) Increase the hardness

(iii) Increase the ductility

(iv) Improve the machinability

(v) Remove the residual stresses

(vi) Improve the wear resistance,

(http:www.appliedprocess.com/austempering)

2.3.1 Common microstructures in heat treated steels and ductile cast irons

The properties of steels and cast iron depend upon their chemical composition and the resultant microstructures. There are three major structures resulting from austenite transformation, and they are: pearlite, bainite and Martensite; the fourth one however is tempered Martensite which is the structure formed when Martensite is reheated to a sub-critical temperature after quenching (Khanna, 2002).

In terms of properties pearlite microstructure has lamellar appearance, softer than bainite and martensite and its hardness usually increases as the lamellar spacing becomes smaller. Banite structure is known to be tougher than pearlite and martensite (http.www.appliedprocess.com). Bainitic microstructure differs from pearlitic structure in that bainite is accicular while pearlite is lamellar in appearance (Harold, 1971).

Marensite is the hardest and most brittle microstructure in steels and cast irons. Transformation of austenite to martensite is not time dependent as is found in bainite, because martensite forms almost instantly during cooling (quenching) of austenite phase. The hardness of martensite at a given carbon content varies with cooling rate (Rajan et al, 1988).

Tempered Martensitic structures as a class are characterized by relatively high toughness at any strength level. Due to their high ductility at a given

hardness, tempered Martensite is the structure that is aimed at in the heat treatment of steels and cast irons for toughness by quenching and tempering.

2.3.2 Stress Relief Heat Treatment

As the name suggests, this process is employed to relieve internal stresses. Stress relief annealing does not involve major microstructure transformations because it is a low temperature treatment aimed to reduce or relieve internal stresses remaining after casting or mechanical working of a metal component (http:www.key-to-steel.com)

These internal stresses according to Rajon et al (1988) are those stresses, which can exist within a body of metal in the absence of external forces. They are also called residual stresses or locked in stresses. Internal stresses are developed during different operations like solidification of castings, welding, machining, grinding, precipitation, and phase transformation during quench hardening. These stresses enhance the tendency of steels towards craking and dimensional instability. The most common causes of internal stresses in casting are non-uniform cooling of different sections of a component due to differences in thickness.

The process of stress relieving consists of heating steel or cast iron uniformly to a temperature below the lower critical temperature, holding at this

temperature for sufficient time, followed by uniform cooling. As a general rule, stress relief treatment should not change the microstructure (Gilbert, 1969).

2.3.3 Annealing Heat Treatment

Annealing in general means softening. The annealing process involves heating steel or cast iron component to a sufficiently high temperature ($900^{O}C$) in the austenite region, holding at this temperature, and slowly cooling in the furnace to room temperature. The temperature to which steel is heated, and the holding time are determined by various factors such as the chemical composition, size and shape of steel component and the final properties desired (Rajan et al, 1988).

The purposes of annealing are:

(i) Soften the component to enhance machinability

(ii) Relieve internal stresses developed during solidification, machining, forging, rolling and welding

(iii) Homogenise the structures

Depending on heat treatment temperature, annealing can be subdivided into three classes, namely, full annealing, partial annealing and subcritical annealing.

When maximum ductility and good machinability are desired, ductile cast irons are generally given a full ferritizing anneal. The microstructure is thus converted to ferrite, and the excess carbon is deposited on the existing nodules (http://www./key-to-steel.com). Amounts of Manganese, phosphorus and alloying elements such as Chromium and molybdenum should be as low as possible if superior machinability is desired because these elements retard the annealing process.

2.3.4 Normalizing Heat Treatment

Normalizing is a heat treatment process whereby a ferrous component is heated to about 870-950°C (depending on the composition), holding for some time and cooling in air to room temperature. Figure 2.1 shows the partial Fe-Fe_3C-Phase diagram indicating the temperature range for normalizing treatment (Rajan et al, 1988).

In steel castings, normalizing treatment usually produce grain refinement as a pre- hardening condition (Gilbert, 1969). It also removes unfavourable structures such as widmanstatten and cementite network present in the as-cast component.

According to Khanna (2002), normalizing produces microstructures consisting of ferrite (white network) and pearlite (dark areas) for hypoeutectiod

steels, pearlite for eutectoid steels, and pearlite and cementite for hypereutectoid steels.

Generally, the purposes of normalizing are to: refine the grain size and produce a uniform structure, reduce internal stresses, improve structures in welds, produce a harder and stronger steel/cast iron than full annealing, eliminates the carbide network at the grain boundaries of hypereutectoid steels. Normalizing generally improves engineering properties of steels and cast irons. The standard time for normalizing temperature (870 – 950^0C) is 1 hour per 25 mm of section thickness of the component (http://www.key-to-steel.com/view Article.asp?ID = 204). Longer times may be required for alloys containing elements that retard carbon diffusion in the austenite.

2.3.5 Quench Hardening Heat Treatment.

Quench hardening is the process of heating steel/cast iron component to the austenite range (850-950 ^{O}C), soaking for sometime, then rapidly cooled by quenching in oil or water. Water is the most widely used quenching medium because of its low cost, availability in abundance and easy handling, no pollution problem and easy disposal. Oil is the next commonly used quenching medium to water. Other quenching media in use are brine, caustic solution, water based polymer, molten salt (http://www.properquenching.optionyeild.htm). During

quenching of steel, the atoms rearrange from the face centered cubic (FCC) austenite to a body-centered tetragonal (BTC) structure (martensite). The rapid heat removal causes carbon atoms to become trapped in highly-stressed position among the other atoms. These inter-atomic stress give the steel its hardness and strength (http://www.macresbluebook.com/search/company.cfm). The rapid rate of cooling suppresses the transformation of austenite to pearlite

($\propto$+ Fe_3C). The TTT diagram for quench hardening is shown in Figure 2.2. The critical cooling rate (CCR) shown on the nose of the C-Curve is the cooling rate that will just produce an entirely martensitic structure. Beyond this rate (nose, CR), other high temperature products such as ferrite and pearlite would form in the quenched component. Therefore, during quenching the nose of the C-Curve must be avoided to ensure full martensitic structure. Steels with less than 0.25 wt% C cannot be hardened by quenching because the nose of the c-curve on the IT-diagram is too close to the temperature axis .When steel is quenched from the austenite + Ferrite region, all of the austenite transforms completely to Martensite while the ferrite remains unchanged. The presence of this ferrite usually lowers the hardness of the quenched component Also, when steel is quenched from the austenite + cementite region, all of the austenite completely transforms into Martensite while the cementite remains unchanged (Pascoe, 1982).

The high hardness developed by this process is due to the phase transformation (austenite to martensite) occasioned by rapid cooling. This formed martensite is a hard and brittle phase. The hardening temperature range is usually (850-950 ^{O}C) depending on section size and alloy content. The factors affecting as-quenched hardness are austenitization temperature, composition, section size, severity of the quenching medium and presence of alloying elements (Wilson,1975). Quench hardening is applicable to the heat treatable steels (with % carbon in excess of 0.3), nodular cast iron and alloy cast irons (Khanna, 2002). As a rule, quenching must always be followed by tempering, except in cases where extreme hardness is required. Quench hardening produces a hardened structure consisting of martensite and retained austenite. The martensite formed is exceedingly hard, brittle and highly stressed, if the component is put into service in this condition, cracking and distortion may occur. Secondly, the retained austenite in the hardened structure is an unstable phase which can change with time and may alter dimensions of the component.

2.4 TEMPERING

Tempering is the final stage in quench hardening. It consists of heating hardened steel/cast iron component below the lower critical temperature, followed by cooling in air (Rajan et al, 1988). Tempering is also defined as the

process of reheating the as-quenched (Martensitic) steel in order to increase its softness and ductility. This process is achieved when steel/cast iron component is heated above A_1, quenched rapidly at a rate fast enough to miss the nose (or knee) of the isothermal transformation (IT) diagram to form Martensite. The quenched component is taken and retreated to a temperature below A_1 (723^0C) and held for 1–2 hrs to achieve the desired hardness (http. www. latrobesteel. com).

Tempering therefore reduces hardness, strength and wear resistance marginally. However, this marginal loss is adequately compensated by advantages gained by relieving of internal stresses, restoration of ductility, toughness and transformation of retained austenite (Rajan et al 1988, Khanna, 2002).

In the tempering cycle, the structure and properties are determined by the tempering temperature and time (Pascoe, 1982). Tempering may be classified into the following types:

(i) Low temperature tempering ($150 – 250^0$C)

(ii) Medium temperature tempering ($350 – 450^0$C)

(iii) High temperature tempering ($500 - 650^0$C) (Khanna, 2002)

As the tempering temperature is raised, all the mechanical properties alter;

the change in Mechanical properties as the tempering temperature increases is shown in a tempering diagram or property chart on figure 2.3 (Pascoe, 1982). The general features on this diagram are that increasing the tempering temperature increases the ductility and toughness, while decreasing strength and hardness. In the process of tempering a quench hardened component, the tempering temperature is chosen to give a desired combination of properties. The major effect of tempering is that it increases the ductility and toughness of the component above that of the as-quenched structure, and decreases the hardness.

In quenching, two stresses are involved, (i) thermal stresses due to rapid cooling and (ii) transformation stresses due to the increase in volume from austenite to Martensite (http//:www.properquenching.opton.com)

2.5 AUSTEMPERING HEAT TREATMENT

Austempering is one of the heat treatment methods used for hardening of ferrous metals. It is an isothermal heat treatment that produce a structure that is stronger and tougher than comparable structures produced by conventional heat treatment methods (e.g water quenching and tempering) (Imasogie et al, 1991). The austempering process consist of austenitizing a ferrous component to a temperature between 825-950^{O}C, Soaking at this temperature for 1 to 2 hours depending on the component thickness, quenching from this temperature into a

hot liquid medium maintained at a pre- selected temperature between 250-450^{O}C then removed and cooled to room temperature in air (Adewuyi and Afonja, 2000; Dennis, 1963; Rimmer, 1997; http://www.appliedprocess.com/austempering/).

Austempering cycle is shown schematically on the isothermal transformation diagram in figure 2.4

When austenite is cooled rapidly and held isothermally at a temperature between 250-450^{O}C, the austenite is transformed not to pearlite but to bainite. In a similar way to pearlite, bainite consist of an intimate mixture of ferrite and cementite but differs in structure. According to Dennis (1963), Singh et al (2001) and Lawrynowicz (2004), Cementite in pearlite appears as plates in lamellar form, whereas it appears much more finely dispersed precipitates in bainite.

The basis of austempering today is the outcome of the research work of Groosman and Bain. According to Rimmer (1997) and http://www.ductile.org/didata/section4/4intro.htm; The history and origin of austempering began in 1933 when Groosman and Bain were evaluating the metallurgical response of steel cooled rapidly from 788^{O}C to intermittently high temperature and held for various times. They developed relationship between cooling rate and properties. The outcome of the research is what is today commonly called the isothermal transformation diagram or TTT (Time-temperature – transformation) diagrams

shown in Figure 2.4. The figure shows a typical austempering process whereby a component is removed from austenite phase and rapidly quenched into a hot molten bath maintained at a temperature above Ms(Martensite start) and held at that temperature for the desired long period (Isothermal treatment) for the austenite to transform to bainite.

Groosman and Bain were familiar with the conventional metallurgical structure of ferrite, pearlite and martensite. But when they quenched steels at temperatures above martensite starts (Ms) and below the pearlite formation region they discovered another structure which took the form of acicular plate or needle like structure with a feathery appearance. X ray- diffraction, optical and electron microscopy later identified this structure as a combination of acicular ferrite and iron- carbide (Fe_3 C) (Rimmer, 1997). This resultant structure was called Bainite. This structure was found to be stronger and tougher than a comparable quenched and tempered structure. It was then that a novel new heat treatment process called austempering was patented.

According to Rimmer (1997), the austempered microstructure in cast iron was documented by Flinn, and was alternately misnamed as Bainite and acicular pearlite. Years later, the structure was officially classified as auferrite by ASTM and identified as a mixture of acicular ferrite and austenite (bainite). According to Rimmer (1997) ductile cast iron was used as a test material for austempering

during the 1960's and the results were encouraging. This gave birth to Austempered Ductile Iron (ADI). In 1972, the first ADI component (a small hollow crankshaft) went into service.

(http://www.ductile.org/didata/section4/4intro.htm). Till date ADI is gaining more and more popularity in the world markets. This implies that one major area of application of austempering is in the production of austempered ductile iron.

According to Kazerooni et al (1997), ADI components are produced for automotive part, agricultural, machine tools, railroad, construction and military applications. Many of these applications according to Kazerooni et al (1997), make use of the excellent wear resistance of ADI in combination with good impact toughness and high damping capacity. Specific component subjected to austempering includes: gears, crankshafts, rolling mill rolls, hard wares of armored vehicles, gun parts, brake calipers, cams, annular gears, differential gears wear plates for mining componets, digger teeth, mill hammers, agricultural implements such as plows, parts of planters, sprayers and harvesters.

(Imasogie et al, 2000; Singh et al, 2001). Austempering is also used to produce Austempered Gray Iron (AGI), which has excellent dampening effects and wear resistance. AGI is ideal for light duty gears, cylinder liners, bearing collars, and other light-to moderate duty parts (http://www.thermexmetal.com/-

10k). In addition to ductile iron and Gray iron, austempering is typically performed on carbon and alloy steel stampings, steel springs, and ductile iron castings

(http://www.pmiheattreat.com/austempering.htm).

2.5.1 Advantages of austempering over conventional quench and temper heat treatment

One major advantage of austempering is that it is suitable for cast iron parts that cannot be successfully hardened by other methods such as water quenching and oil quenching (http://www.thermexmetal.com/-10k). Austempering usually produces steel and iron parts that are stronger, tougher and more wear resistant than conventionally heat-treated parts. (http://www.dutile.org/magazine/issue1-19).

Austempering usually is substituted for conventional quench and temper heat treatment for either or both of two reasons:

a. To obtain improved mechanical properties

b. To reduce the likelihood of cracking and distortion (ASM metal handbook, 1970). In most applications, austempering is less expensive than conventional water quenching and tempering because in austempering the component is simultaneously hardened and tempered in the hot bath, hence time and cost of operation are reduced.

The benefits of austempering also includes less distortion of the parts in quenching and a microstructure with certain mechanical advantages compared to oil or water quenching (http://www,pmiheattreat.com/austempering.htm).

Such mechanical advantages includes higher tensile strength, improved impact toughness, high ductility; less noise, better wear resistance, lighter components and lower overall components cost (http://www.applied process/benefits/default.htm)

Tables 2.1 illustrates advantages of austempering over other heat treatment methods. The comparison of mechanical properties developed by austempering and other heat treatment methods is usually made at the same hardness or strength level. As could be seen in Table 2.1, the superiority of austempering shows up in such properties as high impact values high percentage reduction in areas and high percentage elongation obtained from tensile test. These better mechanical properties obtained are as a result of the microstructure (bainite) developed by the austempering heat treatment.

Disadvantage of austempering however is that it requires the use of thin section component to ensure rapid cooling of austenite to the temperature of the hot liquid to avoid the nose of the TTT curve and prevent formation of pearlite. Another disadvantage is that austempering process is time consuming, because the isothermal transformation reaction resulting to bainite structure is a time

dependent process, which requires hours depending on the composition of the alloy under austempering, and the austempering equipment (Salt Bath furnace) is also expensive. In addition, austempering process involves a thorough understanding of time and temperature and their effort on ferrous metals during heat-treating process (http://www.ductile.org/magazine/issue1- 19)

Table 2.1 Summary of Tensile and Impact properties of 0.78% C Steel Heat Treated by Quench and Temper Method and by Austempering

S/No	Properties	Quench and Temper method	Austempering
1	Hardness (Rc)	49.8	50.0
2	UTS (MPA)	37555.0	37555.0
3	Percentage elongation	3.75	5.0
4	Percentage reduction in area	26.1	46.4
5	Impact strength (J)	19	50

Source: Avner (1984)

2.5.2 Quenching media for austempering

Austempering is known to be one of the interrupted heat treatment methods, which is carried out isothermally in a liquid medium maintained within bainite formation range (250-450^{O}C). The three major quenching media used for

austempering according to available literature (Adewuyi and Afonja, 2000; ASM metal handbook, 1970; Imasogie et al, 1991) are:

1. Molten salt Bath
2. Molten lead Bath
3. Hot oil (vegetable or mineral oil)

The salt bath furnace is the commonest equipment which has been is use for austempering process for the past 50 years (http://www.newmex.com/ebera/metal/heattreat1.htm).

Molten salt bath is a unique quenching medium in which the vapor phase (the first stage) of quenching does not exist. The bath has high thermal conductivity which helps to achieve fast quenching rates resulting in maximum hardness with minimum distortion.

Each of these molten liquids has its advantages and disadvantages when in use for austempering. Some of the disadvantages include:

1. Salt bath furnace is very expensive, at present it is not manufactured in Nigeria hence its acquisition requires importation.
2. Molten salt baths can be dangerous because nearly all the salts and their combination in the molten state tend to spatter with any little disturbance which could be injurios to the operator (s). In addition, the molten salts have very low viscosities and surface tension. It

has also been found (Taylor, 1974) that Nitrate- Nitrite salt bath mixtures undergoes violent reactions at temperatures above $650^{O}C$.

3. The fumes and vapours generated by chemical reaction either with the work piece or with oxygen on the surface of the bath are poisonous, hence hazardous.

4. Lead is a poisonous metal and melts at $327^{O}C$. Lead bath is usually used for austempering, but is not adapted to high temperature because it vaporizes as the temperature gets higher. These vapors are poisons to the operator(s) and the environment hence it is important to use pure lead that is free from sulphur (http://www.newmex.com/ebear/metal/heattreat1.htm1). Lead has a high density of 11 g/cm^3, therefore, when molten, any steel or cast iron component dropped in it tends to floats; thus additional special fixtures are required to force the steel component into the molten lead during austempering. This amounts to additional cost and inconveniences. Molten lead sticks to the component to be austempered, the removal or cleaning of this solidified lead constitutes another problem. Most at times the component is damaged and scrapped due to this problem.

5. Hot oil also has some associated disadvantages ASM-metal handbook (1970) pointed out that, oils exhibit chemical instability at high temperature. The properties of oil generally change (degrade) as the temperature increases; oxidation of the oils take place, the viscosity also changes at the austempering temperatures. Due to persistent vapour phase in oils, they are slower quenchants than molten salt at elevated temperature, and present fire hazards.

2.6 AUSTEMPERED STRUCTURE IN STEELS AND DUCTILE CAST IRON.

Austempered Ductile Iron (ADI) is a group of ferrous materials whose mechanical properties can be varied over a wide range by a suitable treatment such as austempering (http://www.ductile.org/didata/section4/4into.htm).

Adewuyi and Afonja (2000), Singh et al (2001) reported that during austempering two simple types of bainite are formed: upper and lower bainites. Metallographic observation reveals that upper bainite is acicular (Rajan et al, 1988). The Microstructure of upper bainite consists of ferrite lath with carbides precipitated on the surface of the laths, whereas lower bainite consists of ferrite lath with precipitates of carbide within the laths at an angle to the major axis of the ferrite lath. These upper and lower bainites are shown schematically in

Figure 2.5. Both the upper and lower bainites are nucleated by ferrite. According to Rajan et al (1988), the nucleated ferrite grows into bainitic plate on cooling to and holding at a bainitic temperature region provided for by the molten bath. The bainite plate so formed is surrounded by austenite which is enriched in carbon (high carbon austenite).

The mechanical properties of Ductile Iron and ADI are primarily determined by the Metal Matrix. The properties of ADI are due to its unique Matrix of acicular ferrite and carbon stabilized austenite called ausferrite (http://www.ductile.org/didata/secion4/4intro.htm).

Mallia and Grech (1997) pointed out that austempering takes place in two stages as follows:

Stage I: $\gamma \longrightarrow \alpha + \gamma_{hc}$ (austenite decomposes to bainitic ferrite and carbon enriched austenite (γ_{hc}), a product known as ausferrite.

Stage II. $\gamma_{hc} \longrightarrow \alpha + Fe_3C$ (high carbon austenite further decomposes to ferrite and carbide, a product known as bainite. The austempering process in cast iron (especially ductile cast iron) differs from that in steel because in ductile cast iron, the above two stages of reaction take place, whereas, for steels only the second stage occurs. According to Kazerooni et al (1997), Mallia and Grech (1997), the good combination of mechanical properties obtained in austempered ductile cast iron could be attributed to the unique ausferite structure ($\alpha + \gamma_{hc}$)

produced in stage I. The stage II reaction is however undesirable because it leads to embrittlement due to formation of Fe_3C (Cementite). A typical austempering heat treatment cycle for ductile cast iron to produce Austempered Ductile Iron (ADI) is shown on the isothermal transformation (IT) diagram in figure 2.6. In this process, austenitizing is followed by rapid quenching to an intermediate temperature range ($250 – 450^0C$) for a time that allows the metastable austenite (γ) to transform to platelike ferrite ($\propto$) or ferrite + cementite (bainite), depending on the austempering temperature and time (http://www.key-to-steel.com) Figure 2.6 shows that the austempering reation progresses to a point at which the entire matrix has been transformed to the metastable product (Stage I), then that product is "frozen in" by cooling to room temperature before the true banitic ferrite + cementite phases (Stage II) can appear.

