

**POTENTIALS OF USING SOME VEGETABLE OILS AS QUENCHANTS FOR
AUSTEMPERING OF STEELS AND CAST IRONS.**

By

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AUGUST, 2008.

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**A Dissertation Submitted to the Postgraduate School, Ahmadu Bello
University, Zaria, for the degree of Doctor of Philosophy (Metallurgical
and Materials engineering).**

**Department of Metallurgical and Materials Engineering
Faculty of Engineering
Ahmadu Bello University, Zaria.
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August, 2008.

DECLARATION

I **Sani Alhaji Salihu** wish to declare that this dissertation entitled Potentials of Using Some Vegetable Oils as Quenchants for Austempering of Steels and Cast Irons solely composed by me and that it is the result of my own research work under the supervision of Dr. S. B. Hassan. It has never been submitted and accepted any where for any higher degree.

All literatures cited have been duly acknowledged in the reference.

.....
Sani A. Salihu

CERTIFICATION

This dissertation entitled: POTENTIALS OF USING VEGETABLE OILS AS QUENCHANTS FOR AUSTEMPERING OF STEELS AND CAST IRONS by **Sani Alh. Salihu** meets the regulations governing the award of the degree of Doctor of Philosophy (Metallurgical and Materials Engineering) of Ahmadu Bello University, Zaria and is duly approved for the contribution to knowledge and literary presentation.

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DEDICATION

This research work is solely dedicated to my Father, **Alh. Salihu** for

his prayer, love, patience and cooperation.

ACKNOWLEDGEMENT

I thank Almighty God for giving me the will and health to complete this work. Glory be to Allah, the most merciful who made my dreams of becoming Doctor of Philosophy possible even in the face of many oppositions. I wish to express my sincere gratitude and appreciation to Dr. S. B. Hassan, chairman supervisory committee, who provided advice and guidance for this work, from the beginning to the end. His keen interest and expert suggestion have been very useful in the successful execution and completion of this work. My profound gratitude goes to Dr. A. I. Obi, a member of the supervisory committee, for his keen interest, advice and persuasion and made useful suggestions. I am deeply grateful to Dr. O. Aponbeide, the Head of Department, Prof. Oloche, Dr. S.A. Yaro for the encouragements, advice and persuasion at various stages of this work. I thank you all. My sincere thanks to the entire staff of the Department of Metallurgical and Materials Engineering, Ahmadu Bello University, Zaria for the assistance and corporation.

I am grateful to the management of Waziri Umaru Federal Polytechnic, Birnin-Kebbi for granting me the opportunity to study with my pay only. Special appreciation goes to the Kebbi State Government for the financial assistance given to me in completing this work. I also wish to thank Engr. Yakubu M. and Zamani of NMDC, Jos for their assistance during the search for the materials. I am grateful to Mr. Y. Danladi, a Technologist in the Department of Metallurgical and Materials Engineering, Ahmadu Bello University Zaria for his tireless assistance in the laboratory. My sincere thanks to the entire staff of the Department of Metallurgical Engineering, Waziri Umaru Federal Polytechnic, Birnin Kebbi for their Co-operation and understanding while undergoing this research work.

Finally I wish to thank my family for their prayers; encouragement and

support throughout the period of this research work. God bless you all. Ameen.

ABSTRACT

Austempering heat treatment practice helps in achieving high strength with good ductility and toughness by evolving a predominantly bainitic or ausferritic microstructure in steels or cast irons respectively by using salt bath. The potentials of using fatty-based vegetable oils as quenching media for austempering of steels, and cast irons have been investigated. Cottonseed, groundnut and shear butter oils were used as hot quenching bath for austempering of these materials. The effects of these oils as quenching media were evaluated in respect of basic mechanical properties and microstructure. Standard tensile, impact and hardness samples were machined from bar of 0.371%C-steel, low alloyed steel, ductile iron and grey cast iron. The samples were austenitized at 950°C, quenched at 250°C in hot cottonseed, groundnut and shear butter oils and austempered for 1 to 5 hrs. The results of the microstructures obtained showed that hot shear butter and groundnut oils formed bainite and ausferrite structures respectively at different austempering time in carbon steel and ductile cast iron. However, there was partial formation of bainite in austempered low alloyed steel using shear butter and cottonseed oils. The results showed clear disparity in mechanical properties between these oils. The austempered ductile iron and medium carbon steel had the highest tensile strength values of 1041N/mm² and 1020N/mm² using groundnut and shear butter oils respectively; while austempered grey cast iron had the lowest values of 631N/mm² and 684N/mm² in groundnut and shear butter oils respectively. However, the austempered samples of all the materials investigated gave poor tensile strength in cotton seed oil. The austempered medium carbon steel and ductile iron equally showed high impact values of 101J and 68J in shear butter at 5 and 4hrs respectively while the hardness values of these materials were 353HV and 412HV respectively in hot shear butter oil. Based on the microstructure developed and mechanical properties results achieved in this work, hot shear butter and groundnut oils are effective as austempering quenchants for medium carbon steel and ductile iron up to 4hrs austempering time.

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LIST OF ABBREVIATION

| | |
|---------------|---------------------------------|
| G/Nut oil: | Groundnut oil |
| S/Butter oil: | Shear butter oil |
| C/Seed oil: | Cottonseed oil |
| TTT | Time Temperature Transformation |
| ASM | American Society for Metals |
| SG | Spheroidal Graphite |
| CG | Compacted Graphite |
| $V_{h,av}$ | Average heating rate |
| V_h | Heating rate |
| V_c | Cooling rate |
| $V_{c,av}$ | Average cooling rate |
| T_{max} | Maximum Temperature |
| τ_c | Cooling time |
| τ_h | Holding time |
| τ_s | Soaking time |
| τ_h | Heating time |
| τ_{tol} | Total heating time |
| A_{C3} | Upper critical temperature |
| A_{C1} | Lower critical temperature |
| A_{CM} | Upper critical temperature |
| M_s | Martensite starts |
| M_f | Martensite finish |
| ADI | Austempered ductile iron |
| % | Percentage |
| $^{\circ}C$ | Degree centigrade |
| $^{\circ}K$ | Degree Kelvin |
| E | Modulus of Elasticity |
| HV | Vickers Hardness |
| HB | Brinell Hardness |
| HR | Rockwell Hardness |
| F | Load |
| D | Diameter |
| R | Constant |
| SAM | Scanning Acoustic Microscope |
| γ | Austenite |
| α | Ferrite |
| σ_u | Ultimate tensile strength |

| | |
|----------------|--------------------------------|
| $\sigma_{0.2}$ | Yield strength or proof stress |
| δ | Elongation |
| ψ | Reduction in area |
| OES | Optical Emission Spectrometer |
| R | Radius |

CHAPTER ONE

1.0 INTRODUCTION

Nature provides more than 50 metallic elements, and by alloying these elements, the number of theoretically possible alloys reaches approximately 400,000 (John Nsely, 1970). However, technological and economic considerations limit choice to a few basic metallic elements such as Fe, Al, Cu, Ti and a small number of alloying elements.

Most of the progress in the technical field can be closely linked to the advancements made in the development of materials. Alloys, such as steels have been in used for centuries and remain the most importance materials for many industrial applications and economic development of any modern society (Robert et al, 1961).

Despite these widespread applications of alloy steels, technological advancements have put more stringent demand on improved quality and enhanced properties. Improvements in properties are however chiefly acquired by controlling composition and by further processing in the form of heat treatment and mechanical working. Today, civilization is based on the advanced technology that depends on the properties of engineering materials for high strength, toughness, wear resistance, hardness and fatigue strength (Payson, 1962).

The versatility of steel as an engineering material arises from the fact that its properties are very amenable to heat treatment. Heat treatment principle governs the procedures required to obtain particular microstructure in a given metal to suit desired working conditions. The desire to obtain optimum properties in steels and cast irons economically requires that the nature of the factors involved in the processing of

the materials are thoroughly understood so as to facilitate the design of alloys which conform to desired specifications.

Based on the type of applications, majority of steels and cast iron components required specific heat treatment to produce structures having both chemical and mechanical properties for desired applications. In developing countries like Nigeria where these alloys are not available, it is then necessary to up-date the use of already existing alloys by heat treatment.

The development of austempered parts can be traced to 1930s when Bain and Grossman, working for the United State Laboratories evaluating the metallurgical response of steels cooled rapidly from 788°C to intermittently high temperatures and held there for various times. The outcome of their pioneering research is what is now commonly called the "isothermal transformation diagram". The outcome of their work a new structure was discovered known as bainite. The process consists of: 1) Austenitizing the steel at 840-950°C. 2) Quenching at a temperature just above the M_s temperature. 3) Holding isothermally in the quenchant until austenite → bainite transformation is complete. 4) Cooling to room temperature in air. It is designed to produce a structure with unique mechanical properties, compared with conventionally quenched and tempered steel. Molten salt is the most popular quenchant used for austempering process because it transfers heat rapidly and has uniform viscosity over a wide range of temperature (ASM, 1991 and Khanna, 2002).

1.1 Statement of Problem

Molten salt bath is an expensive furnace and scarce in developing countries like Nigeria and in addition has the following shortcomings:

(a) the molten salt bath may cause explosion as a result of instrument failure. (b) Salt bath heat treating is very hazardous especially during cleaning and in maintaining the bath at elevated temperature (Jetley, 2007). (c) Molten salt can be dangerous as it can produce vapours which are toxic to workers. (d) The salt bath is, in general, inefficient and the running costs are high (Koshal, 1993). (e) The surfaces of samples become corroded if, for any reason, they are left too long in the bath. Therefore quenchants, such as vegetable oils, were investigated to provide similar properties with fewer problems. Hence three vegetable oils (cottonseed, groundnut and shear butter oils) were identified and studied. This research work reports this study.

1.2 Research objective.

The major objective of this research work is to assess the potentials of using local vegetable oils (cottonseed, groundnut and shear butter oils) as quenchants for austempering heat treatment of steels and cast irons. To accomplish this, microstructure, tensile properties hardness and impact strength were used as criteria to evaluate the effectiveness of these oils as quenching media for austempering process.

1.3 Justification.

ASM, (1991) and Higgins, (1998) highlighted that molten salt and lead have been the most popular quenchants used for austempering process. However, their substantial deficiencies with respect to environmental friendliness, high cost, non-availability and toxicity

justify the search for alternative replacement media. For centuries, vegetable oils and other non-petrochemical materials were used for heat treatment of steel (Larry Olson, 2001). Nigeria is enriched with varieties of these oils and fats that were mostly used in soap, pharmaceutical, candles and food industries. Larry Olson (2001), and Keith Addison (2003), have reported that vegetable oils can be used for heat treatment of steels and that the benefits of using these oils are great, depending on the following factors: (a) Hardenability of steel (b) Component geometry (c) Furnace equipment (d) Metallurgical properties (e) Safety. The effects of different vegetable oil properties on combustion and pollution formation in a DI Diesel engine have also been studied by Thomas et al (2003). They also observed that the criterion to choose the vegetable oils were their properties, which should vary as far as possible. They concluded that with the exception of particulates and nitrogen oxide emissions measurements, combustion progress and noises analysis revealed no significant differences between the palm, coconut, babassu and sunflower oils. However, not much is known on the effect of using vegetable oils as austempering quenchants for heat treatment of steels and cast irons.

The benefits of replacing molten salt bath for austempering process are: a) biodegradable, b) can be manufactured from renewable plant source, c) it is cheaper and d) produce less harmful by products.

1.4 Background study.

The austempering process was first discovered by Bain et al in 1933. Initially, it was applied to steel and produced an acicular microstructure known as bainite. This microstructure has an exceptional mechanical

properties compared with conventionally quenched and tempered steel. In 1937, the austempering process was applied to grey cast iron and mechanical Properties were enhanced, but full potentials was not realized due to the limiting factor of the graphite shape. In the 1940's, the austempering process was used in the production of gun parts, resulting to low distortion, high strength and toughness (www.key-to-steel.com). By the 1950's the Austempering process was routinely applied to steels, ductile cast iron and malleable iron parts. In the 1990s austempered components were being used for numerous applications worldwide. Nowadays, austempering process is used in the production of austempered ductile iron which is an alternative in many applications that were previously the exclusive domain of steel casting, forging and aluminum. According to Keuogh (1998), ADI market represent all nearly segments of manufacturing which includes: Automobile, Heavy trucks, Railroads, pumps and compressors, Mining and Construction equipment.

Bainitic structures produced during austempering process of medium carbon steel and ausferrite in nodular cast iron exhibits 15% higher in tensile strength, 24% higher yield strength or proof stress and at least a tenfold increase in reduction area and impact when compared to identical specimen conventionally quenched and tempered to an equivalent hardness, (Hphypex Inc.2003). Austempering process will produce better mechanical properties particularly higher ductility, resistance to shock and fatigue stresses in ferrous alloys.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Introduction

Steels and cast irons are ferrous alloys of iron and carbon with carbon not exceeding 1.5% in steel, while cast iron has 2-4% carbon with high silicon content. They are the most importance metallic alloys used in modern engineering (Gulyaev, 1980). The versatility of the steels as engineering materials is evidenced by the many kinds of steels and cast irons that are produced. The equilibrium diagram(Figure 2.0) shows that between iron and cementite there is a eutectic-forming series of alloys with the eutectic reaction occurring at 4.3% carbon and 1130°C (Pascoe, 1978).

2.2 STEEL

Steel is a ferrous alloy of iron and carbon with carbon not exceeding 2.0%. Steels are classified not only based on their methods of production, but also according to their chemical composition, structures and uses, (Polukhin, 1977 and Khanna, 2002).

According to their chemical composition, steels are classified into plain carbon steels and alloy steels. Plain carbon steels are iron-carbon alloys with carbon content not exceeding 2.0% and definite amounts of inevitable impurities, which include silicon, manganese, sulphur and phosphorus. The percentages of these elements in carbon steel are generally low, with maximum values of 0.80%Mn, 0.05% P, 0.50%Si and 0.05%S (Gulyaev, 1980). Figure 2.0 shows the iron-carbon system phase diagram for carbon steel up to 6.67%C.