One of the variable known to interfere with the stability of the austenite/ferrite is the chemical composition of the cast iron or steel (Mallia and Grech, 1997). It is generally known that in the chemical composition of ductile cast iron, silicon content is always higher than in steel. Silicon is known to be ferrite stabiliszer and a graphitizer (Wilson, 1975). The higher amount of silicon in ductile cast iron enables the formation of ferrite and high carbon austenite matrix of the stage I reaction. This high silicon content retards the formation of Fe_3C which is usually associated with bainitic structure in steels. Therefore,

during austempering of steels, only the stage II reaction occurs because in the chemical composition of steel, silicon content is usually low, hence formation of cementite become easier. It has been reported that in ductile cast irons, the presence of 2 to 3 wt% silicon prevents the rapid formation of cemetite (Fe_3C), but in steel the low percentage of silicom makes the condition favourable for cementite to form. The formation of cementite in cast iron microstructures due to longer austempering times leads to lower toughness and ductility of cast iron (Elliot, 1988;http://www.key-to-steel.com)

2.7.0 FACTORS CONTROLLING AUSTEMPERING REACTION KINETICS

All chemical reactions are controlled by certain conditions which enable the reaction to proceed spontaneously. To achieve good mechanical properties Zahiri et al (2001) pointed out that the austempering process need to be properly carried out under favorable conditions (time and temperature) to produce the desired microstructure. The conditions that favour the austempering reaction mechanism process are:

i. Austenitizing temperature

ii. Austempering temperature and time

iii. Alloying elements

v. Thickness or section size of component

2.7.1 Austenitizing Temperature

Austenitizing is the process of heating steel/cast iron components to austenite phase. Heating components to austenitizing temperature is the first step of almost all heat treatment processes applicable to ferrous alloys. Full austenitization usually ensures good quality heat treatment. In general, alloy content, the original microstructure, and section size determine the time required for austenitizing.

The austenitizing temperature controls the carbon content of the austenite which in turn, affects the structure and properties of the austempered component (Rajan et al, 1988).

In steels, austenite is formed on heating an aggregate of either ferrite + pearlite + cementite, or cementite + pearlite, depending on whether the steel is hypoeutectoid, eutectoid or hypereutectoid respectively. Khanna (2002) pointed out that the formation of austenite in eutectoid steel differs from that of hypoeutectoid and hypereutectoid steels in the sense that in the former steel, it occurs at a particular temperature ($727^{o}C$) whereas, it occurs over a range of temperatures in the later. Austentizing temperature is an important parameter in cast irons because it determines the Matrix carbon content. The austenitizing temperature is usually in the range $850 – 950^{0}C$ (Elliott, 1988).

In the austenitizing of ductile cast iron, the usual objective is to produce an austenitic matrix with uniform carbon content as possible prior to austempering. For a typical hypereutectic ductile cast iron, high austenitizing temperature is needed so that the iron would be in two phase (austenite + graphite) region (http://www.key-to-steel.com). This temperature however, varies with alloy content (Kazerooni et al, 1997). The amount of carbon in solution increases as the austenitizing temperature increases.

Kazerooni et al (1997) used salt bath furnace and investigated the influence of austenitizing temperatures of 950, 920, 870, 840, and 800^{o}C on austempering kinetics, austempered microstructure, mechanical properties and hardenability of Mn-Mo-Cu alloyed ductile iron, and found that decreasing the austenitizing temperature accelerates the stage I reaction and the introduction of ferrite into the austempered structure.

2.7.2 Austempering Temperature and Time

Austempering temperature is that temperature at which transformation of austenite to bainite occurs isothermally (Rajan et al, 1988). The austempering temperature is the primary determinant of the final microstructure in any austempering heat treatment process. As the austempering temperature increases, the strength and impact toughness vary. The attainment of maximum ductility at

any given austempering temperature is a sensitive function of time (http://www.key-to-steel.com). In almost all ferrous alloys, austempering temperatures occur over a wide range of temperatures. According to Adewuyi and Afonja (2000), austempering temperature range is between 250 - 450^{o}C. For a eutectoid steel, Rajan et al (1988) reported that bainite was formed from the under cooled austenite in the temperature range of 200 – 500^{o}C, and that the transformation of austenite to bainite occurs below a definite temperature known as Bs (bainite start) temperature, while austenite to bainite transformation at a B_f (bainite finish) temperature.

Higher austempering temperatures in the range of 340 – 450^{o}C are used to produce upper bainite structures that promote high ductility (Kazerooni et al, 1997), while lower austempering temperatures in the range of 200-320 ^{o}C are used to produce lower bainitic structures with high hardness. Yazdani and Elliott (1999) investigated the austempering kinetics of ductile iron alloyed with 0.39%C and 0.13%Mo at 285, 320, 375 and 400^{o}C after austenitizing at 870^{o}C then rapidly transferring the samples into a salt bath containing cassel TS220 (K_2NO_3+$NaNO_3$) salt maintained at each austempering temperature as mentioned above. Isothermal transformations of the samples were allowed to take place for a certain period of 1-4320 minutes (72 hours). The samples were then air cooled

after austempering. The results showed that higher austempering temperatures resulted to higher ductility.

The length of time for austempering is important for attainment of maximum ductility at any given austempering temperature and is dependent on the nature/amount of the alloying element used. According to Adewuyi and Afonja (2000), unalloyed ductile iron requires shorter austempering times in the range of 1 – 4 hours, while alloyed ductile cast iron requires longer times because the alloying elements delay the austempering reactions. In unalloyed ductile cast iron, when austempering time is prolonged beyond 4 hours, according to Adewuyi and Afonja (2000), iron carbide (Fe_3C) is precipitated from the residual austenite which could lead to embrittlement.

2.7.3 Effect of alloying elements

The primary aim of alloying elements is to provide sufficient hardenability to the matrix of the metal intended to be austempered. Alloying additions are also usually made to ensure sufficient hardenability when thicker section castings are to be austempered; otherwise, pearlite will form during quenching from the austenitizing temperature to austempering temperature with adverse effects on the mechanical properties (Kazerooni et al, 1997). Zahiri et al (2001), Hasmid and Elliott (1997), Bayati and Elliott (1997) pointed out that the

desired ADI microstructure consists of ausferritic ferrite and stabilized high carbon austenite, collectively known as “ausferrite”. And that the structure of ADI should also be free of pearlite, bainitic carbide, and martensite. But in practice, formation of the carbide and martensite is inevitable owing to the inhomogeneous alloying elements distribution in the matrix (Yazdani and Elliott, 1997).

According to Adewuyi and Afonja (2000), Juneja et al (1989), Elliot (1988), the major elements that play significant roles in enhancing properties during austempering are: carbon, molybdenum, manganese, nickel, cobalt, boron, silicon and copper. These elements are required and used singly or in combination in varying amounts to prevent/delay pearlite formation in given section sizes. Several researchers have been concerned with the influence of alloying elements on the mechanical properties of austempered ductile iron and steels. According to Yadzani and Elliott (1997), the most effective hardenability promoter is molybdenum, but manganese is potent and is cheaper, however, these alloying additions delay the beginning of the stage I and stage II reactions, and the end of stage I reaction. The last influence is particularly strong and is enhanced by the strong segregation of manganese and molybdenum to the grain boundary areas. This segregation is not removed during austenitizing, as a result, the stage I reaction in the grain boundary area may not be completed before the

stage II reaction starts in the eutectic cell. This segregation can slow down the austempering kinetics, hence increasing the overall processing time required to obtain the optimum properties. For this reason, Rimmer (1997) was of the opinion that alloying additions should be controlled to known specific limits depending on the section size of the component to be austempered. The most important determinant of matrix carbon content in ductile iron is the silicon content. As the silicon content increases for a given austenitizing temperature, the carbon content in the matrix decreases (http:///www.key-to-steel.com). The higher austenitizing temperature with its higher carbon content promotes increased hardenability, Mallia and Grech, (1997).

2.7.4 Effect of component thickness on austempered properties

According to Adewuyi and Afonja (2000), a requirement for the production of austempered ductile iron and steel having optimum combination of properties is that formation of pearlite should be avoided. This limits austempering to relatively thin sections which can be rapidly cooled. Rimmer (1997) suggested that since the molten quenching baths for austempering has lower cooling rate, a higher hardnability alloy is needed for effective austempering of thicker components. Managanese and molybdenum have the most powerful effect upon pearlite hardenabiltiy but will also segregate to

graphite nodule sites and do not form detrimental cemetite. Hence, combination of these elements which segregate in opposite directions are selected for their combined positive effect on hardenability (http:///www.key-to-steel.com; Elliot, 1988). Kazerooni et al (1997) in their investigation concluded that the critical bar diameter which can be through hardened decreases with decreasing austenitizing temperature.

2.8 BITUMEN

Bitumen is a black or brown viscous liquid or solid material (thermoplastic in nature) consisting essentially of hydrocarbons and their derivatives, which is soluble in carbon disulphide (Anil and Ronald, 2000; Goni, 2003). Bitumen is abundantly available in Nigeria, and is obtained from two major sources:-

i. Artificial source:- from fractional distillation of crude petroleum in refineries.

ii. Natural Source:- bitumen has been reported to occur naturally in Ondo and Ogun States in Nigeria and other parts of the world (Goni, 2003; Adegoke, 1980). Bitumen occurs naturally 120km East-west belt in Ondo and Ogun States, and that its occurrence covers about 4-6km wide (Adegoke, 1980). Usmani, A.M. (1977) pointed out that natural bitumen probably would have formed by an old

weathered crude petroleum flow to the surface of the earth from the accumulated one below, such that after a long time, the effect of heat and pressure must have caused the evaporation of more valuable and volatile content, hence leaving behind waste residue in form of natural bitumen in those areas. The presence of natural bitumen in those areas is a clear indication of the presence of crude petroleum many years back.

According to Adegoke (1980), the bitumen in Ondo and Ogun States is found in mixture with sand; hence it is called tar sands or bitumen sands. Therefore, if a technique could be invented and developed to successfully extract/wash bitumen from the tar sand in Ondo/Ogun states, it would go a long way to augment the bitumen obtained from artificial sources (refineries). Some uses of bitumen include roofing operations, surfacing of roads (road pavements), pipe coating, undersealing for concrete pavements, hydraulic applications, protective coatings on steel structures to prevent corrosion, lining irrigation canals, water reservoirs, dams, and sea/marine defence structures, and used as adhesives in electrical laminates (WHO, Geneva, 2004).

Usmani (1997) and Anil & Ronald (2000) pointed out that crude petroleum from different sources (oil fields) is never exactly identical in composition. In the same way, the bitumen produced from different sources of crude petroleum differs in chemical composition. Most crude oils for example,

from Saudi Arabia and Kuwaiti produce good or quality bitumen. The Nigerian crude oil which has been known to be of good quality due to its low sulphur content produces the least quantity of bitumen (about 1%), (Adegoke, 1980). Hence for bitumen production purposes, the refineries in Nigeria usually use imported crude oil from Venezuela.

Chemical composition of bitumen

According to Anil and Ronald (2000), the chemical composition of bitumen is complex consisting mostly complex mixture of hydrocarbon molecules. In general bitumen can be classified into:

i. Asphaltenes (ii) Resins (iii) Aromatics (iv) Saturates; these groups are called "SARA" fractions (i.e staturates, Aromatics, Resins and Asphaltenes).

Amos (2002) reported that although the chemical composition of bitumen is highly complex, it consists mainly:

80% - Carbon

10% - Hydrogen

6% - Sulphur

1% - Oxygen

1% - Nitrogen;

Speight (1992) reported that, although no two bitumen's are chemically identical, elemental analysis indicated that most bitumen contain:

79-88wt% - carbon

7-13wt% - hydrogen

Traces up to 3wt% - Nitrogen

Traces up to 8wt% - Sulphur

Traces up to 8wt% - Oxygen

The elemental analysis of bitumen produced from different crude sources as adapted from Speight (1992) and reported by WHO report, Geneva (2004) is shown in Table 2.3. The physical properties of bitumen as conducted by KRPC are shown below:

Softening point – 50.10˚C

Ductility - > 150

Specific gravity – 1.01

Flash point - 378˚C

Boiling point - 350˚C

The boiling temperature of bitumen as determined in the present investigation was found to be 350˚C. From the above data, the boiling point (350˚C) of the bitumen falls well within the bainite formation range (250-450˚C), hence hot bitumen as a material could offer favourable temperature condition for

bainite reaction. In addition, the flash point of bitumen (378˚C) is higher than the boiling temperature, thus bitumen is stable at 350˚C and could be used for austempering without fear/risk of igniting.

Comprehensive physical and chemical analysis of Nigerian bitumen and that of Athabasca was carried out by Goni (2003), and are shown in Tables 2.4 and 2.5

Table 2.3 Elemental analysis of bitumen from different crude sources.

Elements / Crude Source	Carbon (wt%)	H_2 (wt%)	N (wt%)	S (wt%)	O_2 (wt%)	V(mg/kg)	Ni (mg/kg)
Mexican blend	83.77	9.91	0.28	5.25	0.77	180	22
Arkansa louisiana	85.78	10.19	0.26	3.41	0.36	7	0.4
Boscan	82.90	10.45	0.78	5.43	0.29	1380	109
California	86.77	10.94	1.10	0.99	0.20	4	6

Adapted from Speight (1992).

Table 2.4: Chromatographic and elemental analysis of bitumen samples compared to that athabasca samples.

Nigerian Bitumen (Sample A)								
	% of whole bitumen	% C	% H	%N	% O	%S	Mol. Wt	C/H Atomic Ratio
Whole Bitumen	0	86.02	10.43	0.65	1.76	1.32	667	0.69
Sarurates	22.2	85.96	12.85	0.04	0.55	0	352	0.56
Aromatics	12.4	88.78	10.8	0.11	0.65	0.62	412	0.68
Resin I	34.2	84.89	9.94	0.62	3.72	1.41	644	0.71
Resin II	9	30.4	10.16	0.72	7.48	0.99	954	0.66
Asphaltene	23.2	84.62	8.59	1.25	3.19	1.08	4436	0.82
Benzenelnsolubles	0	77.79	8.49	1.95	7.71	2.5	686	0.76

Source: Goni (2003).

Nigerian Bitumen (Sample B)								
	% of whole bitumen	% C	% H	%N	% O	%S	Mol. Wt	C/H Atomic Ratio
Whole Bitumen	0	36.67	10.44	0.67	1.78	1.11	640	0.69
Sarurates	22.3	36.4	12.86	0.04	0.72	0	363	0.56
Aromatics	12.9	88.91	10.75	1.05	0.45	0.72	412	0.69
Resin I	33.2	81.85	9.86	0.65	4.59	1.24	560	0.69
Resin II	11.6	82.07	10.14	0.87	5.9	1.15	847	0.67
Asphaltene	21.3	85.14	8.68	1.29	2.7	1.17	6173	0.82
Benzenelnsolubles	0	74.03	8.6	2.15	9.33	2.33	539	0.72

Source: Goni (2003).

Table 2.5: Results of water soluble test carried out on four (4) Nigerian Bitumen samples compared against the Athabasca deposit

Cations (mg/kg)	Nigeria A	Nigerian B	Nigeria C	Nigeria D	Athabasca
Calcium	57	26.5	215	145	0.8
Iron	330	20	555	1190	0.37
Lead	0.1	ND	0.1	0.1	0.02
Magnesium	11.0	5.0	26.0	18.0	0.2
Manganese	1.3	0.6	4.7	11.5	0.032
Potassium	5.5	3.0	3.0	2.5	1.4
Silicon	14	6.0	13	12	3.6
Sodium	13.5	6.4	18.0	33.0	35.3
Zinc	0.8	0.4	3.1	7.3	0.08

Source: Goni (2003).

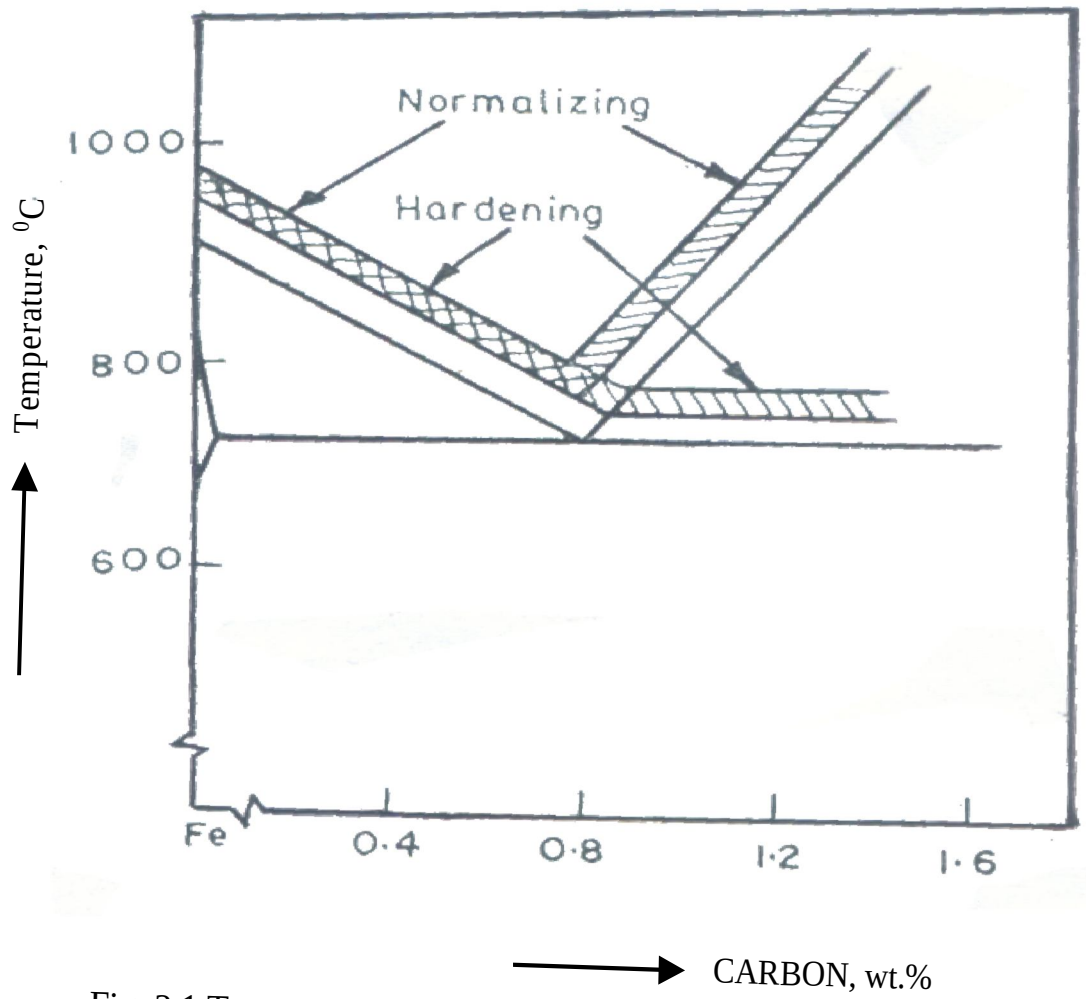


Fig. 2.1 Temperature ranges for normalizing and hardening treatment of steels. (Source: Rajan et al, 1988)

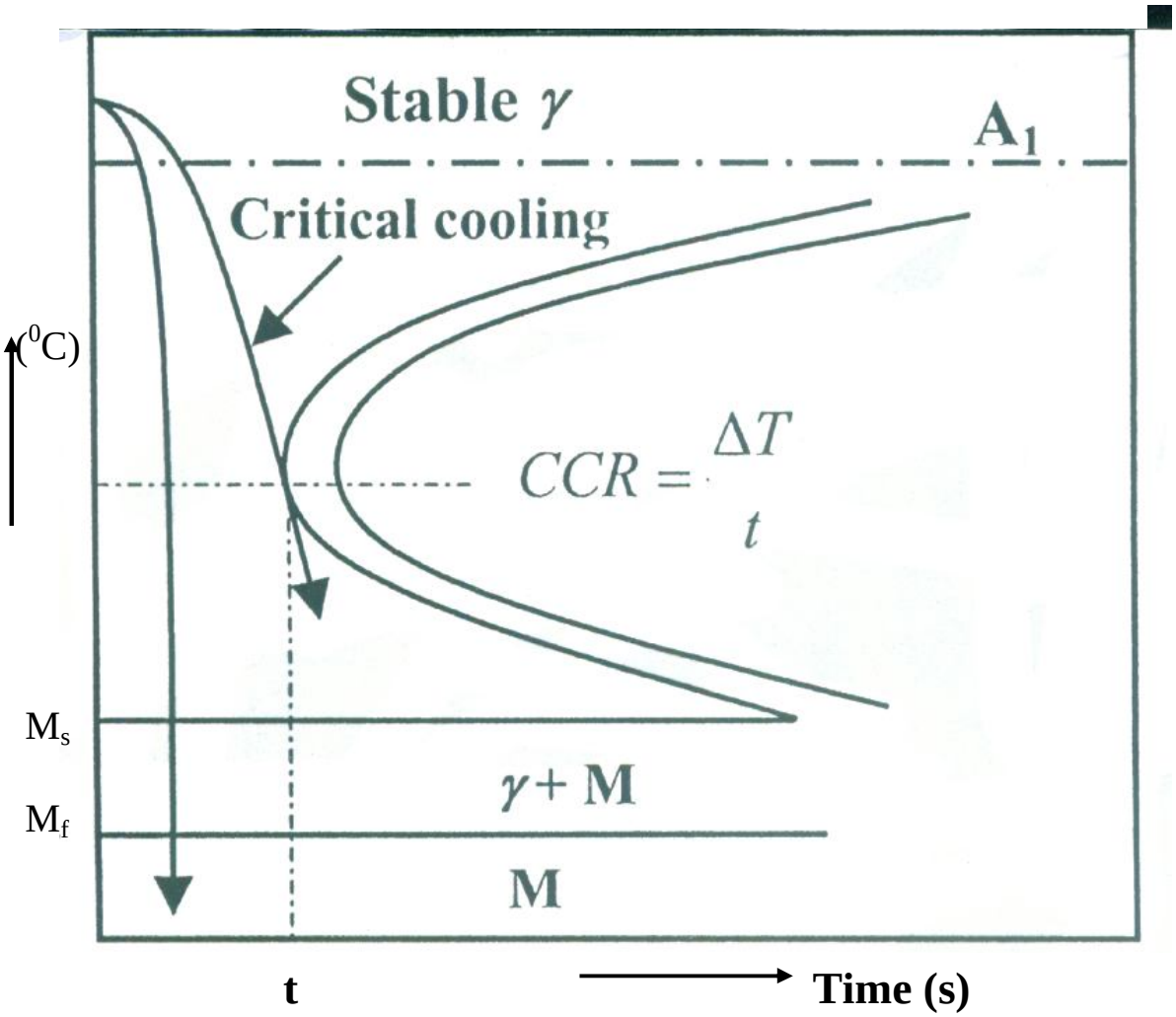


Fig. 2.2: Isothermal transformation (IT) diagram for quench hardening heat treatment. Source: http://appliedprocess.com/austempering/