2.2.1 Plain Carbon Steel

In the former USSR, plain carbon steels are classified into three groups: A, B, and C, depending on their application (Gulyaev, 1980).

Depending upon the carbon contents, plain carbon steels can be classified into the following:

1. Low carbon steels with carbon content up to 0.30%.
2. Medium carbon steels with carbon content from 0.35-0.65%.
3. High carbon steels with carbon content from 0.65-0.90%.
4. Tools with carbon content from 0.90-1.50%

Higgins (1998), grouped plain carbon steels depending on their specific uses as follows:

i. Dead Mild Steel (0.05-0.15%C)

These steels are soft and ductile and cannot usefully harden by heat treatment. The steel can only be hardened by carburizing the surfaces to improve the mechanical properties. The amount of this steel is ferritic, i.e. ferrite is more with little amount of pearlite. They are used where high cold formability is required, e.g. car body work and tin plate.

ii. Mild Steel (0.10 – 0.30%)

This is general purpose steel and though reasonably strong, is used where hardness and tensile strength are not most important requirements. It is ductile and cannot be usefully hardened by heat treatment. Its structure is ferritic and pearlitic. This steel is used for food and beverage cans, as joists in buildings, screws, nails, wire, levers for various forgings (Bolton, 2002).

iii. Medium Carbon Steel (0.35-0.65%)

This is a strong and heat treatable steel to produce a wide range of properties in the quenched and tempered state, austempered state and martempered state. The structure of this steel at ordinary temperature is ferritic and pearlitic. Under the microscope the pearlite appears dark or striped. As the proportion of pearlite increases, the steel becomes less ductile and more resistance to deformation. Depending upon their response to heat treatment, medium carbon steel can be sub-divided into two main groups.

- a. Poor hardening treatment with carbon content (0.25-0.35%).
This is used for making connecting rods, wire, axle crane hooks, etc.
- b. Excellent hardening treatment with carbon content (0.35-0.65%)
and is used for crankshafts, axles, rails, rope, die block, shafts, etc.

(Bolton, 2002).

iv High Carbon Steels (0.65-0.90%C)

This steel has ferrite, pearlite and cementite which is very hard and brittle. The relative amount of these structures depends upon the carbon content of the steel but pearlite will contain exactly 0.8% carbon. The steel has poor fabricability, weldability and machinability, as compared to medium carbon steel. Heat treatment processes such as annealing, tempering, austempering and martempering, can improve these properties. The steel can be used where hardness is more important than ductility. It is used for chisels, machine tools, spring, piston rings, razor, punches, etc (Higgins, 1998).

v. Tool Steels (0.90-1.50%C)

This contains a structure of cementite and pearlite in relative amount, which depend upon the carbon content. These structures are hard and brittle and can be improved by heat treatment. Steels of this type are used for tools that require high toughness and moderate hardness, e.g. cold chisels, forging, dies, rivet, bits, blades, etc.

2.2.2 Alloy Steels

Alloy steels are steels to which alloying elements are added intentionally to improve certain properties.

The principal alloying elements added to steel is widely varying amounts either single or in complex mixtures are nickel, copper, chromium, manganese, tungsten, molybdenum, titanium, vanadium, aluminium, silicon and cobalt, (Rollason, 1978).

Classification of Alloy Steels

Alloy steels can be classified according to the elements they contain (e.g. nickel steels, chromium-vanadium steels, etc) or according to their uses (e.g. high-tensile structural steels, stainless steels, etc) or according to the structure produced by the heat treatment usually employed (e.g. pearlitic, martensitic, austenitic). Pascoe (1978), pointed out that, the most reasonable classification is based primarily on use.

Effects of Alloying Elements on Steels and Cast Irons.

According to Higgins (1998) and Pascoe (1978), the alloying elements affect steel and cast irons the same way. The effects of these alloying elements in the steels may be one or more of the following:

1. It may go into solid solution in the iron, enhancing the strength.
2. Hard carbides associated with Fe_3C may be formed.
3. It may form intermediate compounds with iron e.g. Fe Cr (sigma phase), Fe_3W_2 .
4. It may influence the critical range in one or more of the following ways.
 - a. Alter the temperature
 - b. Alter the carbon content of the eutectoid.
 - c. Alter the "critical cooling velocity", which is the minimum cooling speed which will produce bainite or martensite from austenite.
5. It may produce effects characteristic of the alloying element. Rollason (1978), noted how alloying elements affect the stability of the carbides and the stability of the austenite and ferrite in steels.

a. Elements which tend to form carbides:

These elements may form stable, hard carbides which can if in an appropriate form such as fine particles, increase the strength and hardness. Chromium, tungsten, titanium, columbium, vanadium, molybdenum and manganese have this effect.

b. Elements which tend to graphitize the carbide:

Silicon, cobalt, aluminium and nickel, could cause the breakdown of cementite and lead to the presence of graphite in the alloy. The result is a decrease in strength and hardness.

c. Elements which tend to stabilize austenite:

Some alloying elements like Mn, Ni, Cu and Co alter the critical points of iron in a similar to carbon by raising the A_u point and lowering the A_3 point, thus increasing the range in which austenite is stable, and they also tend to retard the separation of carbides, (Rollason 1978). If a sufficient high percentage of one of these elements is added, the transformation temperature to austenite may be decreased and austenite is retained at room temperature. These elements have a crystal structure (fcc) similar to that of γ -iron in which they are more stable than α -iron.

d. Elements which tend to stabilize ferrite:

Alloying elements like Cr, Mo, V, Si, Al and W are more stable in α -iron than in γ -iron. They diminish the amount of carbon soluble in the austenite and thus tend to increase the volume of free carbide in the steel for a given carbon content.

One particular effect of alloying elements is that it enables martensite to be produced with low rates of cooling and permits larger sections to be hardened than is possible with plain carbon steel. Also, alloying elements retard the process of martensite breakdown, e.g. Cr, Mo, Si, have this effect. The general effect of alloying elements is to reduce the diffusion rate of carbon and so slow down the transformation of austenite to ferrite and pearlite structures.

2.2.3 Steels for Austempering

The selection of steel for austempering process must be based on transformation characteristics as indicated in time-temperature transformation (TTT) diagrams. According to ASM (1991), and Higgins (1998), there are three important points that must be considered in selection of steels for austempering and these are:

- a. Location of the nose of the TTT curve and the time available for by passing it.
- b. The time required for complete transformation of austenite to bainite at the austempering temperature.
- c. The location of the Ms point (Figure 2.1).
 - i. Plain carbon steels containing 0.50-1.00%C and a minimum of 0.60%Mn.
 - ii. High carbon steels containing more than 0.90%C and a little less than 0.60%Mn.
 - iii. Certain low alloy steels such as 5100 steels containing more than 0.30%C, the series of 0.40%C and other steels. Such as 4140, 6145 and 9440.
 - iv. Certain carbon steels such as 1041 with a carbon content less than 0.50%, but with Mn content in the averages from 1.00-1.65%.

As indicated by ASM (1991) and Higgins (1995), austempering can be applied to parts where the transformation to pearlite can be avoided. It means that the specimen must be cooled fast enough to avoid the formation of pearlite.

2.3 CAST IRONS

2.3.1 Cast Iron in General

Basically, cast iron is derived from pig iron, the product of the ferrous blast furnace. Pig iron contains 2-4% carbon and up to 3.5% silicon, depending primarily on the composition of the raw materials charged to the blast furnace (Bailey et al, 1976). Therefore, cast iron is distinguished from steel by a higher carbon content greater than 2.14%, better castability, high compressive strength, and lower ability to plastic deformation and high fluidity, while carbon in ordinary steel exists as cementite (Fe_3C) (Higgins, 1998)

The following types of cast iron are distinguished depending on the state of carbon in the structure:

- a. **White Cast Iron:** in which all the carbon is combined into carbides.
- b. **Grey cast iron:** in which all or an appreciable portion of the carbon is present in free state as lamellar graphite.
- c. **Nodular or ductile cast iron:** in which all or an appreciable portion of carbon present in free state as spheroid graphite.
- d. **Malleable cast iron:** which is obtained by annealing of white cast iron castings; all free state as flaky graphite.

Thus, cast irons (except white cast iron) differ from steels by the presence of graphite inclusions in the structure and from one another, by the form of these inclusions (Gulyaev, 1980).

2.3.2 White Cast Iron

A white cast iron (Figure 2.2) contains both primary and pearlitic cementite. Cementite is a white, hard brittle compound, so that iron which contains much of it will present a white fracture when broken, and will have a low resistance to shock, but possess a high resistance to wear. As expressed by Bolton (2002), it is formed as a result of fast cooling to give a structure and brittleness of which cast iron makes it difficult to machine and hence of limited use.

However, white cast iron is used in considerable amounts as the starting material for the manufacture of malleable irons. Apart from these applications, Bailey (1976), noted that, very few castings are made entirely of white structure. The major applications of white cast iron include rolls, railway wagon wheels, and various parts of crushing, grinding, and materials handling equipments.

2.3.3 Grey Cast Iron

In grey cast iron (Figure 2.3) the graphite is distributed as flakes, which breaks up the continuity of the metal matrix-a carbon steel of itself possessing considerable strength and ductility. In ferritic grey cast iron all the carbon is graphite.

In pearlitic grey cast iron, some is in pearlite. The graphite flakes have no strength and act as internal cracks making the materials weak and brittle and extremely liable to fracture under shock loads (Pascoe 1978).

The grey cast iron is distinctly softer than white cast iron and is readily machineable. However, grey cast iron has useful rigidity and its compressive strength is not so impaired. Moreover, the graphite impacts a useful combination of other properties. (Bailey 1976).

Grey Cast iron structures are favoured by high silicon content and a low rate of cooling, while white cast iron formation is encouraged by the reverse conditions.

The strength and hardness of cast iron according to Bolton (2002), depends very much on its microstructure, i.e. on the form taken by the carbon, and on the nature of the matrix structure. The mechanical properties are generally poor due to the morphology, i.e. presence of graphite flakes. The pointed ends of graphite flakes act as stress raisers.

Table 2.0 shows the properties of unalloyed cast irons.

Table 2.0: Properties of unalloyed cast irons.

| No | Cast iron | Type | Minimum Mech. Props. | | | Typical Uses |
|----|------------------------|---------------------------------------|----------------------|------------|--------|---|
| | | | σ_u | σ_y | Elong. | |
| | | | Mpa | Mpa | % | |
| 1. | Grey 3.5C-2.5Si | Pearlitic | 272 | 240 | <1 | Engine Block Wearing surf. Cam shafts Pipe, mach. bases |
| | | Martensitic | 550 | 550 | nil | |
| | | Bainitic | 550 | 550 | nil | |
| | | Ferritic | 172 | 138 | <1 | |
| 2. | Nodular 2.5C-1Si | Ferritic | 413 | 275 | 18 | Pipe Crackshafts Special Mach. Parts Mach.parts,r/road equips |
| | | Pearlitic | 550 | 380 | 6 | |
| | | Tempered | 825 | 620 | 2 | |
| | | Mart | 1050 | 810 | 18 | |
| | | Bainitic | | | | |
| 3. | Malleable 2.2C-1Si | Ferritic | 365 | 240 | 18 | Hardware Railroad equipments Railroad equipments |
| | | Pearlitic | 450 | 310 | 10 | |
| | | Tempered | 700 | 550 | 2 | |
| | | Mart. | | | | |
| 4. | White 3.5C-0.5Si | As-cast (pearlitic) Martensitic | 275 | 275 | Nil | Wear-resistant products |

Source: Lawrence H. Van Vlack(1985).

2.3.4 Malleable Cast Iron

Malleable cast irons are produced by heat treatment of white cast irons for several days under control conditions. The weakening effect of the graphite is reduced so that malleable cast iron has a higher strength and greater ductility and shock resistance. Major applications of malleable cast iron are found in agricultural, railway and automobile engineering.

Bailey et al (1976) noted that two main annealing processes are used both with annealing atmospheres of either decarburizing or neutral.

The resultant materials are known, respectively as whiteheart and blackheart which terms are descriptive of the colour of their fractured surfaces.

In the whiteheart process, the heating process is carried out under oxidizing condition so that carbon is progressively eliminated from the fracture. As carbon in the surface layers oxidizes and is lost as carbon dioxide, more carbon diffuses outwards from the core and in turn eliminated, leaving a totally ferritic structure through the central core of pearlite remains.

The carbon content for the whiteheart process is commonly higher than that used for the blackheart process. This type of cast iron is widely used in fitting for gas, water and steam pipe, bicycle and switch-gear equipment (Higgins 1995).

In the blackheart process, white cast irons are heated to 900°C and soaked for two day or more so that cementite breakdown to form spherical aggregates of graphite in a matrix of austenite followed by slow cooling, resulting in the austenite changing into ferrite and more graphite (Figure 2.4)

2.3.5 Nodular cast iron

Nodular cast irons have free carbon as graphite inform of spheroids or nodules graphite instead of flakes. This is achieved by adding small amount of magnesium or cerium under control conditions to the molten metal before pouring into the moulds, the graphite forms spherical particles on casting (Fig. 2.5). At room temperature the structure of nodular cast iron is mainly pearlitic with spheres of graphite. This spheroidal graphite cast iron is found to have a strength superior to malleable cast iron and with

suitable annealing, which precipitates further graphite from the pearlite matrix, the ductility is as good (Pascoe 1978).

An extensive range of properties is possible in nodular cast iron according to the nature of the matrix. Depending on composition, foundry practice, and heat treatment, the matrix may vary from pearlitic, pearlitic-ferritic or ferrite.