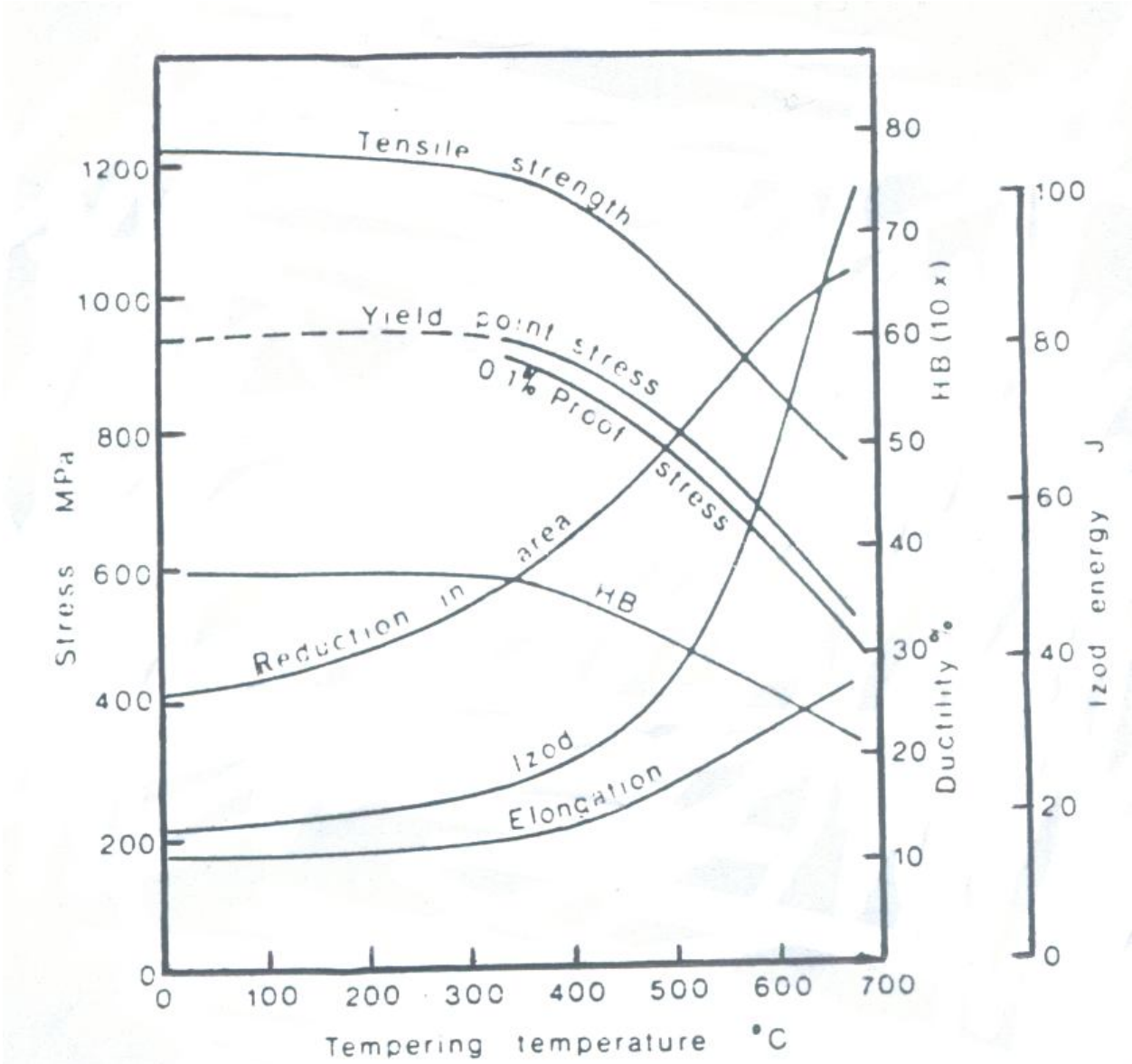


Fig. 2.3: Tempering diagram or property chart for water – quenched 30mm diameter bars of En 1 steel. (Source: Pascoe, K. J, 1982).

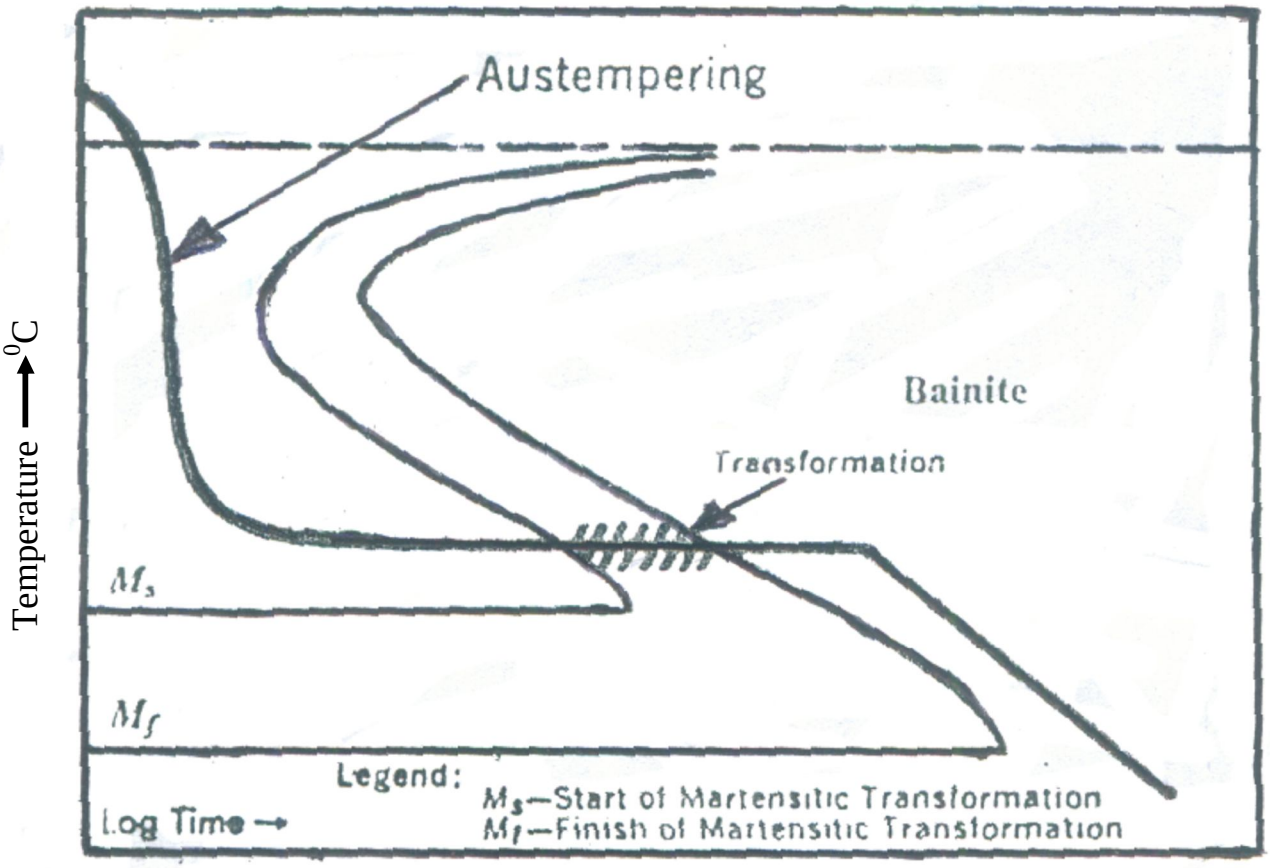


Fig. 2.4: Schematic of a typical austempering process (Source: http://www.appliedprocess.com/austempering/)

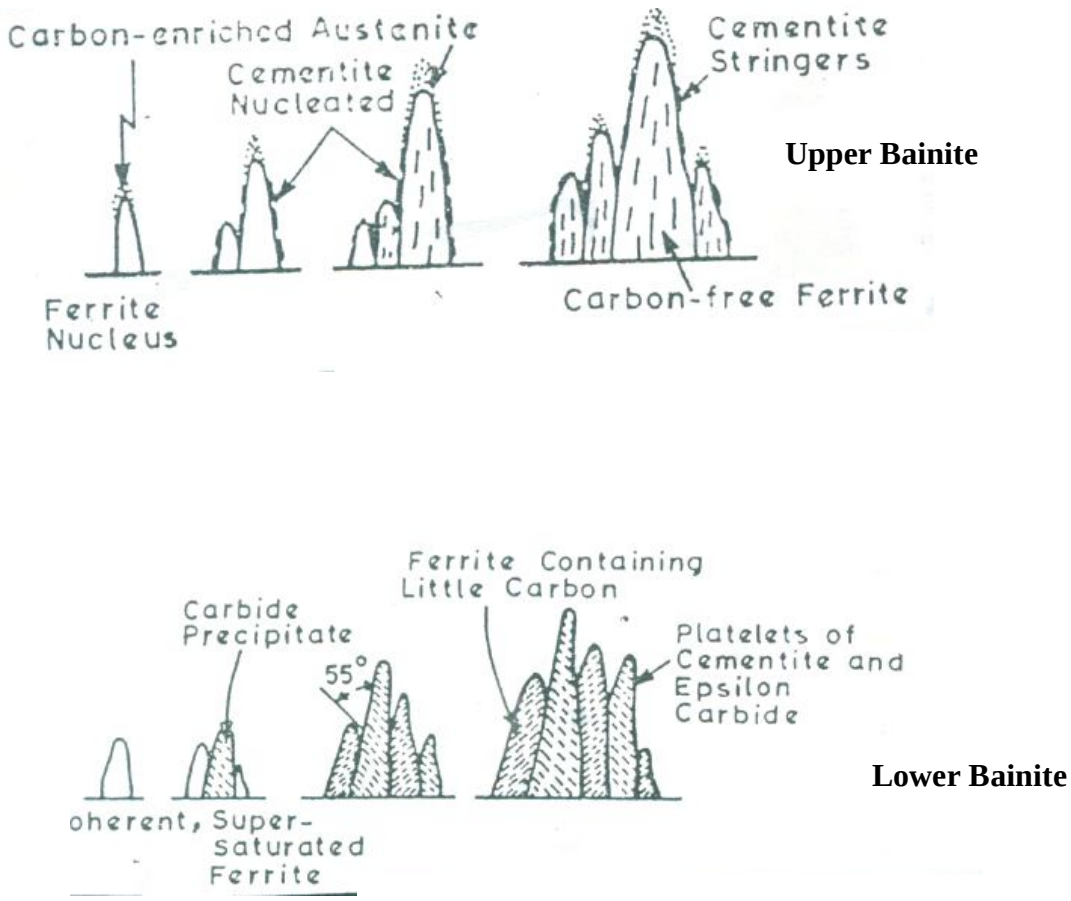


Fig. 2.5: Schematic diagram showing the stages in the formation of upper bainite and lower bainite. Source: (Rajan et al., 1988)

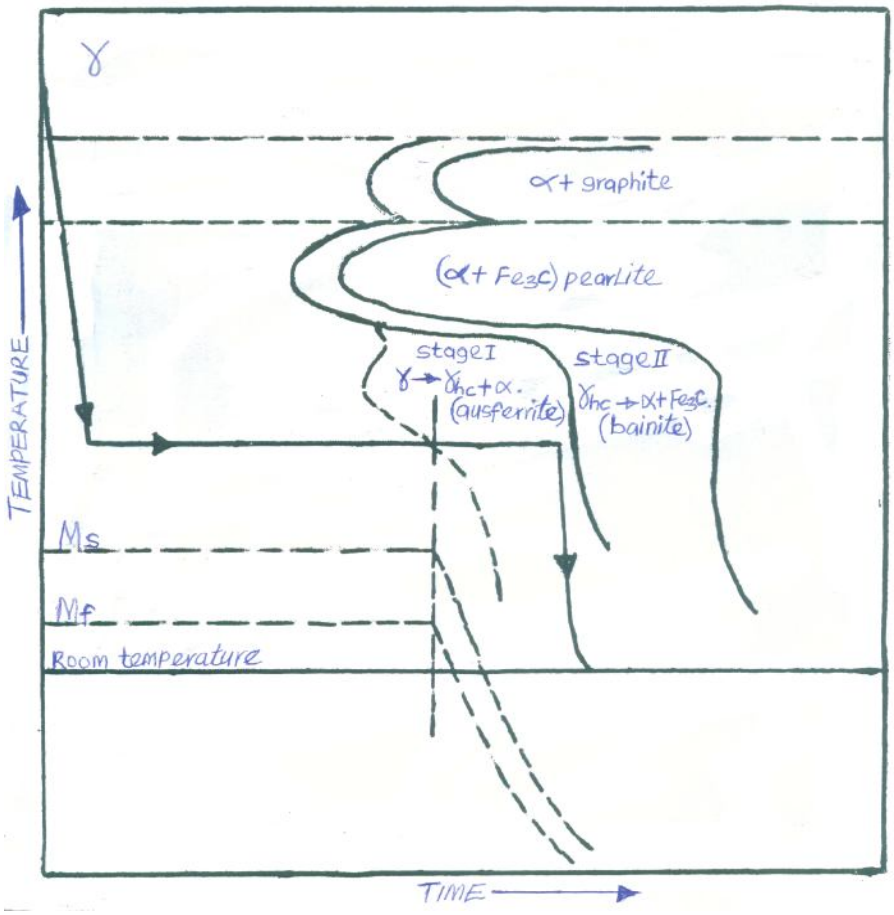


Fig. 2.6: IT diagram of a processing sequence for austempering of ductile cast iron. Source: (htpp://www.key-to-steel.com)

CHAPTER THREE

3.0 MATERIALS AND METHOD

3.1 Material and Equipment

Medium carbon steel, high carbon steel and ductile cast iron were used as test samples to evaluate the hot bitumen bath as an austempering medium. The chemical composition of the alloys is shown in table 3.1

Table 4.1: Chemical composition of the three ferrous alloys used for the investigation.

Grade of alloy	Composition (%)								
	C	Mn	Si	S	P	Mg	Ni	Cr	Balance
0.55%C-Steel	0.55	0.97	0.25	0.030	0.033	-	-	-	Fe
0.75%C-steel	0.75	1.12	0.32	0.02	0.04	-	-	0.001	Fe
Ductile cast iron	3.63	0.36	2.26	0.02	0.03	0.03	0.05	0.07	Fe

The equipment used for the investigation were: An electrically heated carbolite muffle furnace with maximum heating temperature of 1200°C, medium sized kerosene stove and steel pot for the purpose of heating the bitumens, struer's hot mounting press for mounting of all metallographic samples, laboratory mercury thermometer with maximum reading of 380^0C. Avery Denison Vickers/Brinell hardness tester, Carlzeiss Jena optical metallurgical

microscope; Hounsfield tensile testing machine; grinding Buehler strip grinder and polimet polishing machine.

3.2.0 Method

The following steps as outlined below were followed during the experimental process.

3.2.1 Machining of test samples

The three grades of ferrous alloys (0.55%C, 0.75%C and ductile cast iron) were cut and machined to standard izod impact samples, tensile test samples and hardness/microstructural analysis samples. A small hole was drilled on each of the hardness samples through which steel wire was passed to facilitate their removal from the hot bitumen bath.

3.2.2 Preparation of the austempering medium (hot-bitumen bath)

Bitumen obtained from KRPC was collected in a steel pot and placed on a four corner kerosene stove and heated. A laboratory thermometer (380°C Max) was placed into the steel pot containing bitumen and heated to its boiling temperature. The boiling temperature of bitumen was found to be $350^0C \pm 5^0C$. which was used as the austempering temperature in this research work. The

laboratory thermometer was constantly placed in the hot bitumen to ensure that the temperature of hot bitumen was at 350^0C.

3.2.3 Normalizing of Samples

The three alloys were machined to izod impact, tensile and hardness test samples. One sample each from the three grades was taken and kept aside as the untreated (as-received or as-cast). The remaining samples were given a normalizing heat treatment by heating the samples in an electrical muffle furnace to 900°C, soaked for one hour, removed and then cooled in air. One sample each from the grades was taken and kept aside as normalized samples.

3.2.4. Quench Hardening and Tempering of samples in Water and Bitumen at Room Temperature

Two normalized hardness and two izod impact samples were taken from each alloy. These samples were placed in a crucible, loaded into the furnace, heated to 900°C, soaked for one hour, then removed and quenched in water at room temperature. One set (hardness and izod impact sample) was tested in the as quenched condition while the other set was tempered by reheating in the furnace at 350 C for 1 hour. Another set of normalized samples were taken, heated in the same way as described above and quenched in bitumen at room

temperature; tempering was also done after which hardness and izod impact tests were carried out.

3.2.5 Austempering Heat Treatment

a) Normalized hardness, izod impact and tensile test samples of 0.55%C-steel, 0.75%C-steel and ductile cast iron were austempered as outlined below; a. Normalized hardness, izod impact and tensile test samples of 0.55%C-steel were taken and placed in a crucible, loaded into the furnace, heated to 900°C, soaked for one hour, then removed and quenched in hot bitumen bath boiling at 350°C. After five minutes, the first set of samples (one hardness, one impact and one tensile) were removed from the hot bitumen bath, cooled in air and washed in kerosene, then with soap solution. Another set of samples were removed after 30 minutes, 1 hour, 2 hours, 3 hours, 4 hours and 5 hours respectively, cooled in air and washed in kerosene then in soap solution.

b) The same process was applied to 0.75%C steel and ductile cast iron samples respectively.

3.2.6 Austempering of Ductile Cast Iron Samples in Hot-Bitume Bath from different Austenitizing Temperatures

Normalized hardness and izod impact samples of ductile cast iron were placed in a crucible and loaded into the furnace, heated to 800 C and soaked for one hour. The samples were then removed and quenched in hot bitumen bath maintained at 350°C, After five minutes, two samples (each of hardness and izod impact) were removed, cooled in air, washed in kerosene, then rinsed in soap solution. Another set of samples were removed from the hot bitumen bath after 30 minutes, 1 hour, 2 hours, 3 hours, 4 hours and 5 hours respectively. The same process was repeated as described above for austenitizing temperatures of 850, 900 and 950°C. The austempered samples were then subjected to hardness and izod impact tests.

3.2.7 Metallographic Sample Preparation for hardness Test and Microstructure

All the hardness samples (both untreated and heat treated) involved in this investigation were subjected to thorough metallographic sample preparation processes as outlined below:

a. Rough machine grinding of all the samples was carried out successively on 60, 80, 120 and 180 grifts abrasive emery grinding papers.

b. Fine machine grinding of all the samples was carried out on 20, 400 and 600 grits abrasive emery papers using wet type process.
c. All the ground samples were polished on a polymet polishing machine.
d. All the polished samples were tested for hardness using Avery Denison Vickers/Brinell hardness tester. The hardness test was conducted on three different points on the same surface of metal sample and average taken.
e. All the samples were repolished after the hardness test and atched in solution of 2% Nital (2% Nitric acid in ethanol).

3.2.8 Mechanical Tests (Hardness, izod impact and Tensile)

Prepared samples in the untreated and heat treated conditions involved in the investigation were subjected to hardness test using Avery Denison Vickers/Brinell hardness testing machine using Vickers scale.

The izod impact samples were subjected to impact test using Avery Denison impact testing machine; while the tensile samples were subjected to tensile test using Hounsfield Tensometer with Maximum Capacity of 20 Tonnes. Each sample was subjected to tension till fracture after which tensile strength, percent elongation and percent elongation and percent reduction in area were taken. The results obtained are shown in Appendix B (Tables $B_1 - B_{11}$) and in figure 4.1 – 4.6.

CHAPTER FOUR

5.0 RESULTS

The results obtained from this research work are as shown in Appendix A (Microstructures) and Appendix B (Table of results for mechanical properties).

4.1 MECHANICAL PROPERTIES OF 0.55%C – STEEL, 0.75% C-STEEL AND DUCTILE CAST IRON IN THE AS-RECEIVED NORMALIZED, QUENCH AND TEMPERED CONDITIONS.

The results of the mechanical properties (hardness, izod impact and tensile) obtained for 0.55%C- steel 0.75% C-steel and ductile cast iron in the as-received, normalized, quench and tempered are given in Appendix B (Table B1 – B3, and B9 – B11).

For the 0.55%C, 0.75%C- steels and ductile cast iron, the highest hardness values of 645, 780 and 475HV respectively were obtained for the as quenched condition. The water quenched condition also resulted to lowest tensile strength of 102, 51 and 153N/mm^2 for 0.55%C, 0.75%C – steels and ductile cast iron respectively. However, minimum impact strength values of 3J for both grades were obtained in the as quenched condition. For both grades, the quenched and tempered hardness values are more than those for the normalized condition. However, maximum impact strength of 51, 26 and 20J were obtained respectively for the normalized condition.

The as-received samples of 0.55%C – steel 0.75%C- steel and ductile cast iron gave hardness values of 200, 260 and 148HV respectively, while the highest impact values of 55 and 28J were recorded for 0.55%C and 0.75% C-Steels respectively. However, the impact strength value is less for the ductile cast iron in the as received as compared with normalized sample.

In the as-received condition, the tensile strength of 0.55%C, 0.75%C – steels and ductile cast iron were 713, 764 and 789N/mm^2 respectively. Water quenched resulted to lowest tensile strength of 102, 51 and 153N/mm^2 for the 0.55%C, 0.75%C- steels and ductile cast iron respectively.

4.2 MICROSTRUCTURES OF AS RECEIVED, HEAT TREATED AND AUSTEMPERED PLAIN CARBON STEELS AND DUCTILE CAST IRON

4.2.1 As- received structure

The as-cast structure of ductile cast iron is shown in Appendix A (Micrograph A_1). The structure was pearlitic with ferrite shell surrounding graphite nodules. The as-received structures of 0.55%C- Steel and 0.75%C-Steel are shown in Micrographs A_2 and A_3; both structures showed grain boundary ferrite in pearlite matrix.

4.2.2 Normalized structure

The normalized structure of ductile cast iron, 0.55%C and 0.75%C- steel are shown in micrographs A_4, A_5 and A_6 respectively. These structures shows pearlite.

4.5.3 As- quenched structure

The microstructures of ductile cast iron, 0.55%C and 0.75% C- steel quenched in water are shown in micrographs A_7, A_8 and A_9, while those quenched in bitumen at room temperature are shown in micrographs A_{10}, A_{11}, and A_{12} respectively. In all these structures, martensite was present.

4.5.4 Austempered microstructure

Micrographs A_{13} to A_{19} show the structure of ductile cast iron austempered at 350^0C for 5 mins, 30 mins 1 hr, 2hr, 3 hrs, 4 hrs and 5 hrs respectively. At the initial period of austempering, a mixture of martensite and retained austenite was obtained. However, as austempering time was increased progressively, ausferrite structure (Bainite) formed and some quantity of retained austenite. Micrographs A_{20} to A_{26} show the structure of 0.55%C- steel while micrographs A_{27} to A_{32} show the structure of 0.75%C – steel austempered at 350^0C for 5 mins, 30 mins, I hr, 2 hrs, 3 hrs, 4 hrs and 5 hrs respectively. In both steels, at the initial austempering time, a mixture of martensite and retained

austenite was obtained while higher austempering times gave a mixture of bainite and retained austenite.

4.3 HARDNESS AND IMPACT STRENGTH VALUES OF AUSTEMPERED DUCTILE CAST RION, 0.55%C AN 0.75% C – STEELS

Figure 4.1 and 4.2 shows the effect of austempering time on the hardness and impact strength of ductile cast iron austempered at 350^0C from the austenitizing temperature of 900^0C, respectively, while figures 4.3 and 4.4 show the effect of austempering time on the hardness and impact strength of 0.55% C and 0.75%C- steel respectively. The regression analysis of each curve was done using the spss-programme (statistical programme for social sciences and the regression equations are as indicated against each curve.

4.4 HARDNESS AND IMPACT STRENGTH VALUES OF AUSTEMPERED DUCTILE CAST IRON FROM DIFFERENT AUSTENITIZING TEMPERATURES

Table B_7 and figure 4.5 show the effect of austenitizing temperature and time on the austempered hardness of ductile cast iron. Table B_8 and figure 4.6 show the effect of austenitizing temperature on the austempered impact strength

of ductile cast iron. The regression analysis of each curve was done and the regression equations are shown against each curve.

4.5 TENSILE PROPERTIES OF AUSTEMPERED DUCTILE CAST IRON, 0.55%C AND 0.75%C - STEELS

Table B_9 (Appendix B) show the effect of austempering time on the austempered tensile properties of ductile cast iron. This table also show the tensile properties of ductile cast iron in the as-cast, normalized, water quench and tempered condition.

Table B_{10} show the effect of austempering time on the austempered tensile properties of 0.55% carbon steel. Tensile properties of the 0.55% C steel in the as- received, normalized, water quenched, and water quenched and tempered conditions are also shown in this table.

Table B_{11} show the effect to austempering time on the austempered tensile properties of 0.75% steel. The table also show tensile properties of 0.75%C steel in the as-received normalized, water quenched, and water quenched and tempered condition.

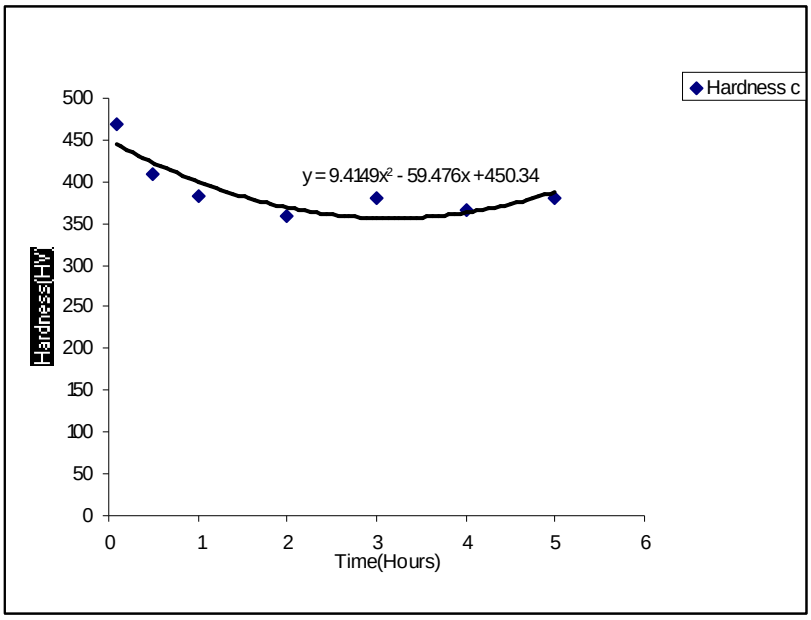


Fig. 4.1: Effects of austempering time on the hardness of ductile cast iron samples austenitized at 900^0C and austempered in hot bitumen bath at 350^0C

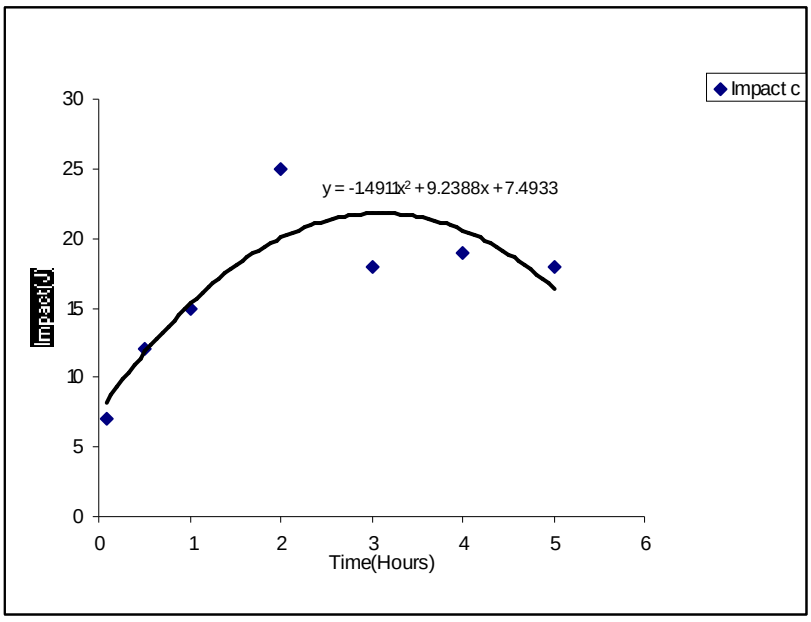


Fig. 4. 2: Effect of austempering time on the impact (J) of ductile cast iron samples austenitized at 900^0C and austempered in hot bitumen at 350^0C

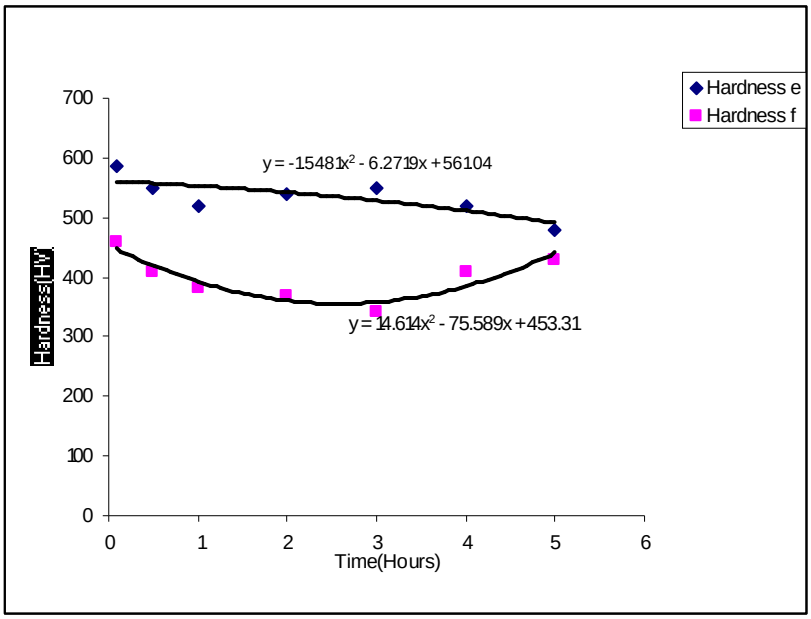


Fig. 4.3: Effect of austempering on the hardness of 0.55%C and 0.75%C – steel samples austenitized at 900^0C and austempered in hot bitumen bath at 350^0C

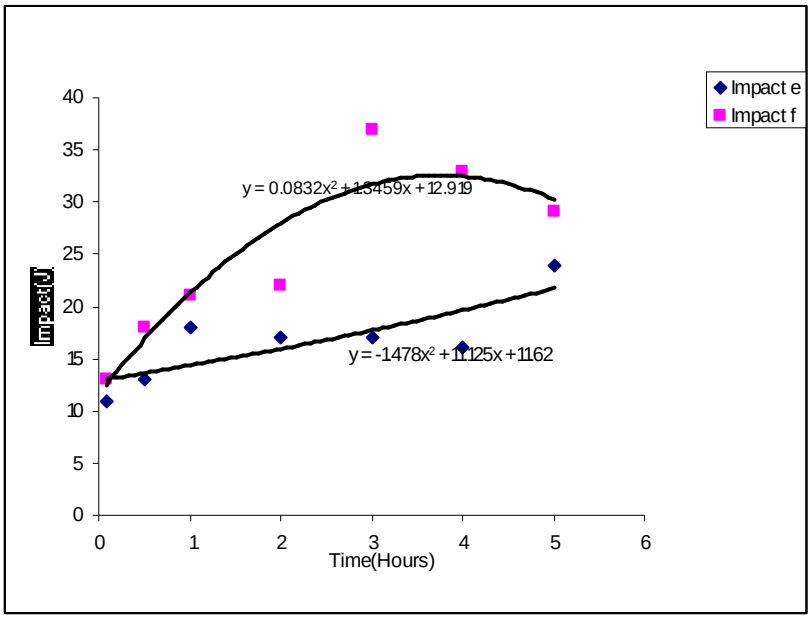


Fig. 4.4: Effect of austempering time on the impact of 0.55%C and 0.75%C – steel samples austenitized at 900^0C austempered in hot bitumen bath at 350^0C.

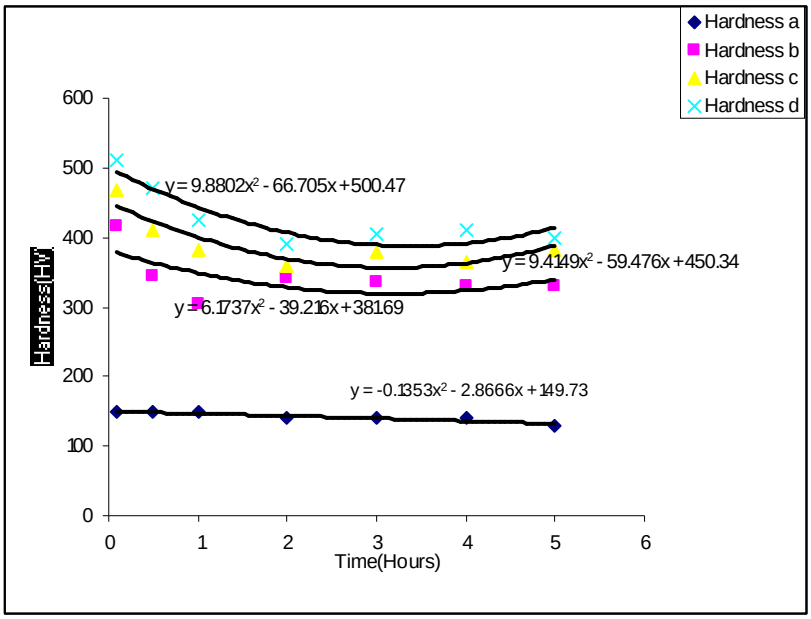


Fig. 4. 5: Effect of austenitizing temperature and time on the hardness of ductile cast iron samples austempered in hot bitumen bath at 350^0C.

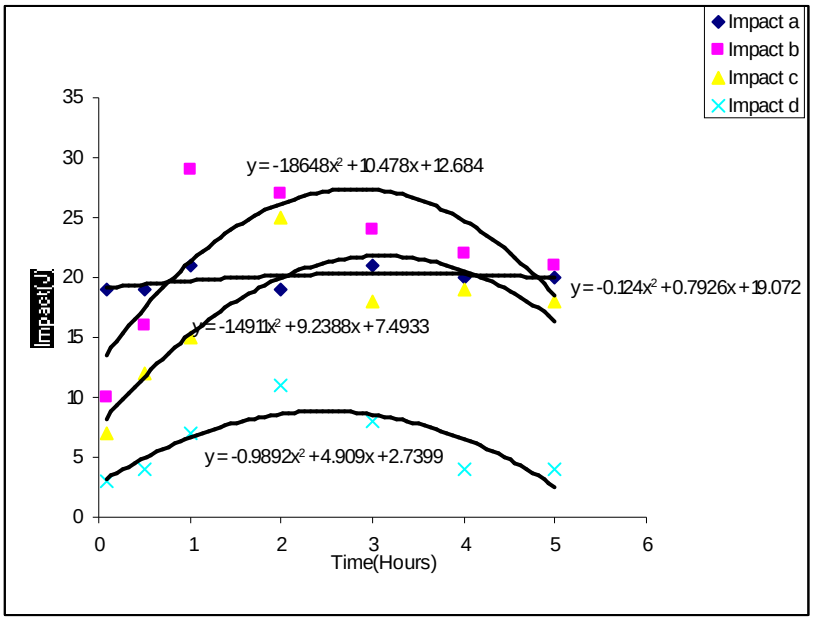


Fig. 4.6: Effect of austenitizing temperature and austempering time on the impact energy of ductile cast iron austempered in hot bitumen bath at 350^0C

CHAPTER FIVE

5.0 DISCUSSION

5.1 EFFECT OF AUSTEMPERING TIME ON THE HARDNESS AND IMPACT STRENGTH OF DUCTILE CAST IRON AUSTEMPERED IN HOT BITUMEN.

Figure 4.1 show the effect of austempering time on the hardness of ductile cast iron samples austempered in hot bitumen at 350^0C from the austenitizing temperature of 900^0C. It is evident that as the austempering time was increased, the hardness decreased, reached a minimum at 2 hours and increased slightly again. The higher hardness obtained at the austempering time of 5 minutes may be due to the fact that the time was too short for reasonable austempering reaction to have occurred before the sample was removed from the hot bitumen and cooled in air. The retained austenite which did not have enough time to transform to ausferrite transformed to martensite. The martensite formed contributed to the high hardness obtained at this austempering time. The drop in hardness at the austempering times of 30 minutes, 1 and 2 hours is attributed to the on set of the stage1 reaction. Austenite has more time to transform to ausferrite structure, hence leading to a decrease in the quantity of untransformed austenite after the austempering heat treatment.

As the austempering time was further increased, the hardness increased. This is in agreement with the findings of earlier researches carried out by Yazdani and Elliott (1999), and Ming (1993). The trend of the hardness in the present work correlates well with the structure obtained in micrographs A13-A19.

Figure 4.2 show the effect of austempering time on the impact energy of ductile cast iron samples austempered in hot bitumen at 350^0C from the austenitizing temperature of 900^0C. It is evident that as the austempering time was increased, the impact energy increased, reached a maximum at 2 hours then decreased. The lowest impact energy obtained at the austempering time of 5 minutes is due to the presence of martensite formed from untransformed austenite in the structure. This is because 5 minutes probably was too short for any meaningful transformation of austenite to ausferrite, hence some of the unreacted austenite transformed to martensite when the sample was cooled to room temperature. As the austempering time was increased to 30 minutes, 1 and 2 hours, the impact energy increased correspondingly. This increase may be due to progression of stage 1 austempering reaction which might have caused considerable quantity of austenite to transform to ausferrite structure.

At the austempering times of 3, 4 and 5 hours, the impact energy decreased. This decrease could be attributed to the setting in of stage II

austempering reaction. In the stage II reaction, austenite transformed to ferrite plus cementite ($\alpha hc \longrightarrow \alpha + Fe_3C$), the ausferrite structure (which is responsible for high impact energy values in ADI) breaks down into banitic structure containing cementite. The cementite formed in this stage was responsible for the decreased impact energy at higher austempering times.This is in agreement with the earlier researches carried out by Mallia and Grech (1997); Yazdani and Elliot (1999) and Kazerooni et al (1997).

5.2 EFFECT OF AUSTEMPERING TIME ON THE HARDNESS AND IMPACT STRENGTH OF 0.55% C AND 0.75%C STEEL AUSTEMPERED IN HOT BITUMEN

It is evident from figure 4.3 that as the austempering time was increased, the hardness decreased, reached a minimum and increased again. The 0.55%C steel exhibited this behaviour more. At all the austempering times used, the hardness of 0.75%C steel was higher compared to 0.55%C steel. This higher hardness is attributed to higher carbon content (Wilson, 1975 and Dennis, 1963). Comparing this figure and the values of hardness in the as-received condition as contained in table B1; it is evident that the hardness of 0.55%C and 0.75%C steel increased after austempering. This increase could be as a result of bainite structure formed due to the austempeirng heat treatment.

Figure4.4 show that the impact energy of 0.55%C and 0.75%C steel was low initially, but as the austempering time was increased, the impact energy also increased, reached a maximum at 3 hours then decreased again. It is also evident from this figure that at each of the austempering times, 0.55%C steel had higher impact energy than the 0.75% C steel, this is due to difference in carbon content. For both grades of steel, the low impact energy obtained at the initial austempering times of 5 and 30 minutes may be due to the presence of high quantity of martensite in the structure.

5.5 EFFECT OF AUSTENITIZING TEMPERATURE AND AUSTEMPERING TIME ON THE HARDNESS AND IMPACT STRENGTH OF DUCTILE CAST IRON AUSTEMPERED IN HOT BITUMEN.

Figure 4.5 show the effect of austenitizing temperature and austempering time on the hardness of ductile cast iron samples austempered in hot bitumen at 350^0C. It was observed that, austenitizing the sample at 800^0C then austempering at various times did not result to much improvement in hardness compared to the normalized samples. The hardness value of the normalized ductile cast iron as shown in table B1 was found to be 180 HV, when these samples were austenitised at 800^0C and austempered for various times as shown in figure 4.5, the hardness reduced to average of 149HV at all the austempering times. The reason for this could be as a result of the high silicon content of ductile cast iron.

Silicon being a graphitizer and ferrite stabilizer, its presence in high quantity may have raised the eutectoid temperature above 800^0C. This means that quenching from 800^0C will result to no transformation hence the same ferrite would still be prediminant in the structure. The presence of this ferrite (soft phase) made the treated samples to have low hardness.

Figure 4.5 also show that at each of the austenitization temperatures of 850, 900 and 950^0C, the hardness values were found to be high at initial austempering times but decreased to a minimum at 2 hours and increased again as the austempering time was increased. The high hardness at 5 minutes, 30 minutes and 1 hour could be due to the presence of martensite formed from retained austenite. The higher hardness obtained at the austempering times of 3, 4, and 5 hours may be attributed to the presence of cementite formed during stage II reaction. An increase in hardness as the austenitization temperature was increased from 850- 950^0C was observed. The reason for this could be that the ductile cast iron samples were fully austenitized such that on quenching, martensite structure formed which resulted to high hardness.

Figure 4.6 show the effect of austenitizing temperature and austempering time on the impact strength of ductile cast iron samples austempered in hot bitumen at 350^0C. It is evident that at the austenitizing temperature of 800^0C, the impact energy remained almost constant with increase in austempering time.

This shows that there was no transformation in those samples austenitized at 800^0C and austempered. As stated above, the reason may be due to the high silicon content (2.26%) of the ductile cast iron. Silicon being a ferrite stabilizer, raised the eutectiod temperature above 800^0C. This is in agreement with Mallia and Grech (1997) that silicon is a graphitizer and a ferrite stabilizer; therefore its presence in ductile cast iron in high quantity raised the eutectoid temperature, on quenching from 800^0C, ferrite formed. This ferrite is known to have low hardness and strength.

Figure4.6 further show that at each austenitizing temperature of 850, 900 and 950^0C, the impact strength values were low at the initial austempering times of 5 and 30 minutes. However, as the austempering time increased, the impact strength increased, reached a maximum at 2 hours then decreased. The low impact strength at the initial austempering times could be attributed to the presence of martensite formed from retained austenite. The highest impact strength obtained at the austenitization temperature of 850^0C is due to full austenitization brought about by the high temperature. This temperature was well above the eutectoid temperature, hence when the ductile cast iron samples were quenched from this temperature, full martensite structure was obtained which resulted to high hardness.

5.6 EFFECT OF AUSTEMPERING TIME ON TENSILE PROPERTIES OF AUSTEMPERED DUCTILE CAST IRON, 0.55% C AND 0.75% C-STEEL IN HOT BITUMEN.

Table B9, B10 and B11 show the values of tensile strength, % elongation, and % reduction in area of ductile cast iron, 0.55% C and 0.75% C steel samples respectively austempered in hot bitumen at 350^0 C. The results in table B9 shows that austempering time of 5 minute resulted to tensile strength of 458 N/mm^2 with 1 % elongation and 0% reduction in area, as the autempering time increased the tensile strength also increased as well as the % elongation and % reduction in area up to 2 hours. The increase may be due to formation of ausferrite structure in the samples. Also the higher ductility obtained within this range of austempering time is higher than the ductility obtained in the as- cast condition, this shows that the austempering process improved the ductility of the ductile cast iron. The tensile strength of the samples austempered at 3, 4 and 5 hours could not be obtained because during the test, the force required to fracture those samples was above the maximum capacity (20KN) of the hounsfield tensometer. However, the % elongation and % reduction in area was seen to decrease as the austempering time increased beyond 2 hours.