Table 2.1: Chemical Composition Ranges for Unalloyed Cast Irons

| Element | White % | Grey % | Malleable % | Nodular % |
|---------|-----------|-------------|-------------|-------------|
| C | 1.8 – 3.6 | 2.5 – 4.0 | 2.0 – 2.6 | 3.0 – 4.0 |
| Si | 0.50-1.00 | 1.0 – 3.00 | 1.10-1.60 | 1.8 – 2.80 |
| Mn | 0.25-0.8 | 0.25 – 1.0 | 0.20-1.00 | 1.10 – 1.00 |
| S | 0.06-0.20 | 0.02 – 0.25 | 0.04-0.18 | 0.03max |
| P | 0.06-0.18 | 0.05 – 1.0 | 0.18max | 0.10max |

Source: Smith,W.F.(1990).

2.4 MICROSTRUCTURE OF STEELS AND CAST IRONS

The microstructures of steels and cast irons are based on the existence of a eutectoid and eutectic structure respectively. In fact, it is the structure of the eutectic in case of cast irons that distinguishes one type of cast iron from the others.

Higgins (1998) highlighted that the structures and physical properties of steels and cast irons depend upon chemical composition and the rate, which it solidifies. The microstructure is as important as the chemical analysis in determining the final properties of a casting. The mechanical properties as well as machinability and wear resistance are almost entirely dependent upon the microstructure (Bolton 2002). To gain an understanding of the properties of engineering materials including steels and cast irons, their microstructures must be considered.

2.4.1 Microstructure of Steels

Pure iron exists at room temperature as body-centred cubic (bcc) structure, this being known as ferrite and is soft and very ductile and shows sharp grain boundaries as in Figure 2.6. With increase of carbon content, pearlite appears in increasing quantity. Under the microscope the pearlite appears dark or striped and may be distinguished from the ferrite as in Figure 2.7. In hyper-eutectoid steels, the cementite is concentrated on the former austenite grain boundaries and appears white between the dark pearlite areas as in Figure 2.8 (Pascoe, 1978).

Slowly cooled steel will contain the following structures at room temperature depending on the carbon content.

- a. With less than 0.025% carbon (i.e. entirely ferrite) are known as pure irons.
- b. With increase of carbon content between 0.025% and 0.8% carbon the structure will contain ferrite and pearlite which appears darks or striped and may be distinguished from ferrite as in Figure 2.7. The relative proportions of ferrite and pearlite appearing in the microstructure will vary according to the carbon content.
- c. With exactly 0.8% carbon, the structure will be entirely pearlite (Figure 2.9).
- d. With carbon content between 0.8% and 2.0%, the structure consists of cementite and pearlite, in relative amounts which depend upon the carbon content.

Gulyaev (1980) noted that, depending on the types of heat treatment and carbon content, steel exhibit different structures. Steel that is cooled rapidly from above its upper critical temperature using different quenching media will have the following structures.

- i. With 0.5% carbon cooled rapidly in water will produce very hard structure which appears under the microscope as acircular crystals, known as martensite (Figure 2.10).
- ii. Less severe quenching gives rise to a structure known as bainite (Figure 2.11).
- iii. With much slower cooling will produce normal pearlite, the coarseness of ferrite and cementite laminations depending upon the rate of cooling.

The general structures of steels depending upon the carbon contents, cooling rates and quenching media are as follows (Higgins, 1998):

1. Any steel containing less than 0.8% carbon will transform to pearlite and ferrite structure with slow cooling.
2. Steel containing more than 0.8% carbon will transform to pearlite structure only with slow cooling
3. Steel containing more than 0.8% carbon will transform to pearlite and cementite if it cools slowly from its austenitic state.
 - a. Medium carbon steel and high carbon steel will transform to martensite when cooled rapidly from the austenite condition.
 - b. A hardened steel (martensite) obtained from rapid cooling and modified by temperature will produce a tempered martensite depending on the tempering temperatures.

2.4.2 MECHANICAL PROPERTIES OF STEELS

The mechanical properties of steel are influenced by the structure of matrix, the type of alloying elements and composition. The presence of alloying elements in steel is responsible for high strength, wear resistance, hardness, corrosion resistance, hardenability and abrasion resistance. These result in high tensile strength, impact strength and toughness. The combined action of heat treatment and alloying is an effective means for improving the mechanical characteristics of steel (Gulyaev, 1980).

Among the possible methods for improving the mechanical properties of steels are increasing the carbon contents, alloying dispersion of structural component (by lowering the point of austenitic transformation, combined with tempering), grain refinement and strain hardening (Gulyaev, 1980).

The matrix structure is very important in both alloy steels and plain carbon steels. The as-cast low carbon steel matrices usually contain ferrite and pearlite. Figure 2.12 shows how the percentage of carbon affects the mechanical properties of steel.

For steels cooled slowly from the austenitic state up to the eutectoid composition, i.e. for hypereutectoid steels, the decreasing percentage of ferrite and the increasing percentage of pearlite results in an increase in tensile strength and hardness, while ductility decrease.

For hypereutectoid steels, increasing the amount of carbon decrease the percentage of pearlite and increase the percentage of cementite. This increases the hardness but has little effect on the tensile strength, the ductility also changes little (Bolton, 2002).

As observed (Figure 2.12), the hardness of plain carbon steels depends upon the amount of carbon content up to 0.8% carbon.

For alloy steels, the hardness depends upon the percentage of alloying elements. As pointed out by Gulyaev (1980), that there practically remain only one method for improving hardenability of steel. This is by alloying.

As has been noted addition of alloying elements first improves the mechanical properties of steel, until through, hardenability is attained at given conditions. After that, further increase in the content of an alloying element will impair the mechanical properties.

Retirement of steel from harmful impurities, when combined with grain refinement by high-temperature thermo-mechanical treatment can raise impact strength and practically form conditions for using steel with $\sigma = 220 - 240\text{Mpa}$ (Gulyaev 1980).

TABLE 2.3: MECHANICAL PROPERTIES OF PLAIN CARBON STEELS OF NORMALIZED AND FULL ANNEALED.

| % Carbon | Treatment | Austenitizing Temp. °C | Tensile Strength Mpa | Yield Strength Mpa | % Elongation | Hardness HB | Impact Strength J |
|----------|------------|------------------------|----------------------|--------------------|--------------|-------------|-------------------|
| 0.2 | As rolled | | 450 | 330 | 36 | 143 | 87 |
| | Normalized | 870 | 440 | 346 | 36 | 130 | 118 |
| | Annealed | 870 | 395 | 295 | 37 | 111 | 123 |
| 0.4 | As rolled | | 620 | 413 | 25 | 200 | 49 |
| | Normalized | 900 | 590 | 374 | 28 | 170 | 65 |
| | Annealed | 790 | 520 | 353 | 30 | 150 | 44 |
| 0.6 | As rolled | | 814 | 483 | 17 | 240 | 18 |
| | Normalized | 900 | 775 | 420 | 18 | 230 | 13 |
| | Annealed | 790 | 625 | 373 | 23 | 150 | 11 |

Source: Bolton, (2002).

TABLE 2.4: COMPARISON OF TYPICAL MECHANICAL PROPERTIES OF AUSTEMPERED AND OIL QUENCHED AND TEMPERED 1090 STEEL

| PROPERTIES | AUSTEMPERED | QUENCHED AND TEMPERED |
|-----------------------|-------------|-----------------------|
| Tensile strength, Mpa | 1415 | 1340 |
| Yield Strength, Mpa | 1020 | 895 |
| Elongation, % | 11.5 | 6.0 |
| Reduction of area, % | 30 | 10.2 |
| Hardness, HB | 415 | 388 |
| Fatigue Cycle (d) | 105,000(e) | 58,000(f) |

Source: ASM, 1964

2.4.3 Microstructure of Cast Irons

The microstructure and physical properties of a cast iron depend upon both chemical composition and the rate at which it solidifies following casting (Higgins, 1995). The following structures are possible for cast irons of different compositions and heat treatments.

The classification of cast irons by the structure of metallic matrix and shape of graphite is illustrated in Figure 2.13.

Primary Cementite and Pearlite

This microstructure is typical of the hard, white, low-silicon irons and is found also in other types of cast iron which have been chilled. A cementite-austenite eutectic forms at 1131°C and as the alloy cools to 723°C the austenite transforms to pearlite (Higgins, 1995).

Primary Cementite, Graphite and Pearlite

These are “molded” irons of such compositions that localized small changes in either composition or cooling rate will favour the formation of either cementite or graphite. The remaining austenite present at 723°C transforms to pearlite (Higgins, 1995).

Graphite and Pearlite

This microstructure is typical of a high-duty grey cast iron which solidified as a graphite-austenitic eutectic and in which the austenite has then transformed to pearlite at 723°C (Higgins, 1995).

Graphite, Pearlite and Ferrite

This will generally be a coarser grey iron which will be weaker and softer. The silicon content is high with this structure. Here, the graphite-austenite eutectic has cooled slowly through the eutectoid temperature 723°C for some of the carbon which was dissolved in the austenite to deposit on to the existing graphite flakes leaving a matrix of ferrite. Some of the austenite has also transformed to pearlite (Higgins, 1995).

Graphite and Ferrite

This variety of cast iron usually has very high silicon content. The graphite austenite eutectic has cooled slowly enough for the whole of the carbon dissolved in the austenite to diffuse on to existing graphite flakes leaving a matrix of ferrite.

Such a cast iron is very soft and easily machined. The ferrite present will contain dissolved silicon and manganese (Higgins, 1995).

The microstructure is as important as the chemical analysis in determining the final properties of a casting. In fact, the mechanical properties as well as machinability and wear resistant are almost entirely dependent upon the microstructure (Higgins, 1995).

2.4.4 Defects in Steels

The structures of a steel ingot depends upon the temperature of the metal during pouring the linear rate of pouring, the temperature of the mould and the thickness of its walls, the chemical composition of the steel, the deoxidizing method, etc, (Polukhin, 1977).

Steel ingot defects associated with steel, such as pipe and porosity, segregation, presence of non-metallic inclusions, blow holes, decarburization, and unsuitable heat treatment are commonly experienced defects found in steels: cracks, cavities, local concentration of impurities and brittle are not common. The formation of the pipe and porosity in the upper part of the ingot is explained by the difference in the specific volumes of the liquid and solidified steel (Donald, 1985).

Segregation in an ingot is the result of redistribution of the constituents when the steel passes from the liquid to the solid state. The carbon, sulphur, and phosphorus segregation are much greater than other elements. The presence of non-metallic inclusions derived from erosion of the refractory lining of the furnace result in oxidizing steel with manganese, silicon and aluminium.

The quality of steel is impaired by non-metallic inclusions (Polukhin, 1977), to the reduction in the solubility of gases (H_2 , O_2 , N_2) when the metal passes from the liquid to the solid state. These gases are retained in the mould in the form of blowholes and affect the structures and properties of steels.

Cracks are formed by non uniform cooling of steel parts. These have serious negative effect on steel properties. Flakes may form in all steels, but

most often they appear in chromium containing steels,. Generally, alloy steels are more often than carbon steels infected with various defects.

2.4.5 Defects in Cast Irons

Solidification in cast iron is accompanied by an expansion which may be used with a rigid mould to feed secondary shrinkage thus eliminating the need for conventional feeders. These characteristics, and those of low melting point and high fluidity, promote castability and make it possible for casting of complex shapes at relatively low cost (Hassan, 2002).

Casting defects associated with cast metals, such as shrinkages and voids are not commonly experienced. However, defects that are associated with molding materials such as surface flows, gas, holes, non-metallic inclusion, segregation, etc., are common. Hassan (2002) pointed out that nodular cast irons are considered as being degenerated if they contain certain irregular graphite morphology being referred as chunky, compacted, quasi-flakes, and coral.

The presence of spheroidizing and anti-spheroidizing elements (Be, Bi, Pb, Ti, Zb) often result in incomplete nodularization of graphite and formation of exploded graphites. Chunky graphite is small graphite particles arranged in groups generally smaller than neighbouring nodules. They are promoted by presence of excessive rare-earth elements, and is more prevalent in a hyper-eutectic nodular irons (Karsay, 1970).

2.5 TREATMENT OF STEELS AND CAST IRONS

The term heat treatment is used to describe a process involving controlled heating and cooling of a material in order to change its

microstructures and properties. Because of the solid state structural changes which take place in suitable alloys, steels and cast irons are among the relatively few engineering alloys which can be usefully heat treated in order to vary their mechanical properties (Gulyaev,1980).

According to Higgins (1998), heat treatment can be applied to steels and cast irons not only to harden but also to improve their strength, toughness and ductility. The types of heat treatment used will be governed by the carbon content of the alloy and its subsequent application.

ASM (1964), Rajan et al (1988) and Khanna (2002) pointed out that the purpose of any heat treatment process is to produce the desired changes in the structure and properties of an alloy by heating to a specified temperature, soak it at this temperature and subsequently cooling to room temperature. The factors that determine the outcome of heat treatment operations are the temperature at which it is cooled to room temperature, length of time the metal is held at elevated temperature and the cooling rate (Rajan et al, 1988).

Heat treatment may be undertaken for the following purposes:

- Improve toughness and ductility
- Relieving internal stresses
- Refinement of grain size
- Increase hardness or tensile strength
- Changes in chemical composition of metal surface
- Modification of electrical conductivity and magnetic properties.

Gulyaev (1980), noted that heat treatment conditions are characterized by the following parameters:

Heating temperature, t_{max} , i.e. maximum temperature to which an alloy is heated, holding time at the heating temperature t_h , Heating rate V_h , Cooling rate V_c .

$$V_{h.av} = \frac{t_{max}}{t_h} \dots\dots\dots(1)$$

$$V_{c.av} = \frac{t_{max}}{t_c} \dots\dots\dots(2)$$

2.6 HEAT TREATMENT PROCESSES

Heat treatment processes are carried out for the purpose of achieving certain structures and properties in metals and alloys. These are: Stress-relief annealing, Full-annealing, Normalizing, Hardening, Tempering, Austempering and Martempering.

Higgins, (1998) and Rajan et al, (1988), explained that, in all of these processes the steel or cast iron is heated slowly to some predetermined temperature and then cooled to room temperature. The rate of cooling determines the resultant structures of the alloy and hence, the mechanical properties of the alloy. The final structure will be independent on the rate of heating, provided this has been slow enough for the steel to reach the structural equilibrium at its maximum temperature.