As shown in tables B10 and B11, similar problem was encountered. Austempering of 0.55% C- steel for 5 mins resulted to tensile strength of 255 N/mm^2 with 1% elongation and 0% reduction in area; as the austempering time

increased to 30mins, the tensile strength, % elongation and % reduction in area increased to 509 N/mm^2 5% and 2 % respectively. From 1 hour to 5 hours, the force needed to fracture the sample was beyond the capacity (20KN) of the hounsfiled tensometer. For the 0.75% C steel, the sample austempered for 5 minutes gave tensile strength of 407 N/mm^2, 0.5% elongation and 0% reduction in area. The tensile strength of samples austempered for 30minutes, 1,2,3,4. and 5 hours could not be obtained because the force needed to fracture them was beyond the capacity of the hounsfield Tensometer .

CHAPTER SIX

6.0 CONCLUSIONS AND RECOMMENDATIONS

6.1 CONCLUSION

In this research, bitumen was identified to be a material that could be used as a quenching medium (austempering) for ductile cast iron and steel.

From the observations and analysis of the results obtained, the following conclusions could be drawn:

1. Hot bitumen at 350°C was able to cause the formation of "bainite structure" in the samples, thus could be used as austempering medium.
2. Austempering in hot bitumen at 350°C resulted to increase in hardness. The as-received hardness (200HV) of 0.55%C- Steel increased to 430HV, while 0.75% C-steel with as –received hardness (260HV) increased to 548HV and the as- cast hardness (148HV) of ductile cast iron increased to 410HV.
3. At 3 hours of austempering, a full austempered microstructure (a mixture of high carbon austenite and ferrite with precipitates of cementite) was obtained in the samples of ductile cast iron.
4. Austempering of ductile cast iron in hot bitumen resulted also to improved impact strength. The impact strength of ductile cast iron samples increased from 17J (as-cast) to 29J (austempered). It was

established that austenitization temperature influenced the impact strength of ductile cast iron. The highest impact energy (29J) was obtained at austenitization temperature of 850^0 C while the lowest impact energy (4J) was obtained at 950^0C. It was also established that at longer austempering times (3,4 and 5hours), there was a decrease in impact strength due to the formation of cementite (Fe_3C)

5. At all the austempering times, the impact energy of 0.55% C- steel was found to be higher than that of 0.75% C- steel.
6. From the results of tensile test, the austempering process resulted to increase in ductility of the ductile cast iron. However, for the 0.55% C and 0.75% C- steels, no conclusion could be drawn from the tensile test because the force needed to fracture the samples was beyond the capacity of the hounsfield tensometer used.

6.3 RECOMMENDATIONS

1. To be able to obtain more knowledge about the microstrcutures formed in the samples of steels and ductile cast iron austempered in hot bitumen (or any quenching medium), microscopes with higher resolution power such as scanning Electron microscope (SEM) and Transmission election microscope (TEM) should be used.

2. Further work should be done on the evaluation of the effects of hot bitumen on tensile properties of steel and ductile cast iron.

REFERENCES

Adegoke, O.S. (1980): "Geotechnical Investigations of the Ondo State Bituminous Sands, Vol. 1, Geology and Reserves estimates". Geological Consultancy Unit report, University of Ife, Ile-Ife, Nigeria.

Adewuyi, B.O. and Afonja, A.A. (2000): "Austempered Ductile Iron – A Viable alternative to steel". Nigerian Journal of Engineering and Management; Vol. 1. No 1; p 6 – 13.

Allen, D.K. (1969): " Metallurgy Theory and Practice". 1st edition; American Technical society, Chicago, p 270 – 290.

Anil, S. and Ronald, V.R. (2000): "Bitumen Performance in hot and arid climates". Pavement seminar for the middle east and north. Retrieved on 13/March/2002 from http://www.docs.vircoment.com/mobility/urban-forjordan.htm

Austempering Process Capacities. Retrieved on 18/Dec/2007 From; http://www.thermexmetal.com/-10k.

Austempered Ductile Iron: Quick facts, Retrieved on 18/Dec/2008 from; http://www.consoltech.com/sw.

Austempered Ductile Iron. Retrieved on 9/Jan/2004 from; http://www.ductile.org/didata/section4/4intro.htm.

Avner, S.H. (1984): "Introduction to Physical Metallurgy" 2nd edition, McGraw-Hill Book Co. Singapore; p 248 – 317.

American Society of Metals (1970)."The heating and Treating of Metals". p 353 – 371.

Amos (2002): " A Moment of Science" Retrieved on 2nd/ Sept/ 2002 From http://amosiindiana.edu/library/scrips/bitamen/htm.

Bahmani, M;, Elliot, R, and Varaham, N. (1997): " Austempered Ductile Iron: a competitive alternative to forged induction – Hardened steel crank

shafts". International Journal of Cast Metals Research Vol. 9. p 249 – 257.

Bayati, H. and Elliot, R. (1997): "Role of Asuterite in Promoting Ductility in an austempered ductile iron". Materials Science and Technology, Vol. 13, April; p 319 – 326.

Bulletin 102. Tempering (Drawing), Reprieved on 16/May/2001 from http://www.latrobesteel.com

Branka Bosnjak and Branko Radulovic (2004): "Effect of Austempering Temperature on Austempering Kinetics of Ni – Mo Alloyed Ductile Iron". Materials in Technologies, Vol. 38, No.6, p 307-312.

Dennis, W. H., (1963): " Metallurgy of the ferrous Metals" 1st Edition, Sir Isaac Pitman and Sons Ltd London. p 219 – 224; 230 – 260.

Donald, R. A. (1985): "The Science and Engineering of Materials." PWS Engineering, Bostom Massachusetts; p 263 – 265.

Elliot Roy (1988): "Cast iron Technology". 1st edition, Butterworths, London. p 136 – 149.

Gilbert, G.N.J. (1969): "The effect of heat treatment on Mechanical properties." Journal of Research and Development; British Cast Iron Research Association; Vol. 3, p 499 – 51.

Goni, M. Sheik (2003): "Bitumen Exploration and Development". Nigerian Society of Engineers, LAGELU, 2003. p 184 – 193.

Furnaces and Baths for Heating Steel. Retrieved on 6/Oct/2002 from http://www.newmex.com/ebear/metal/heattreat1.htm1.

Hamid Ali, S. and Elliot, R. (1997): "Influence of austenitizing temperature on austempering of an Mn-Mo-Cu alloyed ductile iron. Part 2- Mechanical Properties:' Matrials Science and technology, January, Vol. 13; p24– 30.

Harold, E. McGannon (1971): "The Making Shaping and Treating of Steel". 9th Edition, United States Steels (USS); p1086 – 1091.

Heat Treating of Nodular Irons: Part one and two retrieved on 9/Jan/2004 from http://www.key-to-steel.com/viewarticle.asp?Id=204

Heat Treating Practices Retrieved on 16/May/ 2001 from http://www.Macraesbluebook.com/search/company.CFM

Heat Treating of Cast Irons, Gray Irons, Ductile Irons, CFM Malleable Irons, High-Alloy Iron. Mechanical Engineering. Retrieved on 18/Dec/2007 from http://www.mechanicalenginer.de/latest/examples.newsitems-3-php.s

Imasogie, B.I, Ali, J. A and Afonja, A. A. (1991): "Cast Iron in the Light of recent research". Conference Proceedings of Metallurgical Society of Nigeria. p20 – 33.

Imasogie, B.I. Afonja, A.A. and Ali, J.A. (2000): " Properties of Ductile Cast iron Modularised with Multiple Calcium – Magnesium based Master alloy". Materials Science and Technology, Vol. 16, February; p194 – 201.

Juneja, R.H; Chakrabarti, A.K; Basak, A. and Bhattachanya, A. (1989): "Austempering Ductile iron with Copper and Magnanese". Foundary Management and Technology, Vol. 117 (2); p 64 – 67.

Kazerooni, R; Nazarboland, N. and Elliott, R. (1997): "Use of austenitizing temperature in control of austempering of an Mn – Mo – Cu alloyed ductile iron". Materials Science and Technology, Vol. 13; p1007 – 1015.

Khanna, O.P. (2002): " A textbook of Foundry Technology". 9th edition, Dhampart Rai Publications ltd. New Delhi; p431.

KRPC daily laboratory analysis record manual.

Lawrynowicz, Z. (2004): " Transition from Upper to Lower banite in Fe – C- Cr Steel". Materials Science and Technology. Vol. 20; p1447 – 1457.

Mallia, J. and Grech, M. (1997): " Effect of Silicon Content on Impact Properties of austempered ductile iron". Materials Science and Technology, Vol. 13; p 408 – 414.

Millis, K. D. (1972): "The future and development of spheroidal graphite Cast iron." Journal of British Foundryman; p 6 – 8.

Ming-Chang Jeng (1993): " Abrasive wear study of bainitic modular cast iron". Journal of Materials science and Technology, vol. 28, p6555 – 6561.

Pascoe, K. J. (1982): "An Introduction to the Properties of Engineering Materials". 2nd Edition, Van Nostrand Reinhold (UK) Co. Ltd, England; p 273 – 299.

Pennsylvania Metallurgical Incorporated Commercial Heat treaters: Austempering. Retrieved on 9/Jan/2004 from http://www.pmiheattreat.com/austempering.htm

Perry and Chilton (1973): "Chemical Engineers Handbook" 5th edition, McGraw Hill Book company, London. p 136.

Proper Quenching option yields better heat-treating results. Retrieved on 16/May/2001 from http://www.properquenchingoption yeilds.htm.

Rajan, T.V; Sharma, C.P. and Sharma, A. (1988): "Heat Treatment Principles and Technologies". 1st edition, Prentice-Hall of India, PL, New Delhi, p1 – 117.

Rajput, R.K. (2004): "Heat and Mass Transfer." 5th edition, s. Chaud & company Ltd; Ram Nagar, New Delhi; p1-10.

Rimmer Arron (1997): "ADI Is growing" Materials World: The Journal of Institute of Materials, London; Vol. 5, No. 5; p252 – 255.

SAE Technical Papers: Austempered Ductile Iron; Possible Usage for Parts in the Trucking Industry. Retrieved on 18/dec/2007 from http://www.sae.org

Singh, U. P; Roy. B; Iha, S; and Bhattacharya, S.K. (2001): "Microstructure and Mechanical Properies of as rolled high strength bainitic rail steels". Materials Science and Technology, Vol. 17; p 33 – 38.

Smith, W. (1981): "Structure and Properties of Engineering alloys." Butter worth, London. p 315 – 340.

Speight, J. G. (1992): "Asphalt in – Kirk Othmer Encyclopedia of Chemical Technology, 4th edition, vol. 3, New York, NY, John Wiley and Sons. p 689 – 724.

Taylor Edward (1974): "Operating Salt baths property". Metal Progress. p87–90.

Usmani, A.M. (1997): "Asphalt Science and Technology". 1st edition, Marcel Dekkerine, New York. p 249 – 258.

Walton, C.F. (1971): "Gray and Ductile Iron Casting handbook". Gray and Ductile Iron Foundries Society, Cleveland; p 239 – 249, 392 – 408.

What is Austempering? Retrieved on 9/Jan/2004 from http://www.appliedprocess.com/austemparing/.

Wilson, T. (1975): "Metallurgy and heat treatment of tool steels." 1st edition McGraw Hill Book Company, London. p34 – 94.

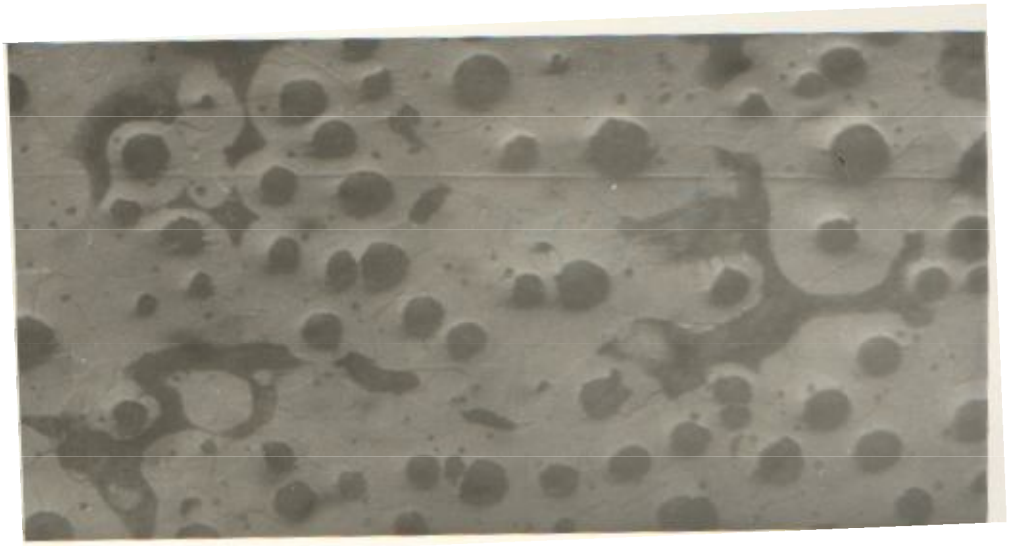
World Health Organization, Geneva (2004): " Concise International Assessment Report; document 59 on Asphalt (Bitumen).

Yazdani, S. and Elliott R. (1999): " Influence of Molybdenum on Austempering behaviour of ductile iron". Part 1 – Austempering kinetics of Ductile Iron Containing 0.13% Mo. Materials Science and Technology, vol. 15; p 531 – 50.

Yescas – Gonzalez, M. A; Bhadeshia, H.K.D. and Mackay, D.J.C (2000). "Estimation of the amount of retained austemte in austempered ductile irons". Materials Science and Engineering A, vol. A311; p162-173.

Zahiri, S.H; Pereloma, E.V. and Davis, C.H.J. (2001): "Application of banite transformation Model to estimation of Processing window boundaries for Mn – Mo – Cu austempered ductile iron". Materials Science and Technology, Vol. 15; p233 – 240.

Appendix A



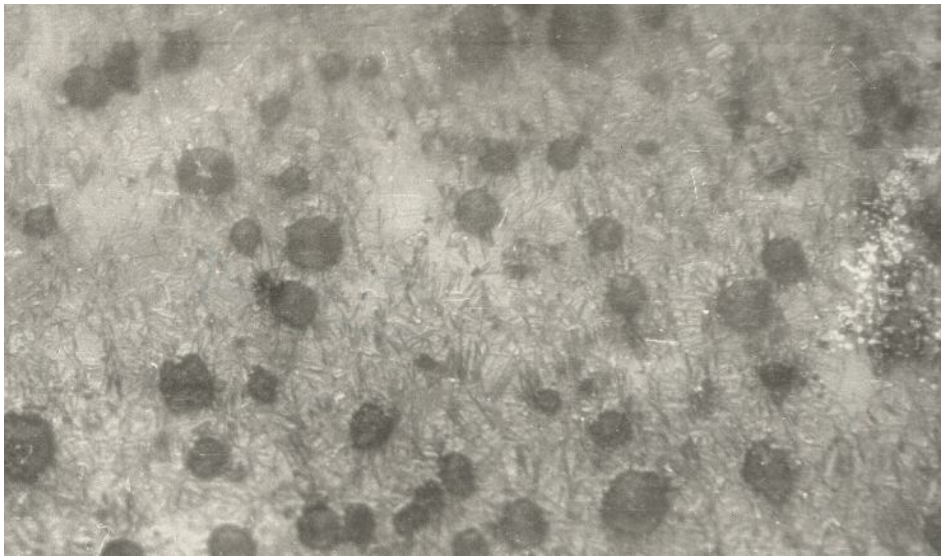
Micrograph A1: As-cast structure of ductile cast iron showing ferrite (white) surrounding graphite nodules in pearlite matrix; (x100).



Micrograph A2: As-received structure 0.55% C-steel showing grain boundary ferrite (white) in the matrix of pearlite (dark); (x100).



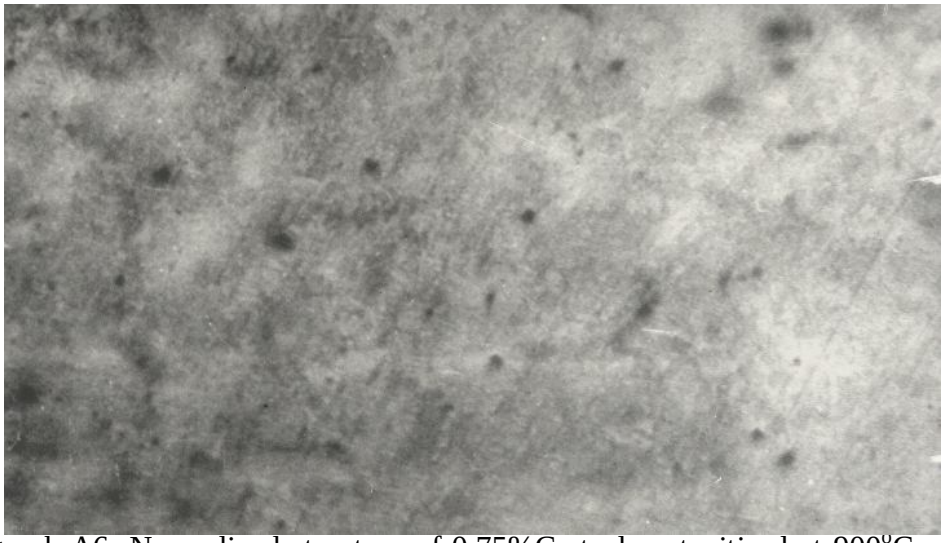
Micrograph A3: As-received structure of 0.75%C-steel showing grain boundary ferrite (white) in the matrix of pearlite (dark); (x100).



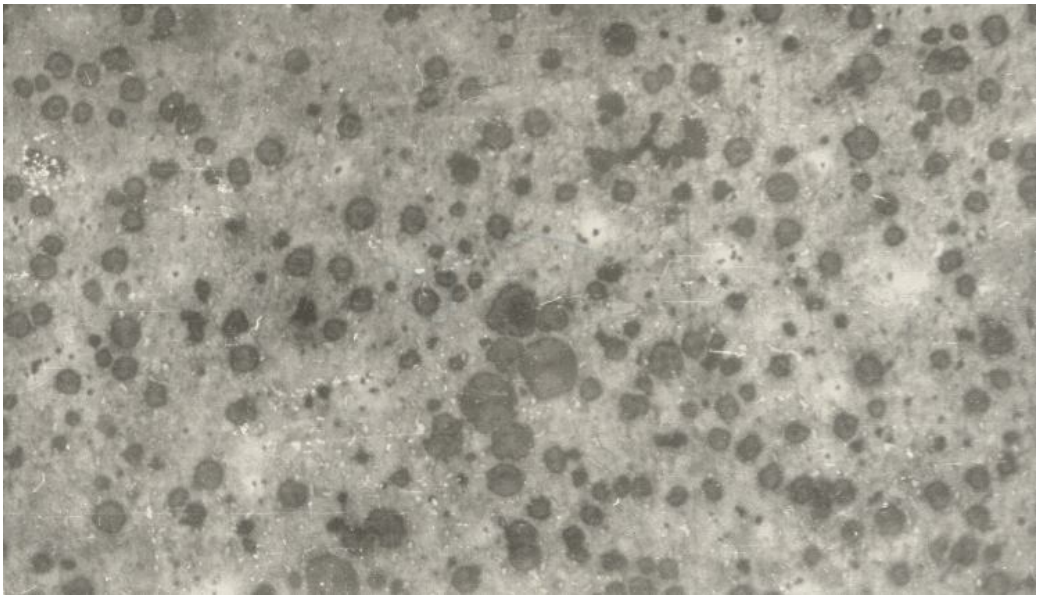
Micrograph A4: Normalized structure of ductile cast iron austenitised at 900^{o}C; Showing graphite nodules in pearlite matrix; (x100).



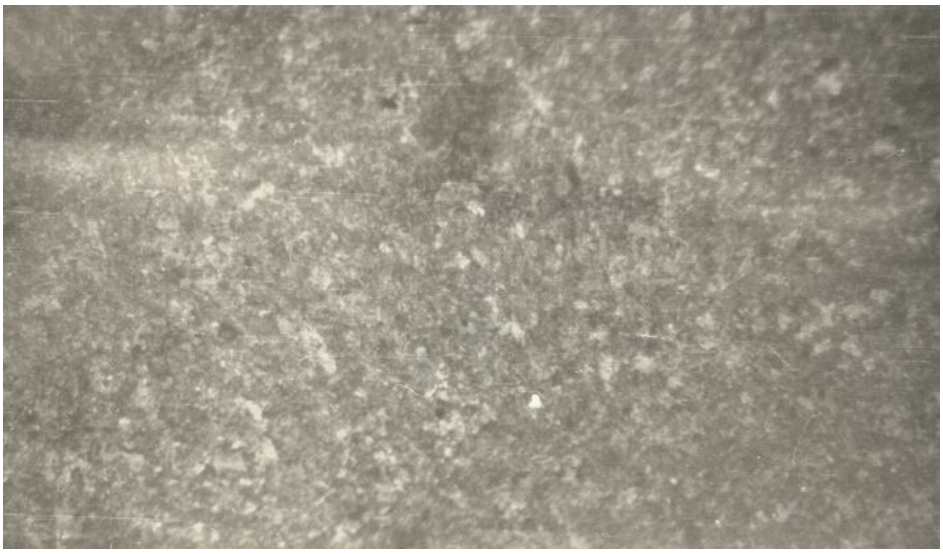
Micrograph A5: Normalized structure of 0.55%C-steel austenitised at 900^{o}C, showing ferrite (white) plus pearlite (dark); (x100)



Micrograph A6: Normalized structure of 0.75%C-steel austenitised at 900^{o}C, showing pearlite structure; (x100).



Micrograph A7: Water quenched structure of ductile cast iron, showing martensite structure; (x100).



Micrograph A8: Water quenched structure of 0.55% C-steel showing martensite (x100).



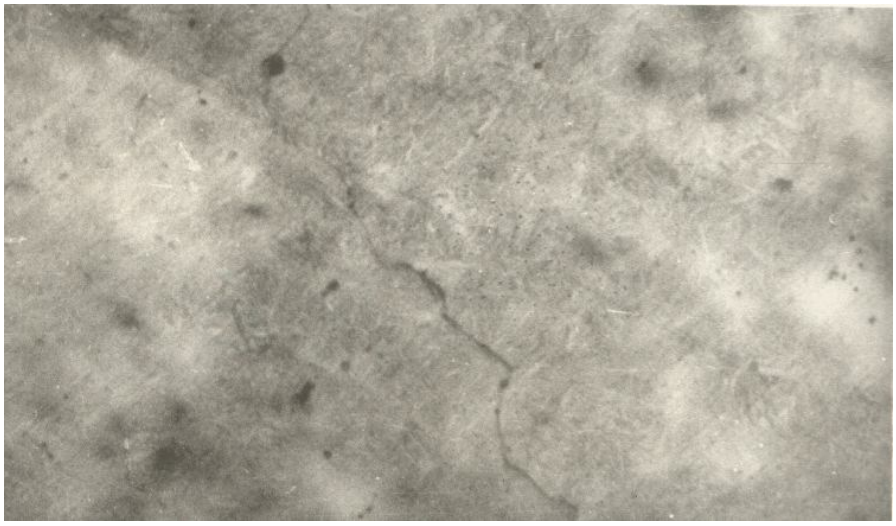
Micrograph A9: Water quenched structure of 0.75%C-steel austenitised at 900^{o}C, showing martensite and cracks due to transformation stresses; (x100).



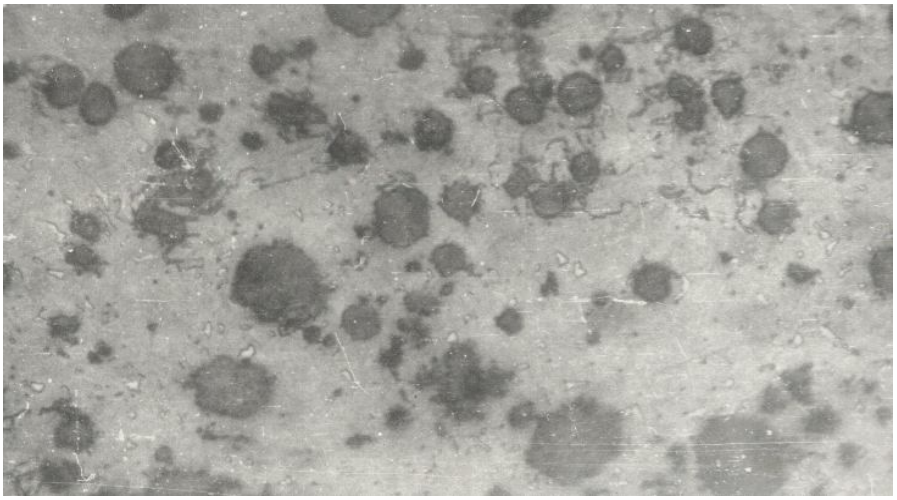
Micrograph A10: Bitumen quenched structure of ductile cast iron austenitised at 900^{o}C. This structure shows martensite with some quantity of retained austenite (x100).



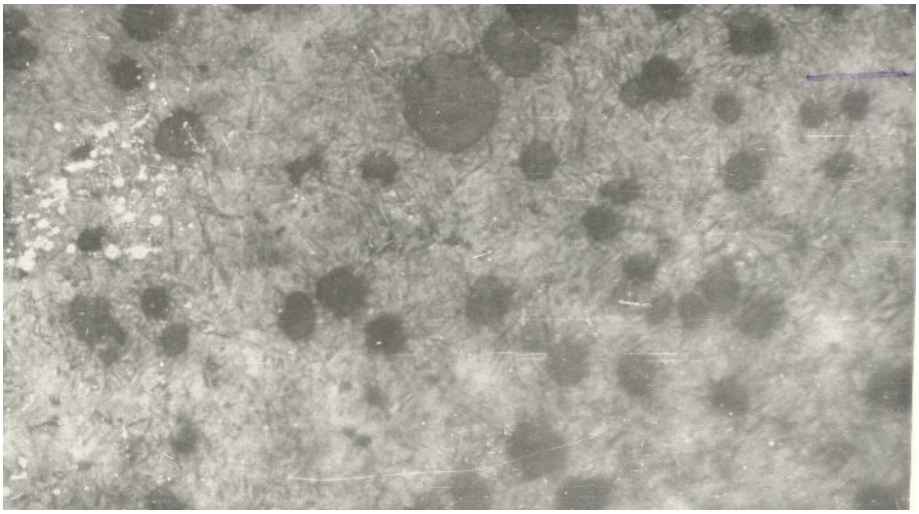
Micrograph A11: bitumen quenched structure of 0.55% C-steel austenitised at 900^{o}C. This structure reveals matensite structure plus some quantity of retained austenite (white); (x100).



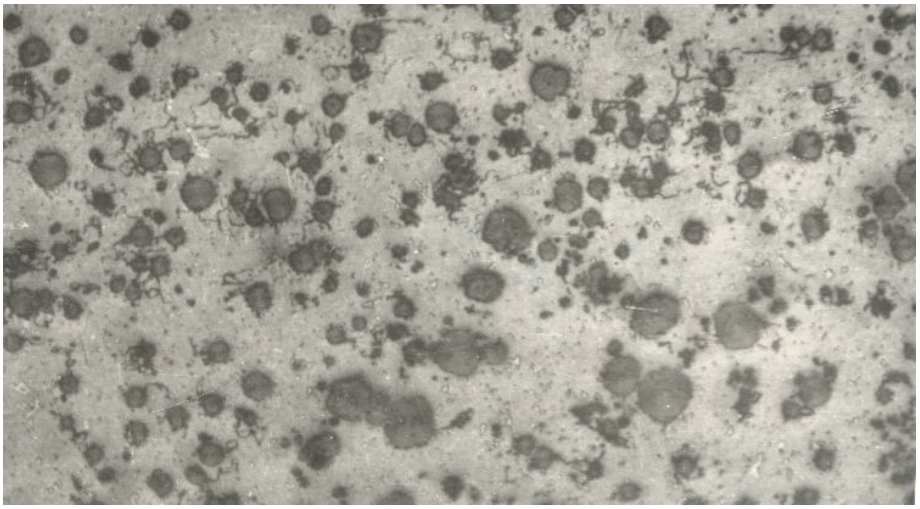
Micrograph A12: Bitumen quenched structure of 0.75% C-steel austenitised at 900^{o}C. This structure shows martensite plus some quantity of retained austete; (x100).



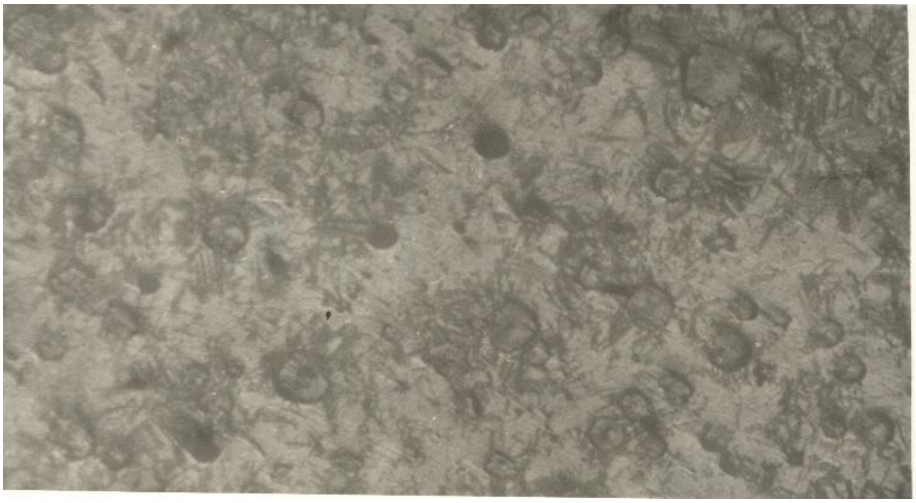
Micrograph A13: Austempered structure of ductile cast iron austenitised at 900^{o}C and austempered at 350^{o}C for 5minutes. The structure shows a mixture of martensite and retained austenite; (x100).



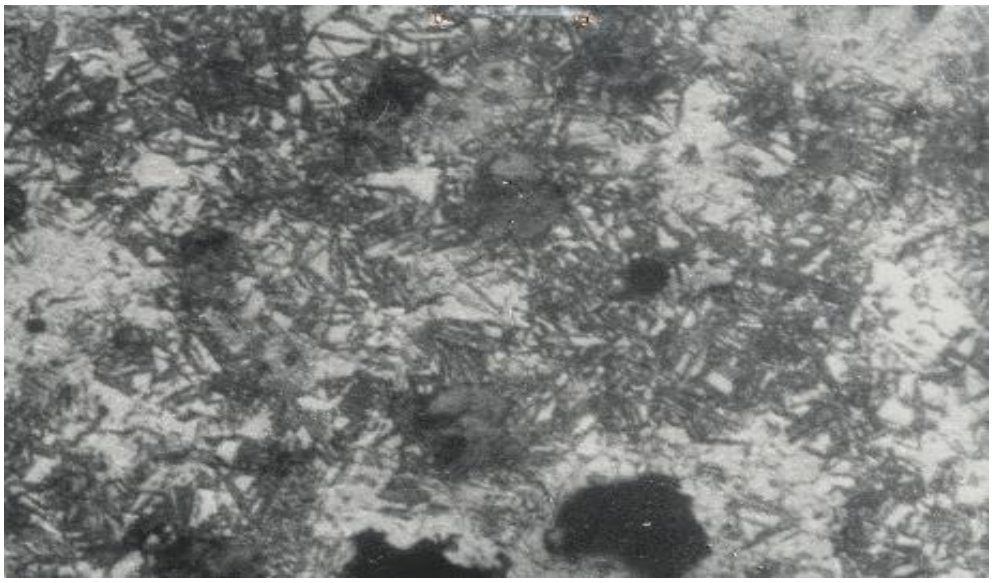
Micrograph A14: Austempered structure of ductile cast iron austenitised at 900^{o}C austempered at 350^{o}C for 30minutes. This structure shows a mixture of martensite and retained austenite; (x100)



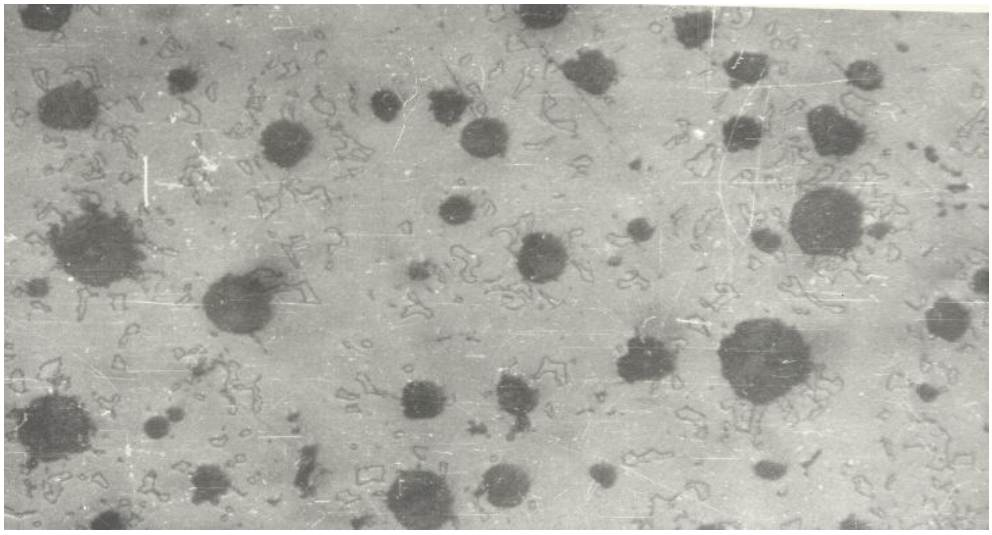
Micrograph A15: Structure of ductile cast iron austenitised at 900^{o}C and austemperd at 350^{o}C for 1hour. The strructre shows ausferrite structure and retained austenite; (x100).



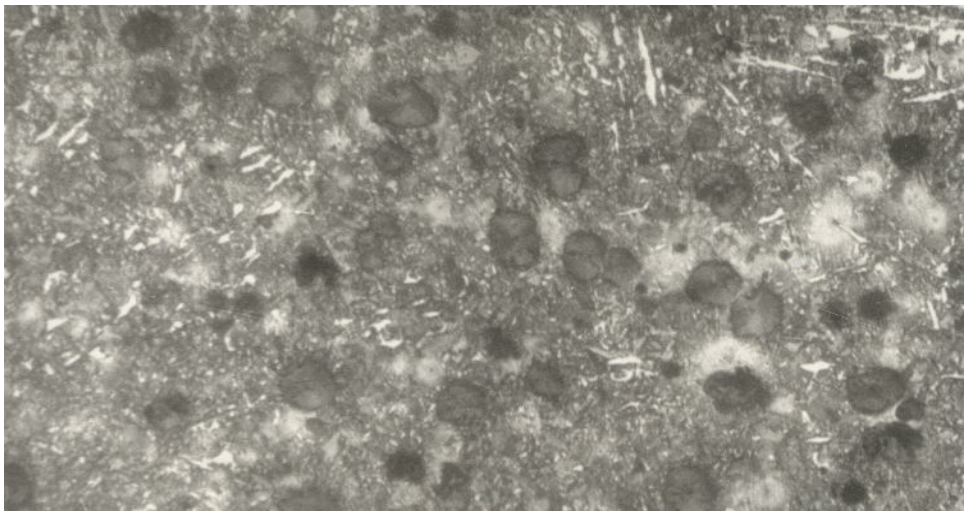
Micrograph 16: Structure of ductile cast iron austenitised at 900^{o}C and austempered at 350^{o}C for 2hours showing ausferrite structure (Bainite); (x100).



Micrograph A17: Structure of ductile cast iron austentised at 900^{o}C and austempered at 350^{o}C for 3hours. The structure shows ausferrite (bainite); (x100).



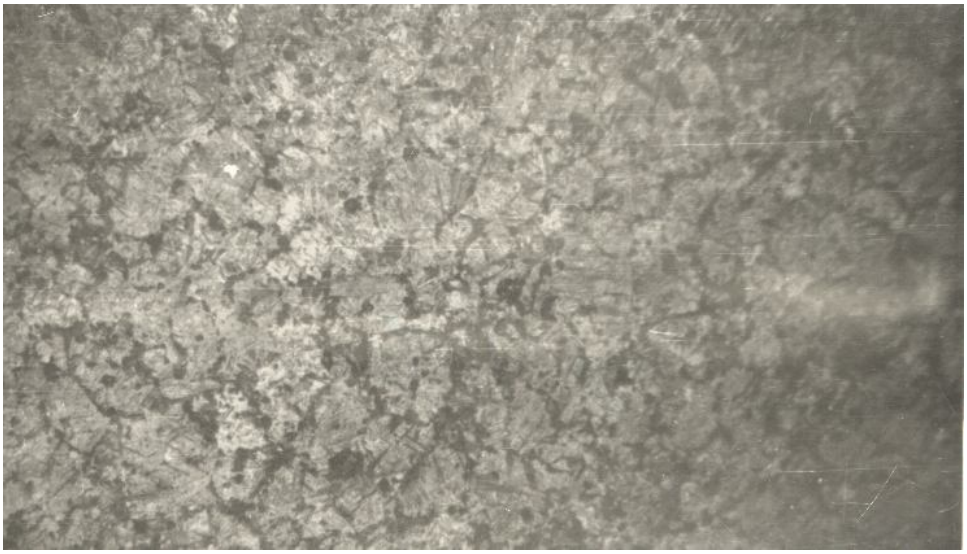
Micrograph A18: Structure of ductile cast iron austenitised at 900^{o}C and austempered at 350^{o}C for 4hours. This structure shows bainite plus cementite (white); (x100).



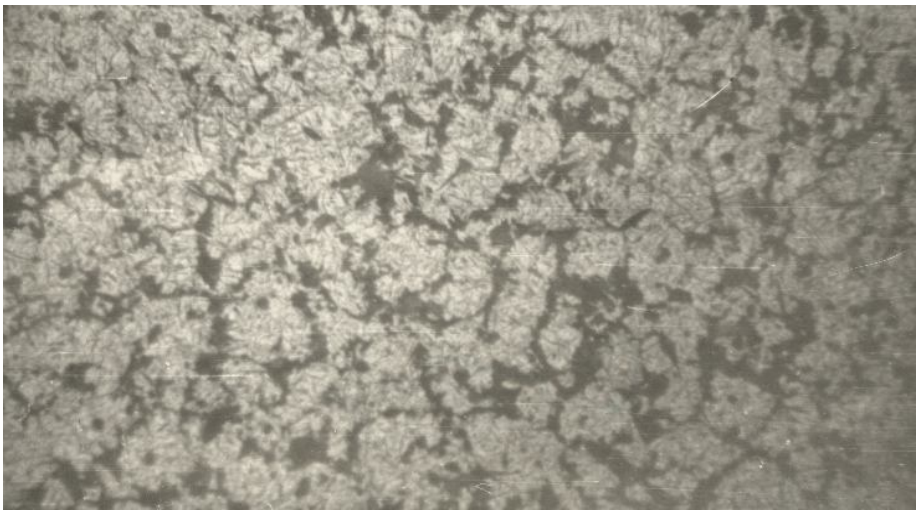
Micrograph A19: Structure of ductile cast iron austenitised at 900^{o}C and austemperd at 350^{o}C for 5hours showing bainite structure plus cementite (white); (x100)



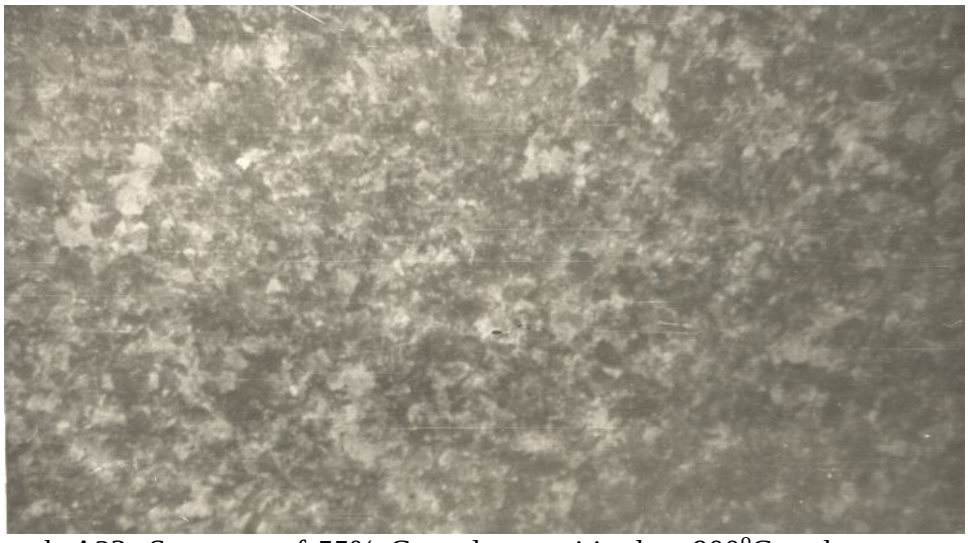
Micrograph A20: Structure of 0.55% C-steel austenitised at 900^{o}C and austempered at 350^{o}C for 5minutes. This structure shows mixture of martensite and retained austenite; (x100).



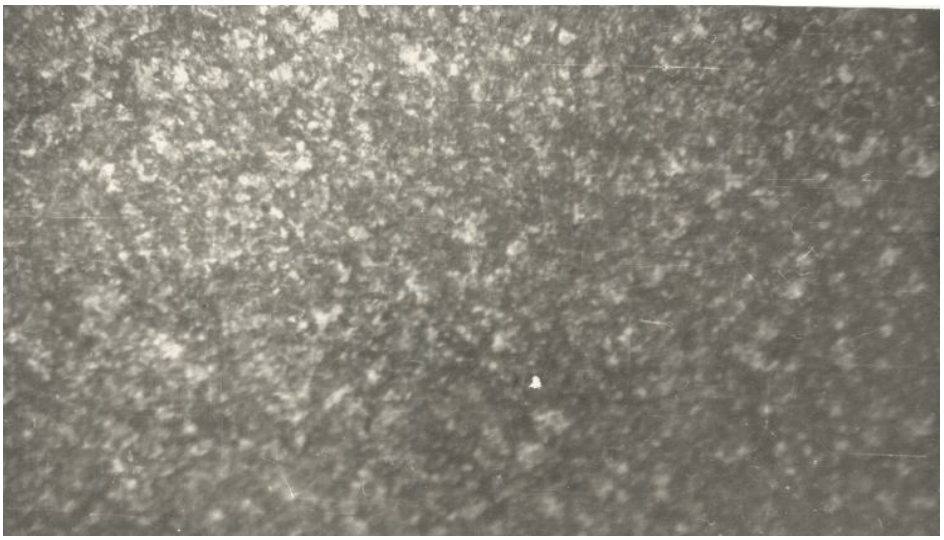
Micrograph A21: Structure of 0.55% C-steel austenitised at 900^{o}C and austempered at 350^{o}C for 30minutes. This structure shows mixture of martensite and retained austenite; (x100).