The subsequent rate of cooling, which determines the nature of the final structure, may vary between a drastic water quench and a slow cooling in the furnace.

2.6.1 Stress-Relief Heat Treatment

Stress-relief heat treatment is used to relieve stresses that remain locked in a structure as a consequence of a manufacturing sequence. Iron castings are rarely free from residual stress, because cooling of a casting in the mould is not always uniform especially if the casting is of complicated shape. Internal stresses, which are generated by differential cooling rates in different sections of the casting, have to be relieved for dimensional stability and improved toughness. For steels, at temperature above 350°C dislocations move very rapidly in the matrix to relieve any internal stress (Bolton, 2002).

The presence of residual stresses can also lead to stress corrosion, cracking near welds and in regions of a component that has been cold strained during processing. As pointed out by ASM (1991), that residual stresses are present whenever a component is stressed beyond its elastic limit and plastic flow occurs. The problems associated with internal stresses are most difficult in brittle materials, like cast irons than in ductile material, like steels.

Heating to eliminate these stresses as a separate heat treatment operation is called stress relief. It is a uniform heating of steels and cast irons to a suitable temperature below the lower critical temperature transformation A_1 , holding at this temperature for a predetermined time, followed by uniform cooling to room temperature. If the rate of cooling is not constant, new residual stresses can result that are equal or greater than those for which the heat treating was intended to relieve (Rajan et al, (1988) and ASM, 1991).

For plain carbon steels and low alloy steels maximum stress relieving temperatures are between 595°C and 675°C while nodular cast iron has between 510-570°C. In general, the extents to which stresses can be relieved depend on the temperature and holding time (Higgins, 1998).

2.6.2 Annealing Heat Treatment

Annealing heat treatment is employed in steels and cast irons to reduce the hardness, improve machinability, facilitate cold working, relieve internal stresses developed during solidification, forging, rolling, or welding, eliminated chemical non-uniformity, reduce the gaseous contents in steels, produce a desired microstructure and properties. ASM (1964), stated that, when applied to ferrous alloys the term “annealing” means full annealing.

Full annealing is a structural re-crystallization consisting in heating a ferrous alloy by austenitizing above A_{c3} and subsequent furnace cooling. The heating temperature and holding time are determined by various factors such as the chemical composition of steel on cast iron, size and shape of the component and the final properties desired.

Depending upon the heat treatment temperature, annealing treatment can be subdivided into three main classes (Rajan et al, 1988).

- i. Full annealing
- ii. Partial annealing
- iii. Sub-critical annealing

Gulyaev (1980), explained full annealing as a structural recrystallization consisting in heating above A_{c3} and subsequent furnace cooling. Alloy steels, especially those containing strong carbide

forming elements, need higher annealing temperature than plain carbon steels. Full annealing heat treatment is carried out on nodular cast irons to produce ferritic matrix structures, by heating into the austenite-graphite region of the equilibrium phase diagram in Figure 2.14 and not into the full austenite region(except when prior homogenization is required

Annealing heat treatment depends almost entirely on two factors: (a) the formation of austenite, and (b) the subsequent transformation of the austenite at high sub-critical temperatures. The upper limit of holding temperature is generally restricted 30 or 50°C above A_{c3} in order to avoid grain growth.

Partial annealing is also referred to as inter-critical annealing. In this process, steel or cast iron is heated between the lower critical temperature(A_1) and upper critical temperature(A_3 or A_{cm}), holding for specific time followed by slow cooling.

The hypereutectoid steels are subjected to this treatment and the resultant microstructure consists of fine pearlite and cementite instead of coarse pearlite and a network of cementite at grain boundaries are observed in the case of full annealing. This process is less expensive than full annealing because of the involvement of low temperatures.

Sub-critical annealing is a process in which the maximum temperature to which steel is heated is always less than the lower critical temperature (A_1). The process does not involve formation of austenite, but thermally activated phenomena, such as recovery, recrystallization,

agglomeration of carbides and softening occur in this process. The three processes are represented graphically as shown in Figure 2.15.

The purpose of sub-critical annealing is to reduce hardness and increase ductility of cold worked steel so that further working may be carried out easily. Parts which are fabricated by cold forming such as stamping, extrusion, upsetting and drawing are frequently given sub-critical annealing treatment as an intermediate step (Rajan et al 1988).

2.6.3 Normalizing Heat Treatment

The general aim of this process is to refine the grain of steel, improve homogenization, enhance mechanical properties, such as hardness and tensile strength or to restore as-cast properties that have been modified by another heating process, such as graphitizing or the preheating and post-heating associated with repair welding (ASM, 1964). For both reasons, the mechanical properties especially in respect to ductility and toughness are somewhat better than annealed component.

Normalizing is applied to hypereutectoid steels and cast irons and involves heating the steel to a temperature range of 30-50°C above the upper critical temperature (A_{cm}), holding for a specified period and then cooling in air to room temperature. Heating temperature has a marked effect on microstructure and on mechanical properties such as hardness and tensile strength (Higgins, 1998).

The structures obtained by normalizing heat treatment will depend largely upon the thickness of cross-section as this will affect the rate of cooling and presence of alloy elements. Alloying elements have an important function in nodular iron castings that are to be normalized.

Moderate additions increase hardenability so that microstructures of fine pearlite may be obtained in normalized castings (ASM, 1964).

Moderate additions increase hardenability so that microstructures of fine pearlite may be obtained in normalized castings (ASM, 1964). Nickel is preferred as an alloying element because of its strengthening and as graphitizing effects. Molybdenum in combination with Nickel, is effective in increasing hardness and strength. The normalizing temperature is usually between 850°C and 950°C depending on composition.

Normalizing is commonly followed by tempering to reduce hardness for thin sections and relieve residual stresses for all castings. This is performed between 500 to 650°C for 1 hour per 2.54cm of section thickness (ASM, 1978). Normalizing also improves the machinability of hypo-eutectoid steels. The resultant surface finish is also superior to that obtained by machining an annealed component, since high ductility in the latter leads to a tendency to local tearing of the surface, (Higgins, 1998).

2.6.4 Hardening Heat Treatment

Certain applications demand high hardness values so that the components may be successfully used for heavy-duty purpose. Higher hardness values can be achieved by a process known as hardening. Hardening heat treatment consist of heating a metal above the upper critical temperature (A_3) holding it at this temperature for specific time followed by quenching in liquid medium such as brine, water, oil or salt baths to obtain a martensite structure.

The high hardness developed by this process, is due to the phase transformation accompanying rapid cooling (Higgins, 1998). Water

quenching of steel containing sufficient carbon produces an extremely hard structures called martensite which appears under the microscope as a means of uniform needle-shaped (acicular) crystals.

Less severe quenching gives rise to a structure known as bainite. The growth of bainite differs from that of pearlite in that ferrite nucleates first followed by carbide, whereas in pearlite it is the carbide which nucleates first (Higgins, 1998 and Nsely, 1970).

Still slower rates of cooling produce normal pearlite, the coarseness of the ferrite and cementite laminations depending upon the rate of cooling. Thus, as stated by Higgins (1998), normalization leads to the formation of a fairly fine grained structure, while annealing produced coarse grained structures.

The hardening temperature depends on chemical composition of an alloy and the presence of alloying elements. The temperature of heating for hardening of carbon steel and cast iron may be determined by reference to the constitution diagram. Hypoeutectoid steels are generally hardened at 30 – 50°C above the upper critical temperature A_3 , while hypereutectoid and eutectoid steels are hardened at temperatures 30 – 50°C above the lower critical temperature A_1 (Rajan et al, 1988).

Generally, ferrite and pearlite transform to austenite at hardening temperature for hypoeutectoid steels. This structure transforms to martensite on rapid cooling from hardening temperature. The martensite is responsible for high hardness value of quenched steel (Thelning, 1984).

High surface hardness developed by this process improves wear resistance of components such as gears, shafts, bearing, etc, but the process has a drawback in that rapid cooling results in developing high

internal stress and brittleness. Therefore, hardened parts are seldom used in as hardened condition. Hardening must therefore be followed by another process known as tempering, which reduces internal stress and makes the hardened steel relatively stable. Chemical composition of steel and cast iron largely determines the hardening temperature but in alloy steels the hardening temperature depends on the amount and nature of alloying elements.

2.6.5 Tempering Heat Treatment

The martensite formed during hardening is generally too brittle for the steel and cast iron to be put into practical use without first tempering it (Thelning, 1984). The extent of brittleness depends on the chemical composition and cooling rate. Hence it is necessary to re-heat or temper the quenched part so that internal stresses will be relieved and brittleness reduced (Higgins, 1998).

Medium carbon steels are also tempered but the temperatures are some what higher so that the strength and hardness are scarified to some extent in favour of greater toughness and ductility. Tempering of steel comprises heating previously hardened or normalized steel to a temperature below the transformation range (A_1) and cooling it at a suitable rate primarily to increase ductility and toughness (ASM, 1978).

As a rule, quenching is followed by tempering to relieve hardening stresses in steels and cast irons. In steels and cast irons, the best combination of strength and toughness, is obtained when a fully martensite structure is tempered. It should be borne in mind that in many steels and cast irons, the martensite reaction does not go to completion on

quenching and varying amounts of austenite are retained (ASM, 1964).

During tempering, martensite tends to transform to the equilibrium structure of ferrite and cementite. The higher the tempering temperature the more closely will the original martensite structure revert to this ferrite-cementite and so strength and hardness fall progressively whilst toughness and ductility increase. Proper tempering treatment results in optimum combination of mechanical properties. By choosing the appropriate tempering temperature a wide range of mechanical properties can be achieved in carbon steels.

Higgins (1998), Polukhin (1977) and Gulyaev (1980) explained the structural changes which take place during tempering of martensite containing more than 0.30% carbon that it occurs in four stages.

FIRST STAGE: 80 – 160°C. Existing martensite begins to transform to another form of martensite, containing only 0.25% carbon, together with very fine particles of a carbide, known as ϵ -carbide ($\text{Fe}_{2.4}\text{C}$), at this stage a slight increase in hardness may occur because of the presence of the finely dispersed but hard ϵ -carbide. Brittleness is reduced as quenching stresses disappear in consequence of the transformation. At 100°C the transformation proceeds very slowly but increases in speed up to 200°C.

The martensite that forms in this low temperature tempering in which c/a ratio is not equal but very close to unity is called temper martensite. Therefore, the first tempering transformation is the transformation of tetragonal martensite into almost cubic temper martensite (Gulyaev, 1980).

SECOND STAGE: This begins at about 250°C when any retained austenite begins to transform to bainite. This will cause the martensite needles to etch a darker colour and formally this type of structure was known as troostite. A further slight increase in hardness may result from the replacement of austenite to a much harder bainite.

THIRD STAGE: (300 - 400°C). At this higher temperature of tempering, a $\epsilon \longrightarrow \text{Fe}_3\text{C}$ carbide transformation takes place and this continues as the temperature rises. The remainder of the carbon begins to precipitate from the martensite and martensite structure gradually reverts to bcc ferrite. In other words, the third transformation of tempering involves definite changes which result in stress relaxation and transformation of carbide. The third transformation is finished at 400°C with the steel structure consisting of ferrite and cementite (Gulyaev, 1980). There is an overall increase in toughness with some loss in strength and hardness.

FOURTH STATE: This begins above 400°C. As a result, the precipitated cementite coalesces into larger rounded globules in the ferritic matrix (Higgins, 1998).

Due to the increased carbide precipitation which occurs at the temperature rises, the structure becomes softer, weaker and more ductile, though above 550°C strengths falls fairly rapidly with little rise in ductility.

2.6.6 Austempering Heat Treatment

In austempering, the microstructural end product of the steel, grey iron and nodular iron matrix is essentially bainite, a structure formed below the pearlite temperature range, but above the martensite range. As shown in Figure 2.16, the steel or cast iron is quenched from a temperature above the transformation range in a hot quenching bath and is maintained in the bath at constant temperature until transformation to bainite is complete. Austempering is an isothermal heat treatment that, when applied to ferrous materials produce a structure that is stronger and tougher than comparable structure, produced with conventional heat treatment. The initial quenching must be at a cooling rate greater than or equal to critical cooling rate so that no pearlite is formed (Bolton, 2002). The final cooling for bainite or ausferrite structure can be at any rate as no further changes take place (www.key-to-steel.com).

Figure 2.17 shows the above sequence of event on a time temperature transformation (TTT) diagram in all hot quenching processes the temperatures to which casting must be heated for austenitizing and the required holding times at temperature prior to quenching in the hot bath correspond to the temperature and times used in conventional hardening that is, temperature between 850°C and 950°C and holding times of 20min per 2.54cm of thickness (ASM, 1978).

Grey cast iron, nodular cast iron and plain carbon steels are usually quenched in nitrite/nitrate salt, oil or lead baths at 230 to 450°C for austempering (ASM, 1964). The temperature of the quenching bath and the composition of the ferrous alloys determine the holding time

required for maximum transformation to bainite. Alloy additions, such as nickel, chromium and molybdenum, increase the time required for transformation (ASM 1991). Bainite structure formed has lower strengths than martensite structure but have better ductility and impact toughness.

ASM (1964), Rajan (1988) and Higgins (1998) explained that austempering of high carbon steels, low alloy steels and nodular cast irons are performed essentially for either of both of two reasons. The first is to obtain improved mechanical properties particularly higher ductility or notch toughness at a given high hardness and the second is to decrease the likelihood of cracking and distortion.

Austempering consist of the following steps (Keough 1998):

1. Heating the casting to the austenitizing temperature in the range of (830 – 950°C) to produce fully austenite matrix.
2. Holding the part at the austempering temperature for a period of time sufficient to get the entire part to temperature and to saturate the austenite with carbon.
3. Quenching the part rapidly enough to avoid the formation of pearlite to the austempering temperature in the range of 230 – 450°C i.e. above Ms.
4. Austempering the part at the desired temperature for a time sufficient to produce a matrix of ausferrite or bainite.
5. Cooling the part to room temperature.

The austenitizing temperature controls the carbon content of the austenite, which in turn, affects the structure and properties of the austempered casting (Keough, 1998). High austenitizing temperature

increases the carbon content of the austenite, increasing its hardenability, but making transformation during austempering more problematic and potentially reducing mechanical properties after austempering. The higher carbon austenite requires a longer time to transform to ausferrite (www.key-to-steel.com).

Once the austempering temperature has been selected, the austempering times must be chosen to optimize properties through the formation of a stable structure of ausferrite. At short austempering times, there is insufficient diffusion of carbon to the austenite to stabilize it and martensite may form during cooling to room temperature. The resultant microstructure would have a higher hardness but lower ductility and fracture toughness. Excessive austempering times can result in the decomposition of austenite into ferrite and carbide (bainite) which will exhibit lower strength, ductility and fracture toughness. At highest austempering temperature 450°C as little as 30 minutes may be required to produce ausferrites. At 230°C as much as four hours may be required to produce the optimum properties (Keough, 1998). The process as compared to conventional quenching and tempering results in better ductility at high hardness levels improved impact and fatigue strength and freedom from distortion.

2.7. FACTORS CONTROLLING AUSTEMPERING REACTIONS

There are certain conditions which enable the austempering reaction to proceed spontaneously. When optimum strength and ductility are required, for austempering process to be properly carried out the following conditions must be adhered (www.key-to-steel.com). These are:

- (i) Austenitizing Temperature and Time
- (ii) Austempering Temperature and Time
- (iii) Section Size and Alloying

Heating specimens to austenitizing temperature is the first step of heat treatment processes to ferrous alloys. The most important determinant of matrix carbon content in ductile iron is the silicon content, as silicon content increases for a given austenitizing temperature, the carbon content in the matrix decreases.

The austenitizing temperatures between 845 and 925°C and austenitizing times of approximately 2 hours are normal and have been shown to be sufficient to recarburize the matrix fully (www.key-to-steel.com). Austenitizing temperature, through its effect upon matrix carbon has a significant effect on hardenability. The higher austenitizing temperature with its higher carbon content promotes increased hardenability, which causes a slower rate of isothermal austenite transformation.

The austempering temperature is the primary determinant of the final microstructure and therefore the hardness and strength of the austempered product. 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 (Keough, 1998). The initial increase in elongation occurs as stage I and elongation proceeds to completion at which point the fraction of austenite is a maximum. Further, austempering merely serves to reduce ductility as the stage II reaction causes decomposition to the equilibrium bainite product. Typical austempering times vary from 1 to 4 hours (www.key-to-steel.com).

As section size increases, the rate of temperature change between the austenitizing temperature and austempering temperature decreases. (Keough, 1998) used salt bath for thin, small parts only and investigates the influences of section size on austempering kinetics of austempered microstructure.

The alloying elements have great influence in austempering reactions. Elliot et al (1999), investigated the austempering kinetics of ductile iron alloyed with 0.39%Cu and 0.13Mo at 320,375 and 400°C after austenitizing at 870°C and results showed that higher austempering temperatures resulted to higher ductility. Lee et al (2003) investigated various ADI with high Si and Mn content, austenitized at 800 and 900°C. They concluded that the optimum Si/Mn ratio of ADI was approximately 1.5 for the best combination of hardness, tensile property, impact property and wear property.

The standard composition of ADI was selected to have 3.5%Si and 2.33%Mn. Effects of combined addition of 0.2%V and 0.1%Ti were proved to be effective for the improvement of wear property. It was concluded that the alloy combination of 3.5%Si, 2.33%Mn, 0.2V and 0.1%Ti austenitized

at 900°C was the optimized condition for mill balls applications.

2.7.1 Austempered Microstructure of Steels and Cast Irons

The microstructure formed in austempered steels is mainly bainitic matrix. According to Novikov, (1978) and Higgins, (1998), the bainitic transformation in steels normally takes place within the temperature range of roughly 500° – 250°C, that is, between the pearlitic and martensitic range. In other words, the bainitic transformation results in the formation of a mixture of α – phase (ferrite) and carbide, which is called bainite. Unlike pearlitic carbide, which has a lamellar structure, the carbide in bainite is in the form of very dispersed particles. The nature of this bainite varies with the temperature at which it is formed.

A distinction is made between “upper” and “lower” bainite to show that the bainitic transformation has taken place in either the upper or the lower region of the intermediate range. Novikov, (1978) and Rajan et al (1988) revealed that the microstructure of upper bainite consists of feather-like, whereas lower bainite has a martensite-like acicular or needle-like structure. In addition, the microstructure of lower bainite also shows the carbide inclusion present inside platelets of ferrite laths, while upper bainite carbide particles may be present between ferrite lathes or at grain boundaries (Gulyaev, 1980). The lower and upper bainite are shown schematically in Figure 2.19.

As observed, both upper and lower bainite are nucleated by ferrite which grows into bainitic plate on cooling to and holding at a bainitic temperature region provided for the molten bath. This plate is surrounded by austenite which is enriched in carbon.

The pearlite is nucleated by carbide which grows into pearlitic plate on cooling to and holding at a pearlitic temperature region.

Bainite structures have lower strengths than martensitic structures but have better ductility and impact toughness. Bainitic structure is stronger than pearlitic structure because the carbide is in the form of very disperse particles, while the carbide in pearlite has a lamellar structure.

Figure 2.19: Schematic Diagram showing the steps in the Formation of Upper and Lower Bainite.

2.8 ADVANTAGES OF AUSTEMPERING OVER CONVENTIONAL QUENCHING AND TEMPERING HEAT TREATMENT

The advantages of austempering over conventional heat treatment have long been recognized by research metallurgists such as Bain, Daveport and Grossman since the invention of the process more than 30 years ago. Austempering process produces a structure that is stronger and tougher than comparable structures produced by conventional heat treatment. It provides the following benefits (www.key-to-steel.com):

1. Increased ductility or notch toughness at a given hardness.
2. Reduced distortion which lessens subsequent machining time, stock removal and cost.
3. The components are produced with less dimensional changes by austempering than by conventional water quench and tempering.
4. The three process steps used in conventional quenching and tempering processes are eliminated in austempering, because in austempering process the components are simultaneously quenched and tempered.
5. The shortest over all time cycle to through harden within the hardness range of 35 to 55HRC with resulting saving in energy and capital investment (www.key-to-steel.com).

2.9 APPLICATIONS OF AUSTEMPERING PROCESS

Austempering is usually substituted for conventional quenching and tempering in order to improved mechanical properties particularly higher ductility or notch toughness at a given hardness and to decrease the likelihood of cracking and distortion (Rimmer, 1997).

One major area of application of austempering process is in the production of austempered ductile iron (ADI). The development and commercialization of ADI has provided the engineer with a new group of cast iron which offers the exceptional combination of mechanical properties equivalent to cast and forged steels and production costs similar to those of conventional ductile iron (Keough, 1998).

ADI has now become an established alternative in many applications that were previously the exclusive domain of steel castings, forgings, weldments, powdered metals and aluminium forgings and castings.

According to Keough (1998), ADI markets represent nearly all segments of manufacturing. ADI components are produced for heavy truck and bus components, light and truck components, railroad components, construction and mining components, agricultural, defense and machine tools.

The followings are specific components subjected to austempering:

Spring hanger brackets, shock brackets, U-bolt plates, brake calipers, knuckles, engine bracket and mounts, timing gears, cams, clutch collars and differential gears, annular gears; Light vehicle ADI applications include suspension components, spindles, cam shafts, crankshafts, engine mounts, knuckles and hubs (Rimmer, 1997).

Construction and mining components include all manners of collets, ring carriers, wear plates, sprockets, mill hammers, digger teeth, conveyor parts, cams, tool holders and rollers. Railroad components include suspension housings, friction wedges, track plates, nipper hooks and car wheels.

Farming and agricultural components include plow points, trash, cutters, seed boots, gears, sprockets, lifting arms, disk parts and harvesters, while defense industry ADI parts include truck links, armor ordinance and various hardware for trucks (Keough, 1998).

2.10 QUENCHING MEDIA FOR AUSTEMPERING

Quenching medium may be classified as a functions of their severity of quench or drastically and may be represented by the time necessary to cool the metal from a given high temperature to room temperature, (Beck 1961 and Higgins, 1998).

ASM, (1964), stated the two major quenching media used for austempering as: (1) Molten salt and (2) Molten lead

Molten salt is the quenching medium most commonly used in austempering because:

- a. It transfers heat rapidly
- b. It virtually eliminates the problems of vapour phase barrier during the initial stage of quenching.
- c. Its viscosity is uniform over a wide range of temperature.
- d. Its viscosity is low at austempering temperature (near that of water at room temperature), thus minimizing drag out losses, and
- e. It remains stable at operating temperature and is completely soluble in water, thus facilitating subsequent cleaning operation.

According to Higgins (1995), and ASM (1964), the most popular molten salts used for austempering are: (i) Sodium nitrate (ii) Potassium nitrate (iii) Sodium nitrite.

The formulations and characteristics of these Quenching salts are given in Table 2.3.

Table 2.5: Composition and characteristics of salts used for austempering (ASM, 1964).

| S/N | SALT | HIGH RANGE | MELTING POINT °C | WORKING RANGE °C |
|-----|-------------------|------------|------------------|------------------|
| 1. | Sodium nitrate | 40 to 50% | 220 | 200 – 650 |
| 2. | Pottasium nitrate | 40 – 50% | 220 | 200 – 650 |
| 3. | Sodium nitrite | 40-50% | 200 | 200 - 650 |

Source: (ASM, 1964).

2.10.1 SALT BATHS FURNACES.

Molten salt bath furnace (Figure 2.20) essentially consists of a container made of ceramic or metal. This container holds molten salt in which work-pieces are immersed. Molten salt bath furnaces can be used within a wide range of temperature (150 – 1300°C) (Rajan et al 1988). The salt may be contained in a pressed mild steel pot or alternatively, in a cast nickel-cromium alloy pot which although much more expensive gives a longer life. The bath should be stirred when fresh salt is added and it must be of sufficient capacity to avoid chilling of the salt during charging. The commonly used salts are nitrates, chlorides, carbonates, cyanides and caustic soda. The mode of heat transfer to the work-piece is by convection through the liquid bath. As the molten baths possess high heat capacity and heat is transferred by convection, the work-piece is heated up very quickly as compared to conventional furnaces (rate of heating is about five times higher in these furnaces.) These furnaces can be heated by fuel or electricity. Electrically heated salt bath furnaces are much more common in use. These can be classified into three types, namely, externally heated. Immersion heating element and immersed electrode type salt bath furnace.

Salt bath furnaces are used for various heat treatment operations such as cyaniding, liquid carburizing, liquid nitriding, austempering, martempering, hardening and tempering (Rajan et al 1988).

These furnaces as highlighted by Rajan et al (1988) and Higgins (1998) offer certain advantages over other furnaces. For example, all work-pieces are at uniform temperature and have identical surroundings. Such a condition results in better surface conditions and consistence and reproducible results. Since work-piece is in direct contact with the molten bath, there is no danger of oxidation and/or decarburizing. Molten lead may also be used as a medium for interrupted quenching, but its use has been restricted because of its high initial cost and because of its density and it also requires protection against oxidation and may give rise to a difficult cleaning problem when it adheres to the metal being quenched (Donald, 1985 and Khanna, 2002).

Hot mineral oil is seldom used for austempering because of its chemical instability and resulting change in viscosity at austempering temperatures. According to ASM (1964) and Novikov (1978) explained that due to its persistent vapour phase, oil is a slower quenchant than salt at elevated temperature and presents a fire hazard. However, recent studies reported by Larry (2001), Niall (2003) and Thomas (2003) have now proved that vegetable oil can be used as viable quenching medium for steels and cast irons. Traditionally, oil was used without additives. As a result it was slow in cooling and susceptible to oxidation.

Research carried out by "Hearth Bath Inc", to overcome these shortcomings by adding certain chemicals to the oil, i.e. antioxidants. In addition, the objective makes oil quenching more reliable and uniform and to control the vapour phase by starting the nucleate boiling stage sooner. They reported that, some of these troublesome components can be removed.

Although, synthetic oils have desirable features, such as low viscosity, high flash point and properties that would enable a wide application range, their cost remain high. Larry (2001) also explained that addition of antioxidants to the vegetable oils improved the chemical stability of the oils.

2.11 Properties of Quenching Media

The most important parameter in quenching and austempering operations is the stages or time periods in relation to temperature of manifestation of the different phases of boiling.

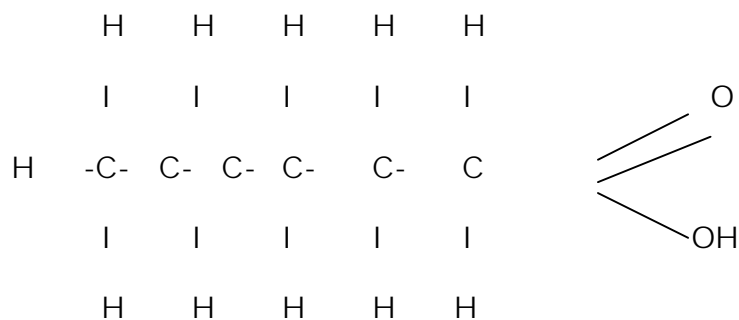
Quenching of steel and cast iron in liquid medium consist of three stages of cooling: the vapour phase, nucleate boiling and the convective stage. In the first stage, a vapour blanket is formed immediately upon quenching. Higgins, (1998) noted that this blanket has an insulating effect and heat transfer is slow since it is mostly through radiation. As the temperature drops, the vapour blanket becomes unstable and collapses, initiating the nucleate boiling stages. Heat removal is the fastest in this stage, due to the heat of vapouration and continues until the surface temperature drops below the boiling point of quenching medium. Further cooling takes place mostly thorough convection and some conduction.