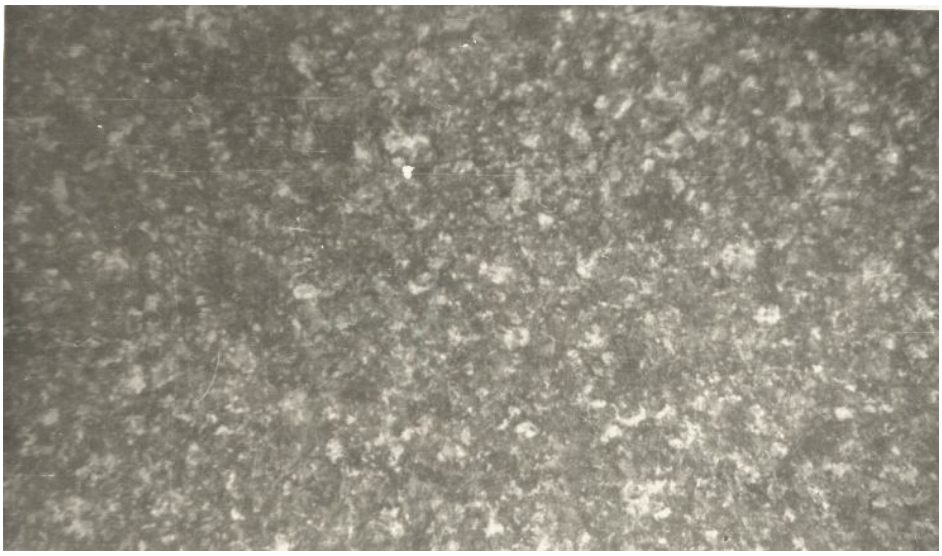
Micrograph A22: Structure of 0.55% C-steel austenitised at 900^{o}C and austempered at 350^{o}C for 1hour. This structure shows a mixture of martensite plus bainite and retained austenite; (x100).



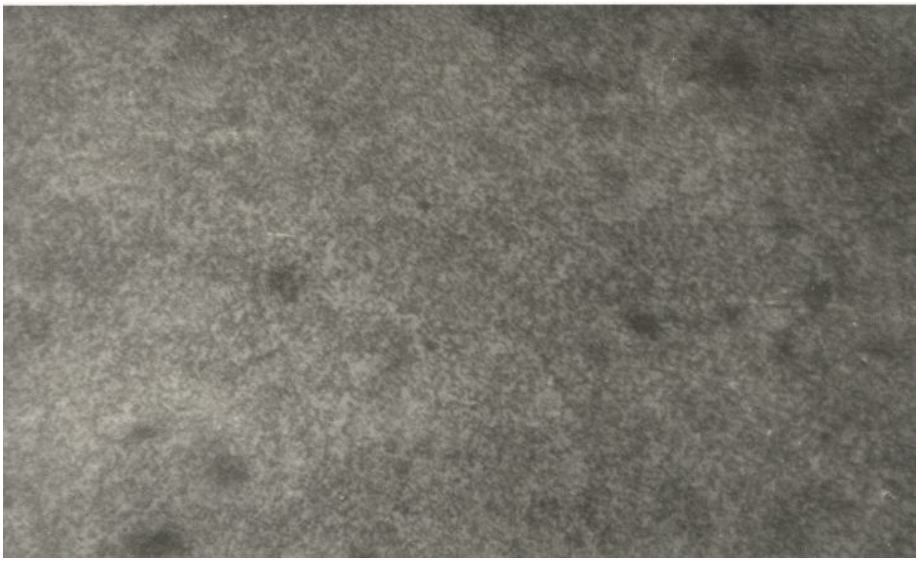
Micrograph A23: Structure of 55% C-steel austenitised at 900^{o}C and austempered at 350^{o}C for 2hours. This structure shows a mixture of martensite plus bainite and retained austenite (white); (x100).



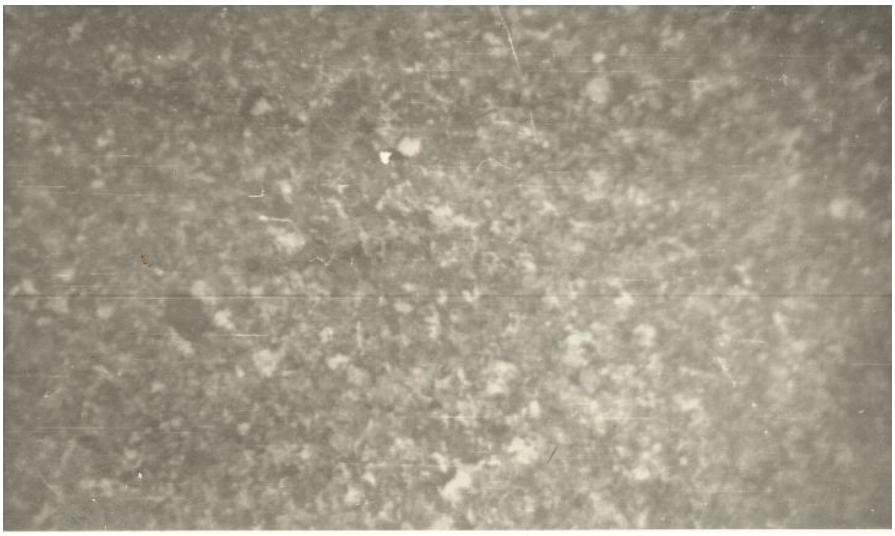
Micrograph A24: Structure of 0.55% C-steel austenitised at 900^{o}C and austempered at 350^{o}C for 3hours. This structure shows a mixture of martensite plus bainite and retained austenite; (x100).



Micrograph A25: Structure of 0.55% C-steel austenitised at 900^{o}C and austempered at 350^{o}C for 4hours. This structure shows mixture of stable austenite and bainite structure; (x100).



Micrograph A26: Structure of 0.55% C-steel austenitised at 900^{o}C and austempered at 350^{o}C for 5hours. This structure shows a mixture of stable austenite and banite; (x100).



Micrograph A27: Structure of 0.75% C-steel austenitised at 900^{o}C and austempered at 350^{o}C for 30minutes. This structure shows a mixture of martensite and retained austenite; (x100).



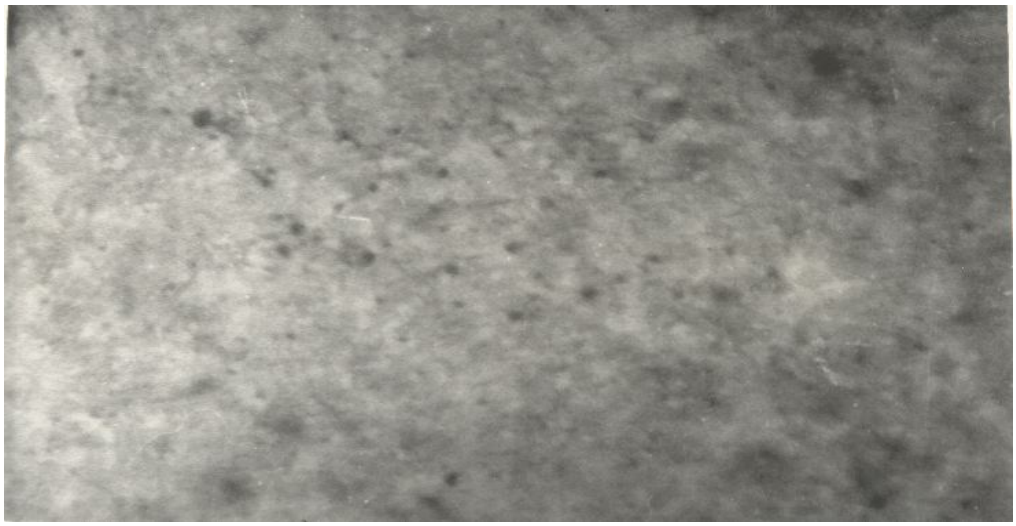
Micrograph A28: Structure of 0.75% C-steel austenitised at 900^{o}C and austempered at 350^{o}C for 1hours. This structure shows a mixture of martensite and bainite; (x100).



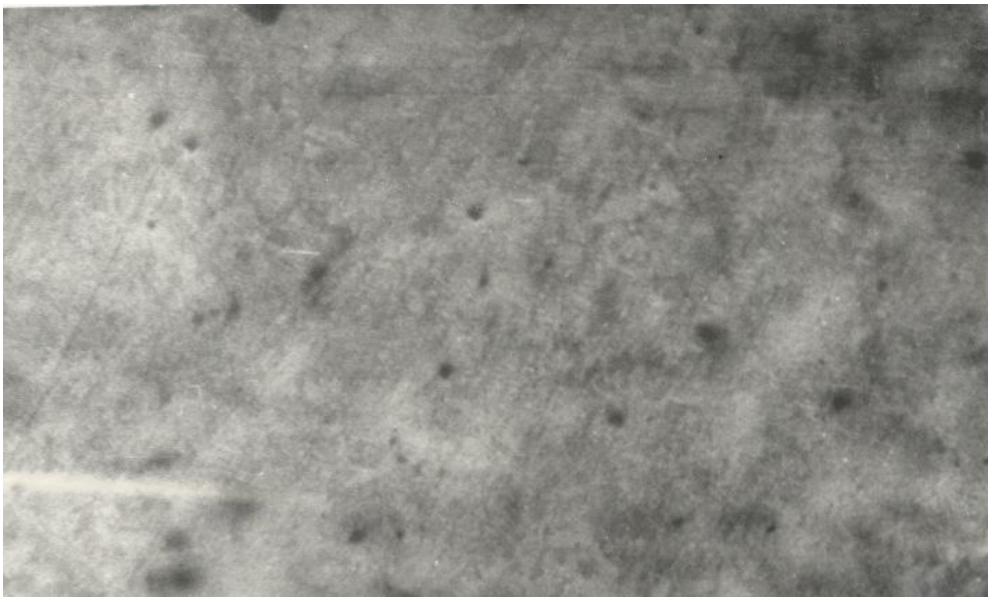
Micrograph A29: Structure of 0.75% C-steel austenitised at 900^{o}C and austempered at 350^{o}C for 2hours. This structure shows a fine structure suspected to be cementite precipitates in ferrite; (x100).



Micrograph A30: Structure of 0.75% C-steel austenitised at 900^{o}C and austempered at 350^{o}C for 3hours. This structure shows a fine structure suspected to be cementite precipitates in ferrite; (x100).



Micrograph 31: Structure of 0.75% C-steel austenitised at 900^{o}C and austempered at 350^{o}C for 4hours. This structure shows a mixture of bainite and retained austenite; (x100).



Micrograph A32: Structure of 0.75% C-steel austenitised at 900^{o}C and austempered at 350^{o}C for 5hours. This structure shows a mixture of bainite and retained austenite; (x100).

APPENDIX B.

Table B1: Results of Hardness and Izod impact tests of as-received and normalized samples.

Grade of Alloy	Hardness (HV)		Izol Impact (J)	
	As-received	Normalized	As- received	Normalized
0.55% C-steel	200	295	55	51
0.75% C- Steel	260	335	28	26
Ductile Cast iron	148	180	17	20

Table B2: Results of hardness and izod impact test of samples quenched in water and tempered at 350^0C.

Grade of alloy	Hardness (HV)		Izol Impact (J)	
	Water Quenched	Tempered	Water quenched	tempered
0.55% C-steel	645	410	3	5
0.75% C- Steel	780	490	3	4
Ductile Cast iron	475	451	3	5

Table B3: Results of hardness and izod impact tests of samples quenched in bitumen at room temperature and tempered at 350^0 C.

Grade of alloy	Hardness (HV)		Izol Impact (J)	
	Water Quenched	Tempered	Water quenched	tempered
0.55% C-steel	553	485	6	8
0.75% C- Steel	620	546	4	5
Ductile Cast iron	418	315	4	5

Table B4: Results of hardness and izod impact test of Ductile cast iron samples austenitised at 900^0C and austempered in hot bitum (350^0C) for 5 mins- hours.

Austemper time	Hardness (HV)	Izod impact (J)
5 minutes	468	7
30 mins	410	12
1hr	382	15
2hrs	360	25
3hrs	380	18
4hrs	365	19
5hrs	381	18
As-quenched	475	3

Table B5: Results of Hardness and izod impact test of 0.55% C- steel austenitised at 900^0 C) and austempered in hot bitumen (350^0C) for 5 mins – 5 hours.

Austemper time	Hardness (HV)	Izod impact (J)
5 minutes	460	13
30 mins	410	18
1hr	382	21
2hrs	370	22
3hrs	342	37
4hrs	409	33
5hrs	430	29
As-quenched	645	3

Table B6: Results of Hardness and izod impact test of 0.55% C- steel austenitised at 900^0 C) and austempered in hot bitumen (350^0C) for 5 mins – 5 hours.

Austemper time	Hardness (HV)	Izod impact (J)
5 minutes	586	11
30 mins	550	13
1hr	520	18
2hrs	540	17
3hrs	548	17
4hrs	520	16
5hrs	480	24
As-quenched	780	3

Table B7: Results of Hardness test on ductile cast iron samples austenitised at 800,850,900 & 950^{0}C and austempered in hot bitumen (350^{0}C) for 5 minutes – 5 hours.

	Hardness values (HV)			
Austemper time	800^0 C	850^0 C	900^{0}C	950^{0}C
5 minutes	150	415	468	510
30 mins	148	345	410	470
1hr	148	305	382	425
2hrs	140	342	360	390
3hrs	140	335	380	405
4hrs	140	330	365	410
5hrs	130	330	381	400
As-quenched	150	420	468	520

Table B8: Results of impact test (J) on ductile cast iron samples autenitised at 800^0C, 850^0C, 900^0 and 950^0 C and austempered in hot bitumen (350^0C) for 5 mins – 5 hours.

	Izod impact strength (J)			
Austemper time	800^0 C	850^0 C	900^0C	950^0C
5 minutes	19	10	7	3
30 mins	19	16	12	4
1hr	21	29	15	7
2hrs	19	27	25	11
3hrs	21	24	18	8
4hrs	20	22	19	4
5hrs	20	21	18	4
As-quenched	20	10	6	3

Table B9: Results of Tensile test conducted on Ductile cast iron samples.

Sample condition/Treatment	Tensile properties		
	T. Strength (N/mm^2)	% elongation	% Reduction in Area
As- Cast	789	2	5
Normalised	811	8	10
Water quench	153	0	0
Water Quench & temper	299	0	0
Austemper for 5 minutes	458	1	0
Austemper for 30 mins	611	2	4
Austemper for 1 hours	815	9	15
Austemper for 2 hours	815	10	15
Austemper for 3 hours	Above max	10	14
Austemper for 4 hrs	“	7	10
Austemper for 5 hrs	“	6	10

Table B10: Results of Tensile test conducted on 0.55 % C- Steel Samples.

Sample condition/Treatment	Tensile properties		
	T. Strength (N/mm^2)	% elongation	% Reduction in Area
As- received	713	15	40
Normalised	815	8	12
Water quench	102	0	0
Water Quench & temper	204	1	0
Austemper for 5 minutes	255	1	0
Austemper for 30 mins	509	5	2
Austemper for 1 hours	Above Max	0.5	0
Austemper for 2 hours	″	0	0
Austemper for 3 hours	″	0	0
Austemper for 4 hrs	″	0	0
Austemper for 5 hrs	″	0	0

Table B11: Result of Tensile test conducted on 0.75% C- Steel sample

Sample condition/Treatment	Tensile properties		
	T. Strength (N/mm^2)	% elongation	% Reduction in Area
As- received	764	8	10
Normalised	1018	12	30
Water quench	51	0	0
Water Quench & temper	306	0.5	0
Austemper for 5 minutes	407	0.5	0
Austemper for 30 mins	Above max.	0	0
Austemper for 1 hours	″	0	0
Austemper for 2 hours	″	0	0
Austemper for 3 hours	″	0	0
Austemper for 4 hrs	″	0	0
Austemper for 5 hrs	″	0	0

APPENDIX C

HEAT ANALYSIS

Heat transfer may be defined as: “The transmission of energy from one region to another as a result of temperature gradient”. In heat transfer, the driving potential is temperature difference (Rajput, 2004).

In almost every branch of engineering, heat transfer (and mass transfer) problems are encountered. Heat treatment of metals/alloys is one of the engineering areas covered under the descipline of heat transfer.

According to Rajput (2004), the energy in transit is termed heat; and that according to modern theory of heat, “Heat is a form of energy”. Any theory of heat should be concerned with the following facts.

i. Whenever there is an exchange of heat, heat is consumed (heat lost by the hot body always equal to heat gained by the cold body).
ii. The heat flow takes place from higher temperature to lower temperature.
iii. The substances expand on heating
iv. In order to change the state of a body from solid to liquid or liquid to gas without rise in temperature, certain amount of heat is required.

v. When a body is heated or cooled, its weight does not change (Rajput, 2004).

The study of heat transfer is carried out for the following purposes:

1. To estimate the rate of flow of energy as heat through the boundaries of a system under study (both under steady and transient condition).
2. To determine the temperature field.

MODES OF HEAT TRANSFER

Heat transfer, which is defined as the transmission of energy from one region to another as a result of temperature gradient takes place by the following three modes:

i. Conduction
ii. Convection
iii. Radiation

Heat always flows in the direction of lower temperature. It is also important to note that the above three modes of heat transfer are similar in that a temperature differential must exist and the heat exchange is usually in the direction of decreasing temperature; each method however, has different controlling laws.

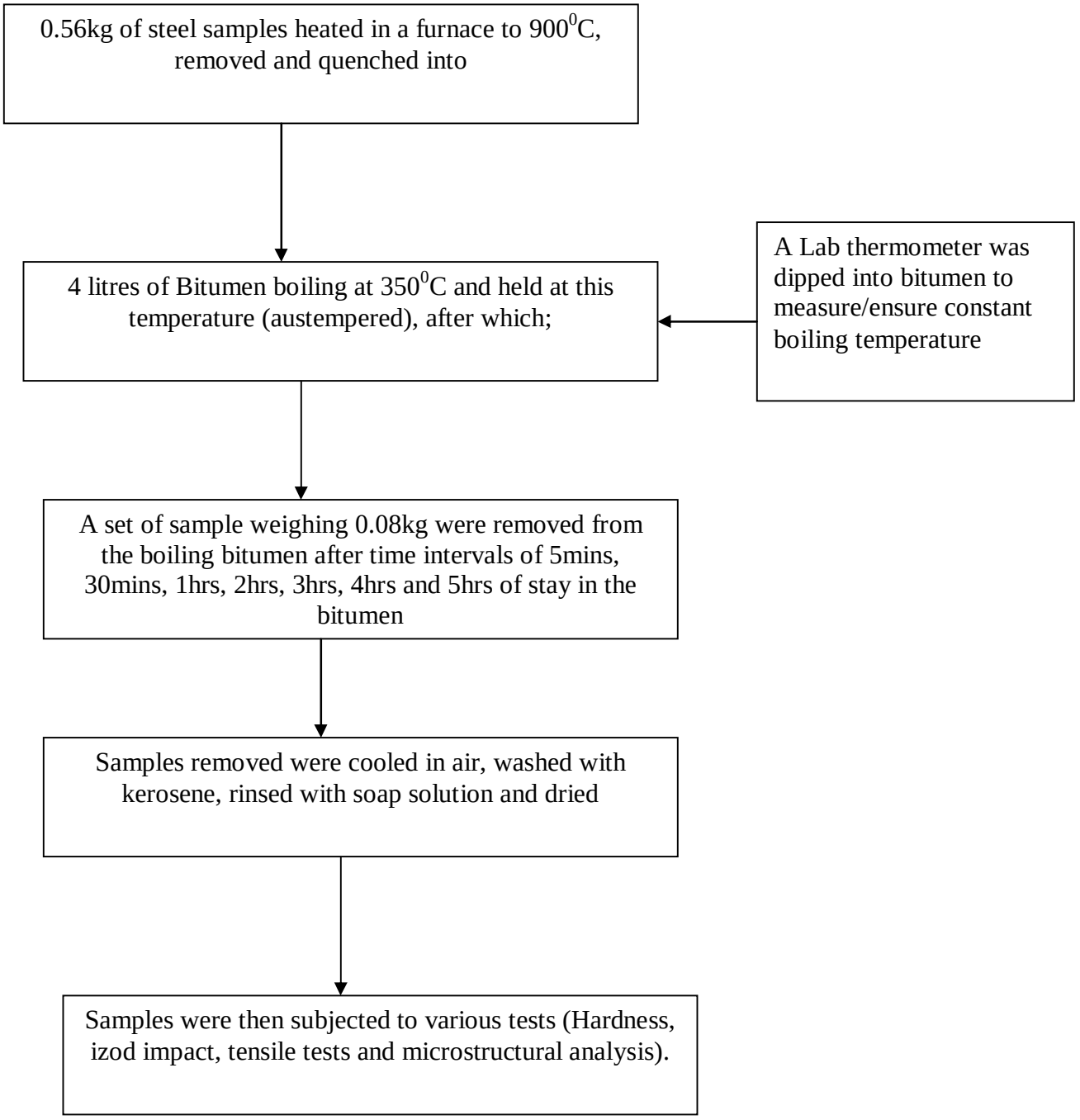


Figure: C1: Flow chart for the austempering heat treatment process carried out in hot bitumen bath at 350^{0}C

THE HEAT ANALYSIS OF AUSTEMPERED SAMPLES IN HOT BITUMEN BATH AT 350^0C

The flow chart in figure C_1 shows the steps followed during the austempering process using hot bitumen bath at 350^0C.

The following parameters were used for heat analysis.

- Volume of bitumen used per one cycle of heat treatment = 4 litres = $4000Cm^3 = 0.004M^3$
- Mass of bitumen = 2.0Kg
- Total mass of steel samples removed from furnance at 900^0C and quenched into hot bitumen bath at 350^0C = 0.56Kg.
- Total mass of samples removed from hot bitumen at intervals after austempering = 0.08Kg
- Specific heat capacity of bitumen = $0.22Cal/g/^0C$
- Specific heat capacity of steel = $0.12Cal/g/^0C$ (Perry and Chilton; 1973).
- Density of bitumen = $1.5Kg/M^3$
- Density of steel = $7.6Kg/m^3$

In the process of heat flow assuming no heat loses to the air, then

Heat lost by hot body = Heat gained by cold body →(1)

(a) The heat content of the samples removed from the furnance at 900^0C

$\Delta Hs = Ms\ Cs\Delta ts$ ……(2)

ΔHs = quantity of heat contained by samples removed from furnance at 900^0C.

Ms = Mass of the samples removed from furnance at 900^0C (= 0.56Kg)

Cs = specific heat capacity of samples ($0.12Cal/g/^0C$)

Δts = change in temperature of samples ($t_f - t_0$) where t_f is temperature in the furnance = 900^0C, while t_0 is the reference temperature (room temperature) = 30^0C.