Davies et al (1983) explained that, these stages depend on the vapouration characteristics of the medium and these in turn on the chemical and physical properties. The variables which affect the heat extraction ability of the liquid include viscosity, flash point, thermal conductivity, stability of oils oxidation resistances, temperature of bath, specific heat, fire point and immersion depth of sample, among others as assessed by Keith, (2004) and Larry, (2001).

The oils under investigations are local oil. Property analyses are however based on established parameters as used for known quenching mineral oils and solution of organic polymers.

2.11.1 Vegetable Oils

Vegetable oils are triglycerides, made up for three fatty acid chains linked to a molecule of glycerol. They are composed fundamentally of the elements carbon, hydrogen and oxygen. The fatty acids can be saturated or unsaturated. Unsaturated fatty acids have carbon-to-carbon double bonds. In saturated fatty acids all the carbon atoms are linked to two hydrogen atoms and there are not double bonds. The general formula of fatty oils is written as $C_nH_{2N-1}COOH$ (Kirschenbauer, 1984), and the structure is shown below:



Fatty oil (Unsaturated Molecules). Source: Kirshenbauer, 1984

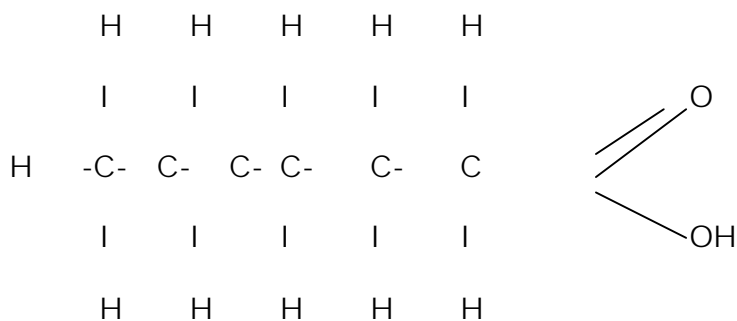
Nigeria is richly endowed with different types of vegetable oils, found in various parts of the country. While most of the vegetable oils produced in the country are mainly used in food, soap, candles and pharmaceutical industries.

Although, the country has different variety of vegetable oils which includes, Cottonseed oil, Groundnut oil, Palm kernel oil, Palm oil, Coconut oil, Castor oil and shea butter oil. Information available from the Raw Materials, Research and Development Council indicates that these vegetable based oils are available in abundance in different parts of the country.

2.11.2 Chemical Analysis of Vegetable Oils

The fatty vegetable oils consist predominantly of fatty acids in glycerin molecule. They are polar substances having very long carbon chains with one or more chemically active polar group at open end.

The properties of these oils may contain some unsaturated and saturated acids. These oils are composed fundamentally of the elements carbon, hydrogen and oxygen. Unsaturated fatty acids have carbon-to-carbon double bonds. In saturated fatty acid all the carbon atoms are linked to two hydrogen atoms and there are no double bonds. The general formula for fatty oils is written as $C_nH_{2n-1}COOH$ (Kivshenbouer, 1984).



Niall (2003) and Addison (2004) highlighted that the relative amount of these polar substance in fatty oils has been found to be a measured of degree of unsaturated fatty acid in the oil. Hence, the iodine that is absorbed by 100 grams of oils give a numerical value indicating the amount of reactive or polar radical in the oil

Naylor, (1967) indicated the presence of chemical such as sulphur, phosphorus or chlorine in the oils under investigation. These elements react chemically with metal surfaces under heat by forming metallic soap. Also, Addison,(2004) in his journey to forever, expressed different vegetable oils with their chemical analysis as shown in Tables 2.4 to 2.7.

The degree of saturation is indicated by the iodine value of the oil. Low iodine value oils are more saturated with fewer double bonds. High iodine value oil is more unsaturated with more double-bonds. Low iodine value oils have higher Cotane number and one more efficient quenchant than high iodine value oils, but they also have higher melting points and are usually solid at room temperature (Lauralice et al, 2006).

Table 2.6: Properties and Average cooling Rates of Quenching Media (Addison, (2004)

| S/N. | Quenching Medium | Cooling rates compared to water | Flash point °C | Fire point °C |
|------|----------------------|---------------------------------|----------------|---------------|
| 1. | 10% Sodium hydroxide | 2.01 | - | - |
| 2. | 10% Brine | 1.39 | - | - |
| 3. | 10% Caustic soda | 1.00 | - | - |
| 4. | Water | 0.44 | - | - |
| 5. | Palm oil | 0.36 | 185 | 207.2 |
| 6. | Fuel oil | 0.36 | 196.1 | 104 |
| 7. | Cottonseed oil | 0.33 | 234 | 315 |
| 8. | Neat's foot oil | 0.33 | 260 | 327.2 |
| 9. | Sperm oil | 0.29 | 260 | 305 |
| 10. | Castor oil | 0.29 | 260 | 328 |
| 11. | Peanut oil | 0.18 | 271 | 339 |
| 12. | Shear butter oil | 0.18 | 281 | 346.7 |
| 13. | Rapeseed oil | - | 290.6 | 362.8 |
| 14. | Still air | | 246 | 315 |

Table 2.7: Chemical Properties of Vegetable Oils Investigated (Ajala, 1982) and Addison, (2004)

| Oil | Free fatty acid(%) | Saponification value | Iodine value | Viscosity at 27°C (mm ² /sec) | Phosphorus content (mg/kg) |
|--------------|--------------------|----------------------|--------------|--|----------------------------|
| Groundnut | 0.11-0.24 | 223 - 230 | 92-115 | 225 | 0.067 |
| Shear butter | 0.06-0.65 | 167 - 178 | 68.2-68.7 | 257 | 0.07 |
| Cottonseed | 0.11-0.33 | 228 | 121.35 | 33.5 | 0.105 |

Table 2.8: Chemical Composition of the Vegetable Oils Investigated (Ajala, 1982)

| Fatty acids | Chain length | % in Cottonseed | % in Groundnut | % in Shea butter |
|-------------|--------------|-----------------|----------------|------------------|
| Myristic | 14.0 | - | - | - |
| Palmitic | 16.0 | 7.5 | 21.5 | 8.0 |
| Steraic | 18.0 | 4.5 | 2.0 | 35.0 |
| Caprylic | 8.0 | - | - | - |
| Capric | 10.0 | - | - | - |
| Lauric | 12.0 | - | - | - |
| Arachidic | 2.0 | 3.0 | - | - |
| Beharic | 22.0 | 2.0 | - | - |
| Oleic | 18.0 | 62.0 | - | 49.0 |
| Linoleic | 18.0 | 20.0 | 46.0 | 4.0 |
| Lignoceric | 24.0 | 2.0 | - | - |

Table 2.9: Characteristics of other Vegetable Oils (Addison, 2004)

| S/n | Oil | Iodine value | Flash point °C | Ash | Specific Heat | Viscosity at 27°C (mm ² /sec) | Catane |
|-----|-------------|--------------|----------------|-------|---------------|--|--------|
| 1. | Cottonseed | 100-121 | 264 | 99.95 | 0.2-0.4 | 33.5 | 55 |
| 2. | Corn oil | 115-124 | 277 | - | - | 34.9 | 53 |
| 3. | Coconut | 8-10 | - | - | - | - | 70 |
| 4. | Olive | 77-94 | - | - | - | - | 60 |
| 5. | Sunflower | 125-135 | 274 | - | - | 37.00 | 52 |
| 6. | Palm kernel | 12-18 | - | - | - | - | 70 |
| 7. | Palm oil | 44-50 | - | - | - | -39.6 | 65 |
| 8. | Pea nut | 80-106 | 271 | - | - | 29.7 | - |
| 9. | Castor | 82-88 | 260 | - | - | 27.2 | - |
| 10. | Linseed | 178 | 241 | - | - | 34.60 | - |
| 11. | Soybean | 130 | 254 | - | - | - | 53 |

CHAPTER THREE

3.0 EXPERIMENTAL PROCEDURE

3.1.0 Materials and Equipment

3.1.1 Materials

The experimental materials used in this investigation are: plain carbon steel, low alloyed carbon steel, grey cast iron and nodular cast iron. The two cast irons and low alloyed carbon steel in the form of 45mm diameter rods were obtained from National Metallurgical Research Centre (NMDC), Jos, Nigeria, while the plain carbon steel in the form of 22mm diameter rods were obtained from Mechanical workshop of Ahmadu Bello University, Zaria, Nigeria. The chemical composition of these materials were analysed at Nigerian Foundries Ltd. Ilupeju, Lagos, Nigeria and are contained in Table 3.1. The three quenchants (groundnut oil, shear butter oil and cottonseed oil) used in this study were sourced locally in Birnin Kebbi, Kebbi State, Nigeria and used as purchased

Table 3.1 Chemical Compositions of Materials Used

| Materials | Composition, % | | | | | | | | | | | |
|---------------------|----------------|------|-------|------|-------|-------|-------|-------|-------|-------|-------|------|
| | C | Mn | Si | Cr | P | S | Cu | Ni | Ti | Al | Mo | Mg |
| Medium carbon steel | 0.366 | 0.51 | 0.135 | - | 0.004 | 0.046 | 0.119 | - | 0.004 | 0.006 | - | - |
| Low alloyed steel | 0.492 | - | 0.047 | 0.26 | 0.137 | 0.018 | 0.139 | 0.043 | 0.005 | 0.06 | 0.013 | - |
| Nodular cast iron | 3.50 | 0.35 | 2.60 | - | 0.036 | 0.015 | - | 0.08 | - | - | - | 0.05 |
| Grey cast iron | 3.45 | 0.24 | 2.50 | - | 0.035 | 0.015 | - | - | - | - | - | - |

Table 3.2: Properties of Local oils Used (Ajala, 1982)

| Oil | Free Fatty acid, % | Saponification value | Iodine Value | Viscosity at 38°C mm ³ /sec | Specific heat, J/kg K | Phosphor content mg/kg | Flash point °C |
|--------------|--------------------|----------------------|---------------|--|-----------------------|------------------------|----------------|
| Groundnut | 0.11-0.24 | 223-230 | 91.73-114.53 | 225 | 0.2-0.4 | 0.067 | 271 |
| Cottonseed | 0.11-0.32 | 200-225 | 101.97-121.35 | 33.5 | 0.2-0.4 | 0.105 | 264 |
| Shear Butter | 0.06-0.69 | 167-178 | 68.2-68.7 | 257 | 0.2-0.4 | 0.07 | 281 |

3.1.2 Equipment

The equipment and tools used for this research study are as follows:

- a). Electrical muffle furnaces model BALCHYIC capable of attaining maximum temperature of 1200°C.
- b). An electrical oven model HRAEUS with a maximum temperature of 300°C.
- c). Optical Metallurgical microscope model METALLUXZ with an in-built camera.
- d). Hounsfield Tensiometer for tensile strength tests.
- e). Vickers and Rockwell hardness scale machine
- f). Avery Denison Impact tester
- g). Grinding and polishing machines
- h). Optical emission spectrometer (OES) model Ep 980 used for chemical analysis.
- i). Stove and pots for pre-heating the oils
- j). Metallic containers adapted as austempering bath.
- k). Thermocouple, Vanier caliper and tongs of different sizes.

3.2.0 Methodology

In this research work, tests were carried out to determine:

1. The effect of variations of austempering time on the austempered characteristics of nodular cast iron, grey cast iron, medium carbon steel and low alloyed carbon steel in some locally available vegetable oils (Groundnut, Cottonseed and Shear butter oils).

2. For this purpose, hardness measurements, tensile strength, percent elongation and impact strength tests were carried out in as received, annealed, normalized, and austempered conditions.
3. Metallographic analysis was carried out on each of the grade and for each condition.

The evaluation of the austempering characteristics of these grades of steels and cast irons was based on the tensile strength, percent elongation, impact strength and hardness values obtained from austempered specimens of the investigated materials subjected to the aforementioned temperature and time conditions.

3.3.0 HEAT TREATMENT OPERATIONS

3.3.1 Annealing

Annealing of as-received medium carbon steel, grey cast iron, nodular cast iron and low alloyed carbon steel samples were carried out at austenitizing temperature of 950°C, soaked at this temperature for a period of one hour and then furnace cooled to room temperature.

3.3.2. Normalising

Normalizing was carried for all other medium carbon steel, grey cast iron, nodular cast iron and low alloyed carbon steel specimens to be austempered as a pre-austempering treatment. This was done by heating the specimens to austenitising temperature of 950°C holding at that temperature for one hour and then cooled in air.

3.3.3 Austenitising Procedures

The usual objective of austenitising is to produce an austenitic matrix with as uniform carbon content as possible prior to thermal processing.

The normalized samples of ductile iron grey cast iron, medium carbon steel and low alloyed carbon steel, austenitized by heating the specimens at temperature of 950°C for one hour to achieved complete austenitisation of these materials, after which the samples were removed and treated as described below.

3.3.4 Austempering Procedure

Austempering was carried out for all other medium carbon steel, nodular cast iron, grey cast iron and low alloy carbon steel specimens, which had been previously normalized.

Specimens were packed into metallic container and charged into the furnace in batches. Each batch contains medium carbon steel, nodular cast iron, grey cast iron and low alloyed carbon steel specimens. Each batch of specimen was austenitized at 950°C and soaked for one hour followed by quenching in the various hot vegetable oil baths at a constant temperature of 250°C and held isothermally for one to five hours, at one hour intervals and thereafter cooled in air. Prior to austempering, oils were pre-heated with stove before being transferred to an electric oven. The three vegetable oils under investigation were used individually as austempering bath on all the specimens.

The heat treatment conditions and the total number of samples used are shown in Table 3.3.

Table 3.3 Heat Treatment Conditions for the Materials under Investigation

| Grade | Condition | Heat Treatment Temperature °C | Austempering Time (hr) | No of Sample |
|--------------------------|-------------------|-------------------------------|------------------------|--------------|
| Medium Carbon Steel | As-Received | - | - | 1 |
| | Annealed | 950 | - | 1 |
| | Normalized | 950 | - | 1 |
| | Austempered in: | | | |
| | - Groundnut oil | 250 | 1-5 | 15 |
| | - Shea butter oil | 250 | 1-5 | 15 |
| | - Cotton seed oil | 250 | 1-5 | 15 |
| Nodular cast Iron | As-Received | - | - | 1 |
| | Annealed | 950 | - | 1 |
| | Normalized | 950 | - | 1 |
| | Austempered in: | | | |
| | - Groundnut oil | 250 | 1-5 | 15 |
| | - Shea butter oil | 250 | 1-5 | 15 |
| | - Cotton seed oil | 250 | 1-5 | 15 |
| Grey Cast Iron | As-Received | - | - | 1 |
| | Annealed | 950 | - | 1 |
| | Normalized | 950 | - | 1 |
| | Austempered in: | | | |
| | - Groundnut oil | 250 | 1-5 | 15 |
| | - Shea butter oil | 250 | 1-5 | 15 |
| | - Cotton seed oil | 250 | 1-5 | 15 |
| Low alloyed Carbon Steel | As-Received | - | - | 1 |
| | Annealed | 950 | - | 1 |
| | Normalized | 950 | - | 1 |
| | Austempered in: | | | |
| | - Groundnut oil | 250 | 1-5 | 15 |
| | - Shea butter oil | 250 | 1-5 | 15 |
| | - Cotton seed oil | 250 | 1-5 | 15 |

3.4.0 Mechanical Properties Tests

The mechanical properties of heat treated medium carbon steel, nodular cast iron, grey cast iron and low alloyed carbon steel specimens were determined by tensile, impact and hardness tests.

3.4.1 Tensile Test

The tensile test was carried out on the medium carbon steel, nodular cast iron, grey cast iron and low alloyed carbon steel specimens that have been subjected to the under listed heat treatment as indicated in Table 3.3. Hounsfield Tensiometer testing machines was used to carry out all the tensile tests.

Figure 3.1 shows the dimensions of standard test bar used in these tests. The specimens were machined to the standard diameter size 5mm as specified in BS 2789:2002.

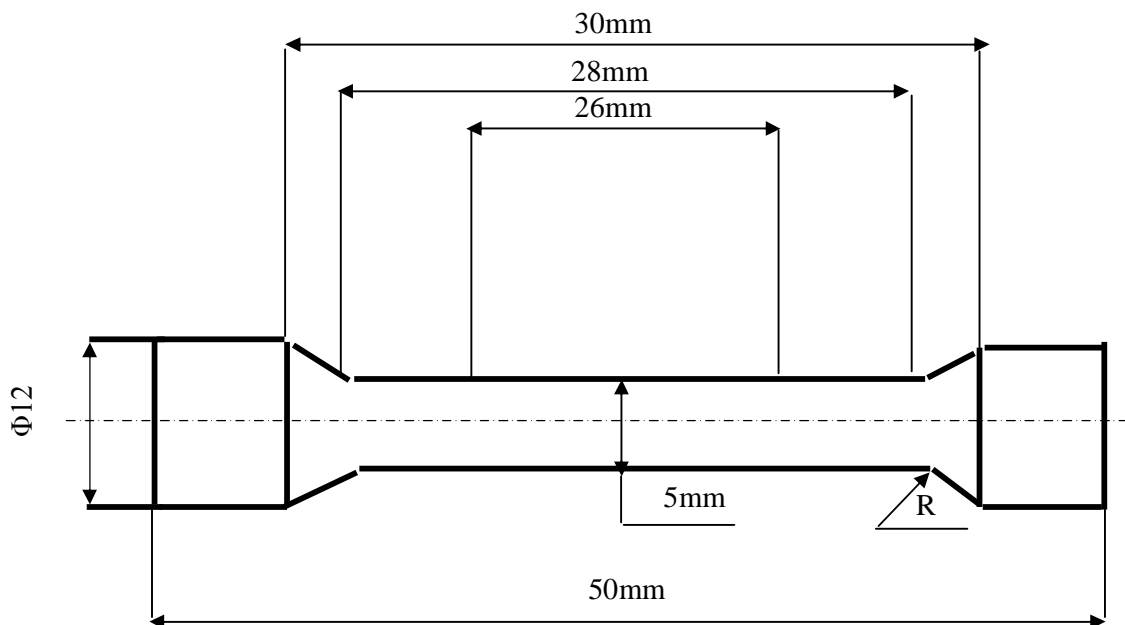


Figure 3.1: Tensile Test Specimen Used: (BS 2789:2002)

3.4.2. Impact Test

The impact test was carried out on the medium carbon steel, nodular and grey cast irons and low alloyed carbon steel specimens subjected to the under listed heat treatment conditions as indicated in Table 3.3. A 150 Joules capacity Avery Denison Charpy machine with a striking velocity of 5.24m/sec. was used to carry out all the impact tests.

Figure 3.2 shows the dimensions of standard test bar used in these tests. The specimen is fixed on horizontal position and the hammer strikes the impact specimen on the notched face. The energy absorbed is a measure of the impact strength of the material.

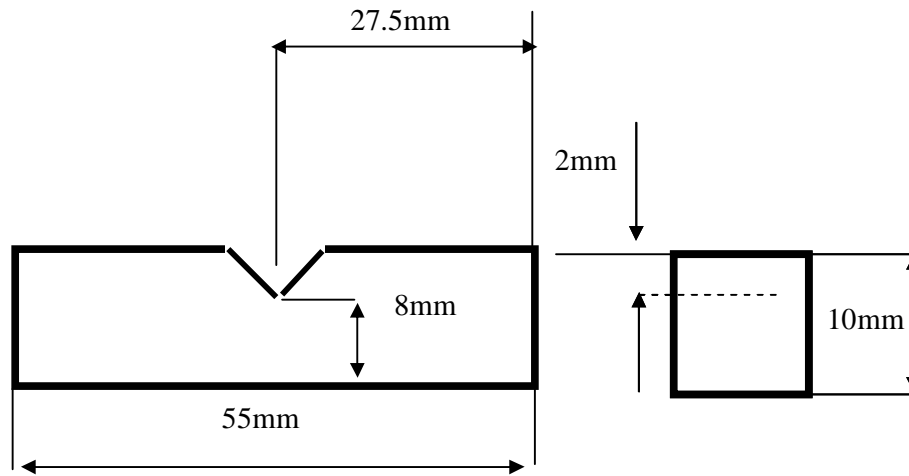


Figure 3.2 Charpy Test Specimen Used

3.4.3 Hardness Test

Hardness tests were carried out using a Vickers Hardness Testing machine with a diamond indenter, in the form of a square pyramid with an apex angle of 136° , and pressed less than 30kg for 15 seconds, into the surface of the medium carbon steel. For the austempered ductile iron, grey cast iron and low alloyed carbon steel, hardness test was carried out

by applying a load of 150kg on the diamond indenter and using Rockwell Hardness testing machine. The hardness values obtained were converted to Vickers hardness number. This was necessary because Vickers hardness measurement is not suitable for hardened materials.

Hardness measurements of this type require smooth surface preparation as well as ensuring a flat, firm seating of the specimen on the machine. The specimens of medium carbon steel, ductile cast iron, grey cast iron and low alloyed carbon steel under various heat-treated conditions were cut and mounted in fused bakelite powder for easy handling. Each surface of the specimen was prepared by grinding to obtain a smooth finish. The hardness values were obtained in at least 3 different points and the average of these was recorded. This is shown in Tables B2-B5

3.4.4 Metallography

Metallographic examination was carried out on the medium carbon steel, ductile cast iron, grey cast iron and low alloyed carbon steel specimens that have been subjected to the various heat treatment conditions as indicated in Table 3.3 Standard techniques of specimens preparation for the microstructure observation were adopted for metallographic samples mounted in bakelite using silicon carbide abrasive papers (240 to 600 grits) sizes. Following the polishing of specimens on a rotating disc and subsequent surface cleaning, etching of the polished specimen was done using 2% Nital (2% Nitric acid, HNO_3 , in ethyl alcohol) for 15-30 seconds to develop the structure. The structures obtained were photographed, and are shown in Appendix A (Micrographs A1 to A62).

3.5.0 EXPERIMENTAL DETAILS

3.5.1 Comparative Mechanical Properties

Tensile, hardness and impact tests were used to evaluate the mechanical properties of medium carbon steel, ductile cast iron, grey cast iron and low alloyed carbon steel austempered in the three selected vegetable oils (groundnut, shear butter and cottonseed oils) at 250°C for 1-5 hours. The results obtained are shown in Tables B2-B5 in Appendix B. Both the tensile, hardness and impact values were used to evaluate the austempered properties of all the selected materials.

3.5.2. Effect of Pre-austempering Heat Treatment on the Austempered medium carbon steel, low alloyed steel, nodular cast iron and grey cast iron.

Four fireclay ceramic crucibles were selected. One of these was packed with three specimens of medium carbon steel, another with three specimens of nodular cast iron, while the other was packed with three specimens of grey cast iron and the remaining with three specimens of the low alloyed carbon steel and treated as above. The four crucibles were loaded into the furnace and heated to temperature of 950°C and soaked for one hour and thereafter cooled in air. Results are shown in Table B1 (Appendix B).

3.5.3. Austempering Characteristic of Medium Carbon Steel, Low Alloyed Carbon Steel, Nodular Cast Iron and Grey cast iron.

Twelve fireclay crucibles were selected. Three of these were packed with five specimens of medium carbon steel; the other crucibles were packed with five specimens of low alloyed steel while the other three crucibles were each packed with five specimens of nodular cast iron. The remaining three

were each packed with five specimens of grey cast iron. The twelve crucibles and their contents were individually loaded in the furnace and heated to 950°C for one hour. At that temperature fifteen of each grade of specimen was quenched rapidly to the selected austempering temperature of 250°C, held isothermally for 1hr, 2hrs, 3hrs, 4hrs and 5hrs in each of the local vegetable oils (groundnut, cottonseed and Shear butter oils).

The results are shown in Tables B2-B5 (Appendix B) and Figures 4.1 - 4.12.

CHAPTER FOUR

4.0 RESULTS

4.1 COMPARATIVE MECHANICAL PROPERTIES.

The mechanical properties obtained for medium carbon steel, low alloyed steel, ductile cast iron and grey cast iron in the as-received, annealed, and normalized and austempered conditions are given in Appendix B.

For austempered medium carbon steel, the highest tensile strength and hardness values of 1020N/mm² and 353HV respectively were obtained from using hot shear butter oil as quenching medium. Austempered specimen in hot shear butter oil gave the highest percentage elongation and impact energy of 88 N/mm² and 101J respectively at 5hrs, while the minimum tensile strength hardness and impact values of 606 N/mm², 196HV and 35J respectively were obtained from austempered sample in hot groundnut oil.

However, austempered ductile cast iron samples in hot shear butter and groundnut oils recorded the highest tensile strength values of 104 N/mm² and 1030 N/mm² for 5hrs and 4hrs while the hardness values were 398HV and 412HV respectively. Sample that was austempered in hot cottonseed oil had the least tensile strength and impact values of 480N/mm² and 19J for 1hr respectively.

4.2: MICROSTRUCTURES OF HEAT-TREATED MEDIUM CARBON STEEL, DUCTILE CAST IRON, GREY CAST IRON AND LOW ALLOYED STEEL.

4.2.1. As received structures.

Micrographs A1, A19, A36 and A51 show the as-received structures of medium carbon steel, ductile iron, grey iron and low alloyed carbon steel respectively. The structure of medium carbon steel shows pearlite in ferrite matrix while the as-cast structure of ductile iron reveals graphite nodules in pearlite matrix. The structure of grey cast iron shows graphite flakes in pearlite matrix and that of low alloyed steel was ferrite-pearlite combination. All these structures obtained were as expected.

4.2.2. Annealed structures.

Micrographs A2, A20, A37 and A52 showed the annealed structures of medium carbon steel, ductile iron, grey iron and low alloyed steel respectively. As expected, the annealed structures of both grades of steels and cast irons were all ferritic.

4.2.3. Normalized structures.

The normalized structures of medium carbon steel, ductile cast iron, grey cast iron and low alloyed steel were shown in Micrographs A3, A21, and A53 respectively. These structures were all pearlitic as expected.

4.2.4. Austempered Microstructures.

The austempered medium carbon steel structures in hot groundnut, cottonseed and shear butter oils produced retained austenite at initial stage (1hr.) of austempering. However, with increase of austempering time bainite started to form from samples that were austempered in shear butter and groundnut oils.

Complete bainite structure was observed for austempered samples in hot shear butter oil for 3 and 4hrs (Micrographs A6 and A7) while in hot groundnut oil was for 3hrs (Micrograph A11).

Micrographs A22 to A35 revealed the structures of austempered ductile cast iron in groundnut, cottonseed and shear butter oils at different period of time. At initial stage of austempering a mixture of retained austenite and martensite was observed. With increase of austempering time to 3hrs, ausferrite structure with retained austenite was formed from austempered samples in hot shear butter and groundnut oils (Micrographs A23-A29). However, austempered samples in hot shear butter oil showed more ausferrite structure than other samples that were quenched in groundnut and cottonseed oils.

Micrographs A38 to A50 revealed the structure of austempered grey cast iron samples for 1 to 5hrs in hot groundnut, cottonseed and shear butter oils. Austempered grey cast iron samples in hot shear butter oil for 3 and 4 hrs (Micrographs A44 and A45) revealed martensite, retained austenite and ausferrite structures while austempered samples in groundnut and cottonseed oils shows martensite and retained austenite microstructure.

The austempered low alloyed steel samples in these oils produced retained austenite at initial stage. The microstructure of austempered samples of low alloyed carbon steel in groundnut and shear butter oils showed more retained austenite and martensite than bainite structure.

4.3 EFFECT OF AUSTEMPERING TIME ON THE HARDNESS OF AUSTEMPERED SAMPLES IN VARIOUS VEGETABLE OILS.

Figures 4.2, 4.7, 4.11 and 4.15 showed the effect of austempering time on the hardness of austempered medium carbon steel, ductile cast iron, grey cast iron and low alloyed carbon steel specimens in groundnut, cottonseed and shear butter oils at 250°C respectively.