Therefore, from equation (2)

$\Delta Hs = Ms\ Cs\ \Delta ts$

$= 0.56 \times 0.12 \times (900 - 30)$

$= 0.0672 \times 870$

ΔHs = 58.454 Calories (this is the quantity of heat contained by the samples removed from the furnance at 900^0C to be quenched into hot bitumen.

(b) The heat content of bitumen at 350^0C

$\Delta H_b = M_b\ C_b\ \Delta t_b$ -→ (3), where

ΔH_b = quantity of heat in bitumen at 350^0C

M_b = Mass of bitumen used for the austempering heat treatment (2kg)

Cb = Specific heat capacity of bitumen (0.22)

Δt_b = Change in temperature of bitumen ($t_b - t_0$) from room temperature, t_0 (30^0C) to boiling temperature, t_b (350^0C)

From equation (3)

$\Delta H_b = M_b C_b \Delta t_b$

$= 2 \times 0.22 \times (350 - 30)$

$= 0.4 \times 320$

ΔH_b = 149.8 Cal (this is the quantity of heat contained by hot bitumen boiling at 350^0C).

It was observed during experiment that when the samples were removed from the furnance at 900^0C and quenched into hot bitumen at 350^0C the temperature of the bitumen rose suddenly to 358^0C but dropped back after 3 minutes and remained steady thereafter at 350^0C. More evaporation was noticed during the three minutes intervals.

Before the temperature dropped back to 350^0, the total heat content in the pot at 358^0C was

$\Delta H_s + \Delta H_b$ (i.e equation(2) + (3)

58.454 + 149.8

208.254 Cal

When the temperature dropped back to 350^0C after three minutes, the total heat content = $M_b C_b \Delta t_b + M_s C_s \Delta t_s$ ……. (5)

Where,

M_b = Mass of bitumen at 350^0C

C_b = specific heat capacity of bitumen

Δt_b = temperature of boiling bitumen which was constants at 350^0C

Therefore from equation (5),

The total Energy (at 350) $= M_b C_b \Delta t_b + M_s C_s \Delta t_s$

$= (2x\ 0.22\ x\ 350) + (0.56\ x\ 0.12\ x\ 350)$

$= 154 + 23.52$

$= \underline{177.52Cal}$

The energy (heat) loss to vaporization may be assumed to be :

Equation (4) minus equation (5)

→ 208.254 – 177.52

30.734 cal

The heat supply from the source (kerosene stove) was kept constant and continuous and the temperature of boiling bitumen was constant from this stage to the end of the austempering period (5hrs).

(i) After 5 minutes, a set of samples with mass of 0.08kg were removed from hot bitumen bath and cooled in air. Therefore the heat content of these samples removed is = $MC\Delta t$ …….. (6)

= 0.08 x 0.12 x 350 – 30

= 9.6 x 320

= 3.072 Cal.

(ii) The total heat content left after the removal of the first at of samples becomes

Equation (5) minus equation (6)

177.52 – 3.072

= 174.48Cal.

(iii) After 30 minutes, 1hr, 2hrs, 3hrs, 4hrs and 5hrs another set of samples of the same mass (0.08kg) were removed from the hot bitumen bath respectively.

APPENDIX D

MICROSTRUCTURES OF AUSTEMPERED DUCTILE CAST IRON OBTAINED BY OTHER RESEARCHERS USING MOLTEN SALT BATH.

The microstructure and composition of a metal/alloy are among the major factors that determine the subsequent mechanical properties. A change in microstructure usually results to change in mechanical properties.

The microstructures shown below in plates D1–D5 are those obtained by various researchers that used molten salt bath as austempering medium as reported in literature. It can be seen that in all the microstructures shown in these plates (D1–D5), ausferrite structure (bainite) is evident. The feathery morphology of ferrite laths with carbide precipitates which is the characteristic of bainite is present in the microstructures.

Comparing those microstructures of the samples austempered in hot bitumen bath with those austempered using molten salt bath (as shown in Appendix D; plates D_1 – D5) shows that the microstructures looks similar (there is close resemblance). The feathery structure of ferrite lath which is one of the distinguishing characteristics of bainite structure is seen in both austempered structures. This indicates that the hot bitumen bath used for austempering was able to cause the formation of bainite structure in the ductile cast iron samples austempered.

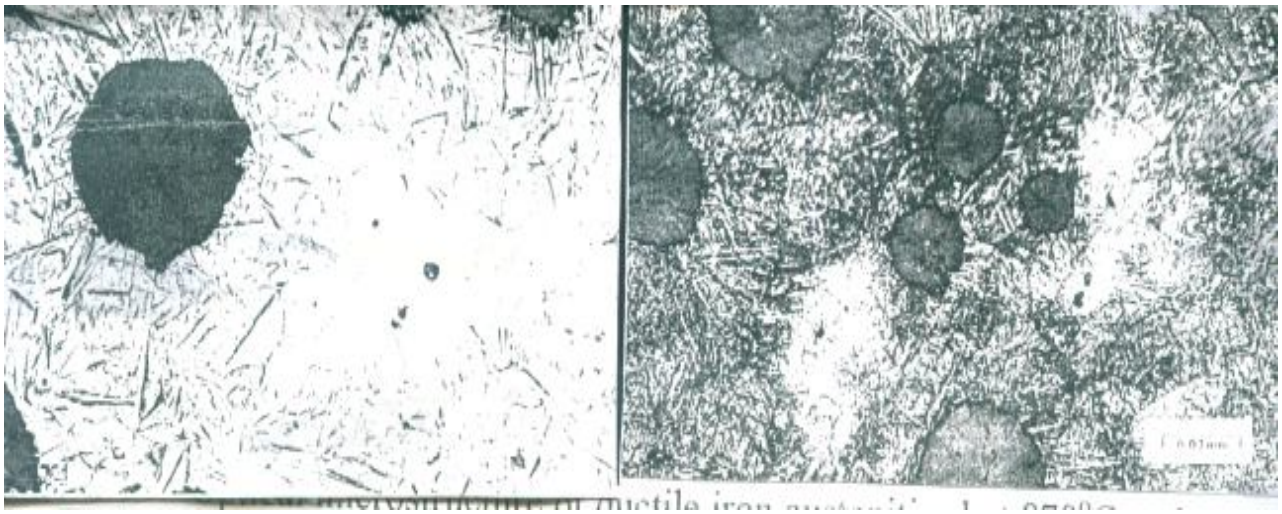
a  b

Plate D1(a & b): Optical microstructure of ductile iron austenitised at 870^0C and austempered in a salt bath furnane containing molten cassel TS220 (KN_{03} + $NaNo_3$ salt held at 400^oC of various times, then removed and air cooled as reported by Yazdani and Elliot (1999).



Plate D2: Optical microstructure of Mn – Mo – Cu alloyed ductile iron asutenitised at 880^oC and austempered in salt bath furnance containing cassel Ts220 ($KN0_3$ + $NaN0_3$) salt selt at 375^0C, then removed and cooled in air, as reported by Kazerooni et al, (1997).

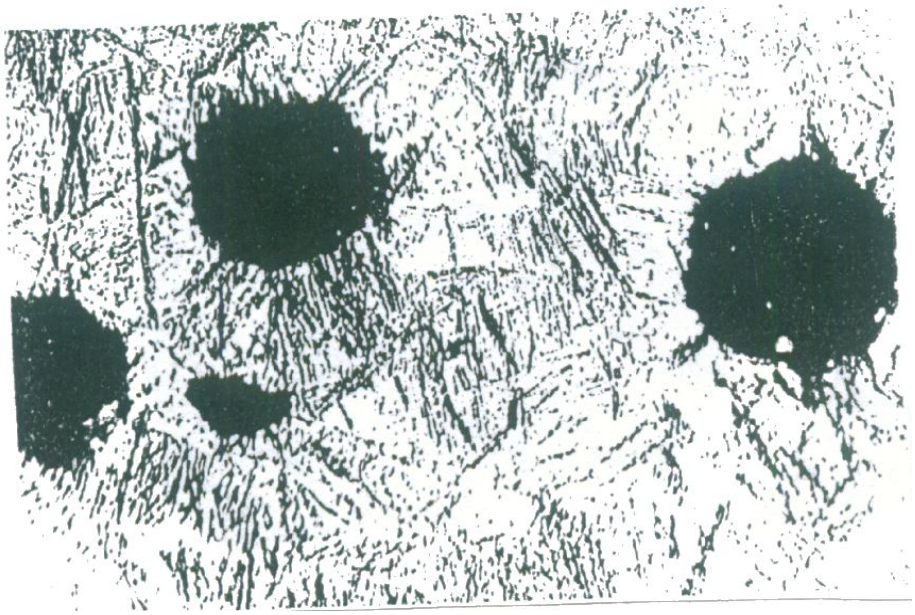


Plate D3: Optical microstructure of ductrile cast iron austenitised at 900^0C and austempered in salt bath furnance held at 350^0C, then removed and cooled in air; as reported by Ming-chang Jeng (1993).

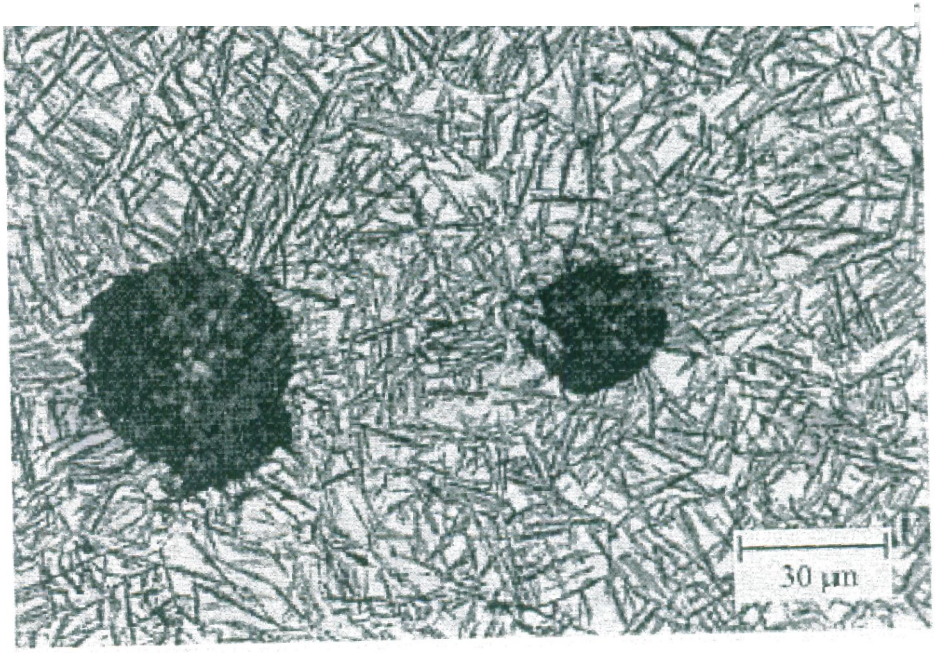


Plate D4: Optical microstructure of ductile cast iron austempered in motten salt bath as reported by Yescas – Gonzalez et al, (2001).

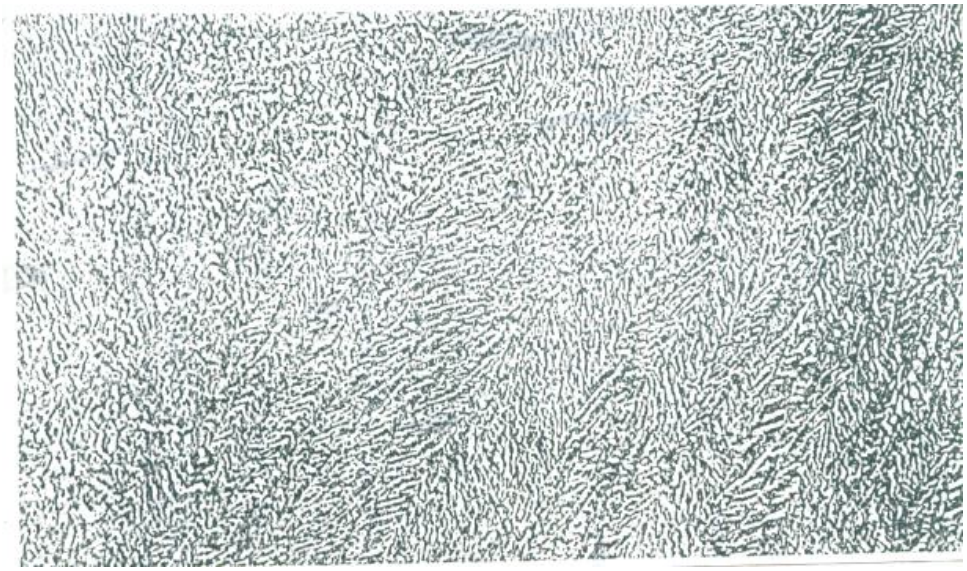


Plate D5: Microstructure (scanning electron microscropy) of plain high carbon steel austempered in salt bath furnance showing bainite structure as reported by Harold (1971).

APPENDIX E

THE COOLING RATE OF BITUMEN AND WATER

Water was heated to the boiling temperature of 100^0C and poured into a calorimeter. A laboratory thermometer and a stirrer was inserted/dipped into the hot water in the calorimeter. The cooling rate (drop in temperature with time) at the interval of five minutes was recorded for a period of 110minutes.

Bitumen was heated to 100^0C and poured in a calorimeter as was done with water, and the same interval of five minutes was used to record the drop in temperature with time. During the experiment, the content of the calorimeter was constantly stirred to ensure uniform temperature in the liquid.

Figure E_1 below show the cooling curves for water and bitumen. It shows that when bitumen and water were heated up and allowed to cool, bitumen cooled faster than water.

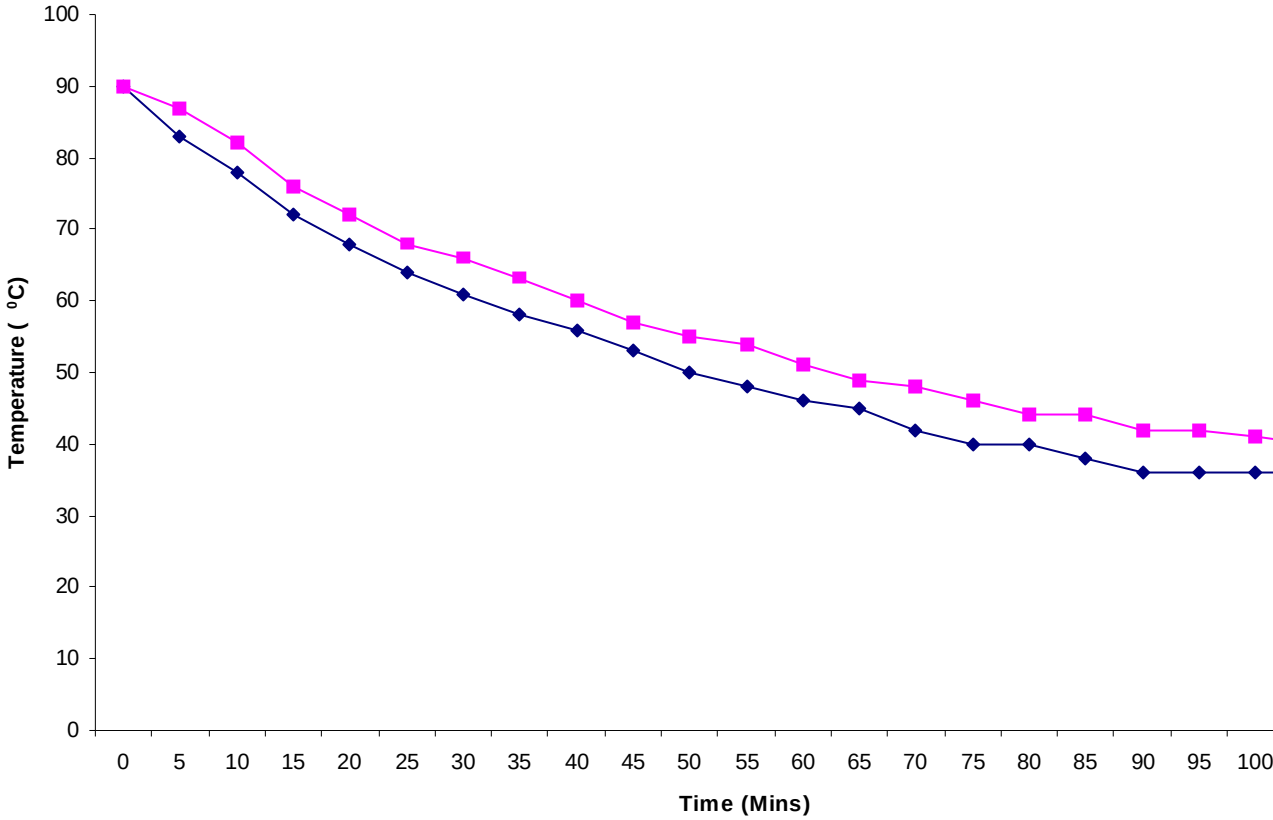


Figure E_1: **Plot of the cooling rate of Bitumen and Water**

APPENDIX F

A SKETCH OF A PROPOSED FUEL (KEROSENE) FIRED AUSTEMPERING BATH

(A) Dimensions of the container (pot) for the proposed austempering bath

Volume of cylinder = $\prod r^2h$

Proposed volume of the pot = $5000cm^3$ (5litres)

Proposed height of pot = 20cm,

From $V = \prod r^2h$

$500 = \prod r^2h\ (20)$

$r^2 = \frac{500}{20\prod}$

$r^2 = \sqrt{\frac{500}{20\Pi}}$

r = 8.9 = 9cm

:- Diameter = 2r = 2 x 9 = 18cm

(B) Dimensions of the fuel tank

Proposed volume = $8000cm^3$ (8 litres)

Proposed height = 35cm

From $V = \prod r^2h$

$8000\ \prod r^2\ (35)$

$r^2 = \frac{8000}{35\prod}$

r = $\sqrt{\frac{8000}{35\Pi}} = 8.5$

D = 2r = 2 x 8.5 = 17cm

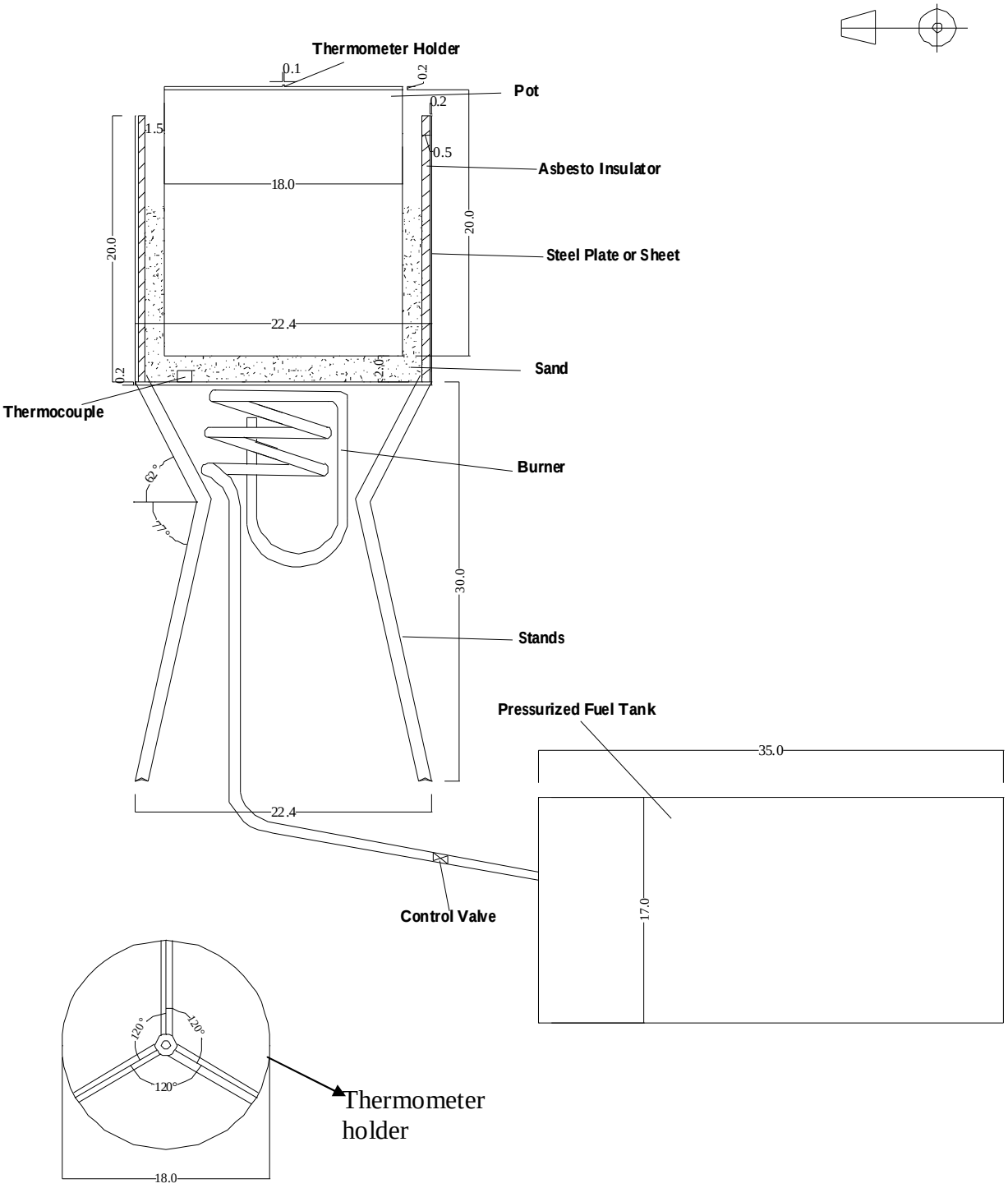


Fig. F: A SKETCH OF A PROPOSED FUEL (KEROSENE) FIRED AUSTEMPERING BATH