4.4 EFFECT OF AUSTEMPERING TIME ON THE TENSILE PROPERTIES OF AUSTEMPERED SAMPLES IN VARIOUS VEGETABLE OILS.

Tables B2 to B5 showed the values of tensile strength and percent elongation of austempered samples of medium carbon steel, ductile cast iron, grey iron and low alloyed carbon steel respectively in hot groundnut, cottonseed and shear butter oils. Figures 4.1, 4.5, 4.9 and 4.13 showed the effects of austempering time on the tensile strength of medium carbon steel, ductile cast iron, grey cast iron and low alloyed carbon steel austenitized at 950°C and austempered at 250°C.

4.5 EFFECT OF AUSTEMPERING TIME ON THE IMPACT STRENGTH OF AUSTEMPERED SAMPLES IN VARIOUS VEGETABLE OILS

Tables B2-B5 in Appendix B showed the values of impact strength of the four different materials investigated. Figures 4.4, 4.8, 4.12 and 4.16 showed the effects of impact strength of medium carbon steel, ductile cast iron grey cast iron and low alloyed carbon steel samples austempered at 250°C.

4.6 EFFECT OF OILS COMPOSITION ON THE AUSTEMPERED SAMPLES.

The structure of fatty vegetable oils bears structural resemblance to that of paraffinic mineral oils, which are known as quenching oils. Fatty oils unlike paraffins, are unsaturated. They contain unsaturated fatty acids in addition to carboxylic group, which are also reactive (Ajala, 1982). The unsaturated content of the fatty oils, usually contributes to a strong molecular adhesion to a metal surface which results when the metal is brought into contact with the oil. Fatty oils contain saturated and unsaturated acids. They are composed fundamentally the elements carbon, hydrogen and oxygen.

Unsaturated fatty acids have carbon-to-carbon double bonds, while in saturated fatty acids all the carbon atoms are linked to two hydrogen atoms, and there are no double bonds. The iodine number of the oils varies according to the degree of unsaturation in the oil (Keith, 2004). This indicates the amount of reactive radicals present in the oil and by extension, the stability of the fatty acids molecules film that forms on the metal surface.

Cotton seed oil has the highest iodine value of 121 while shear butter oil has the least iodine values of 68.7 and hence more saturated. The relative content agrees well with the observed heat extraction properties of the oils.

Cottonseed oil according to these properties rated least effective as austempering oil due to the stability of the film, higher iodine value, lower flash point and higher saponification value. On the other hand, shear butter oil gave the best austempering characteristics as it had the least stable

film due to its low iodine value (Larry, 2001).

The results indicated that cottonseed oil like seed oils has very high degree of unsaturation due to a higher percentage of unsaturated fatty acids present in the oil, while shear butter oil has the least value of unsaturated fatty acids and therefore the most stable at high temperature.

The oils viscosity and flash point were given in Table 3.2. Shear butter oil was found to have the highest viscosity of 25.7mm²s and flash point of 281°C. This viscosity variation justifies the temperature dependence of the oil characteristics as reflected in the results. The higher the viscosity the lower the cooling rate and consequently the better the probability of forming bainite in the austempered cast iron or steel. This is due to the considerable thermal resistance that exists in viscous oils (Evan, 1984).

This confirmed to us that vegetable oils performance is composition dependent and this is true as it relates to the use of vegetable oil as replacements for mineral oil and salt bath for steel quenching applications (Lauralice at al 2006).

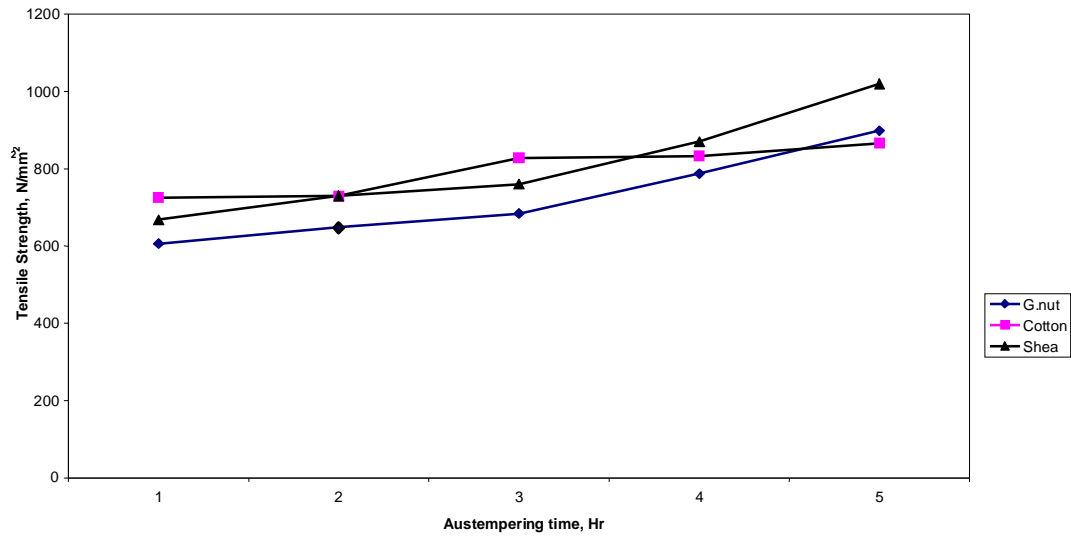


Fig 4.1 Effect of Austempering time on the tensile strength of medium carbon steel sample austenitized at 950 °C and austempered at 250 °C

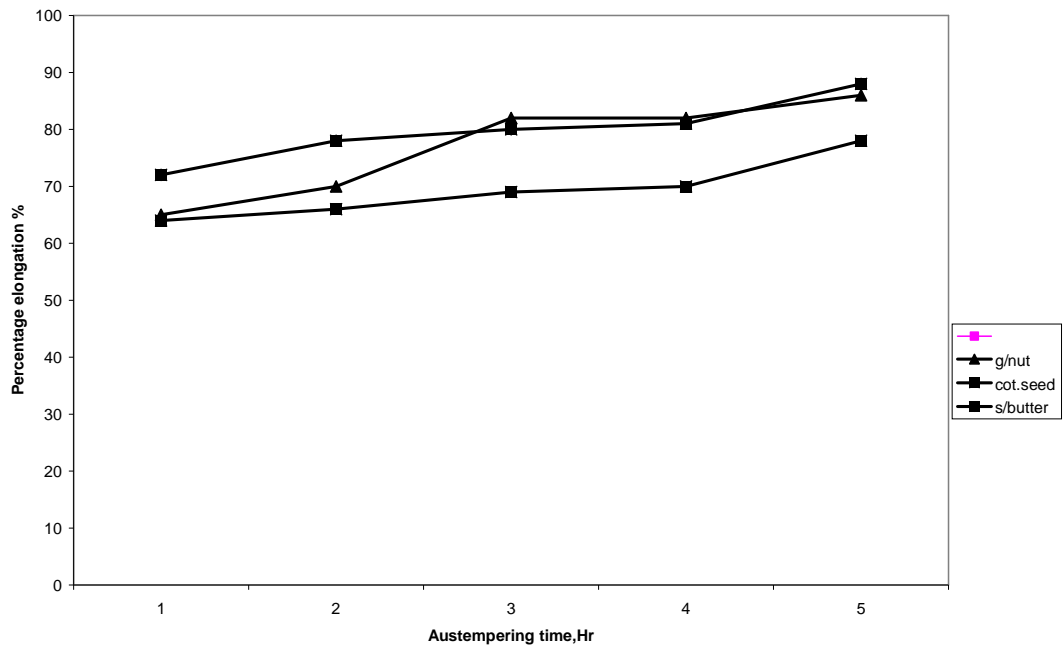


Fig.4.2. Effect of austempering time on percentage elongation of austempered medium carbon steel

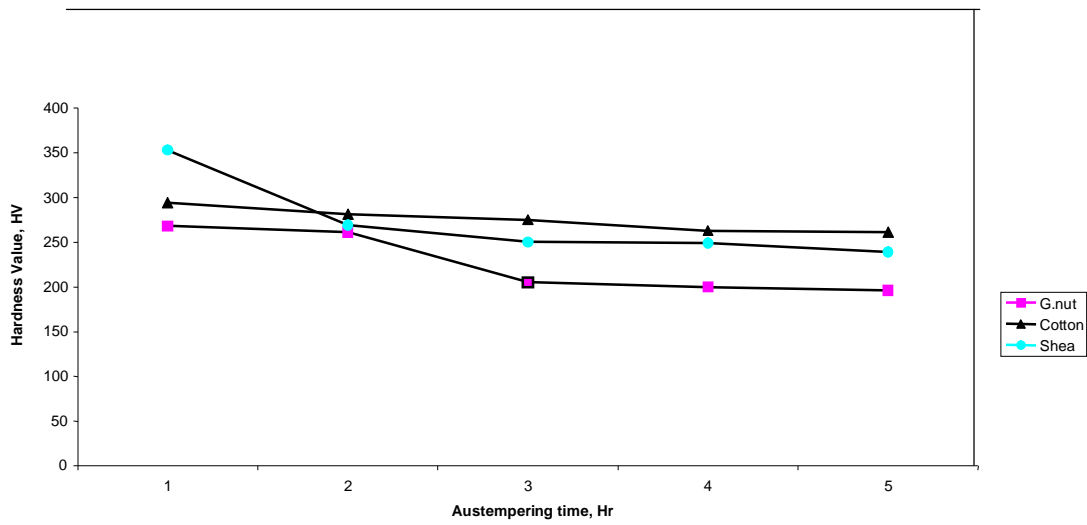


Fig 4.3 Effect of Austempering time on the Hardness of medium carbon steel sample austenitized at 950°C and austempered at 250°C

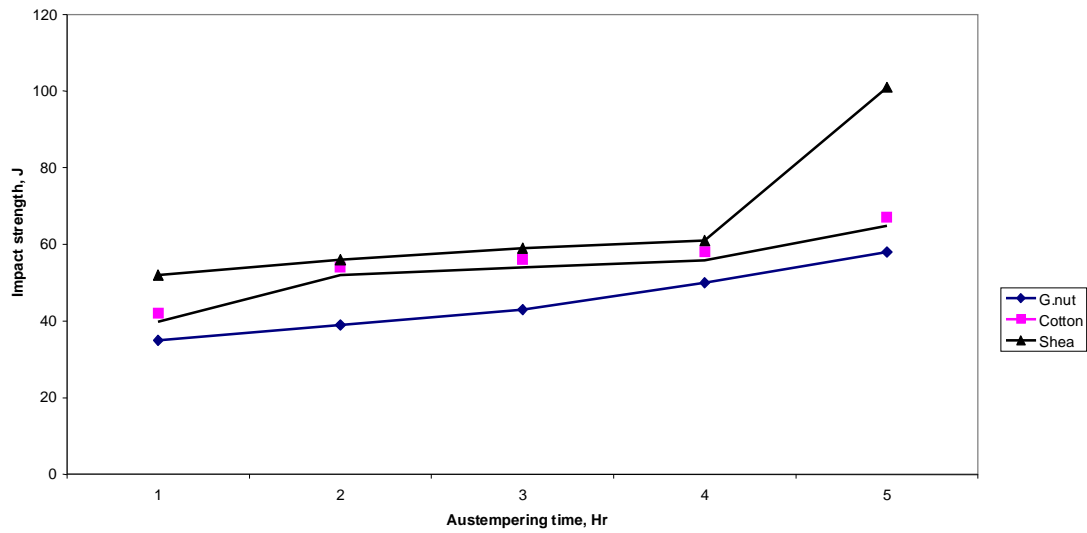


Fig 4.4 Effect of Austempering time on the Impact strength of medium carbon steel sample austenitized at 950°C and austempered at 250°C

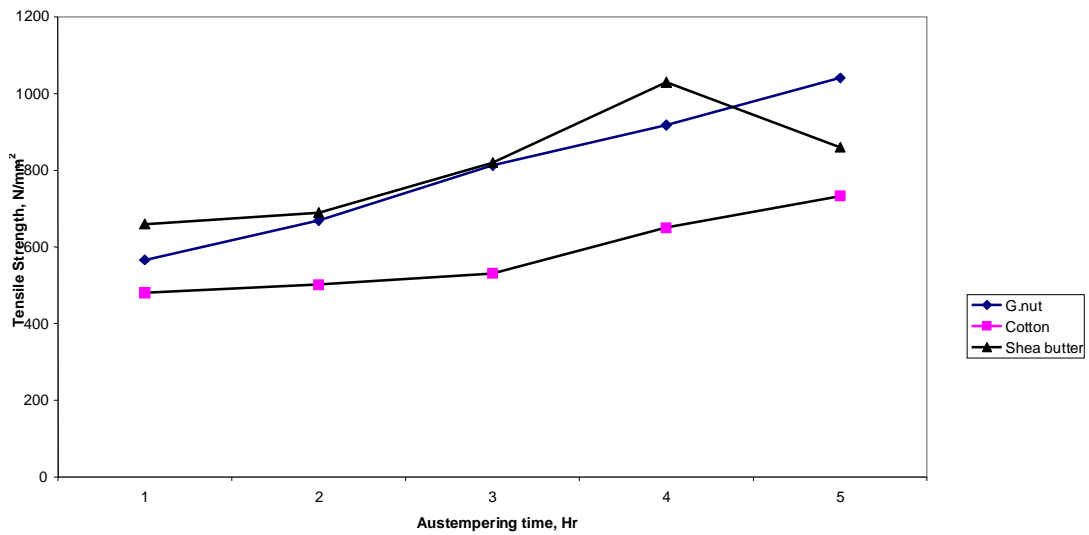


Fig 4.5 Effect of Austempering time on the tensile strength of nodular cast iron sample austenitized at 950⁰C and austempered at 250⁰C

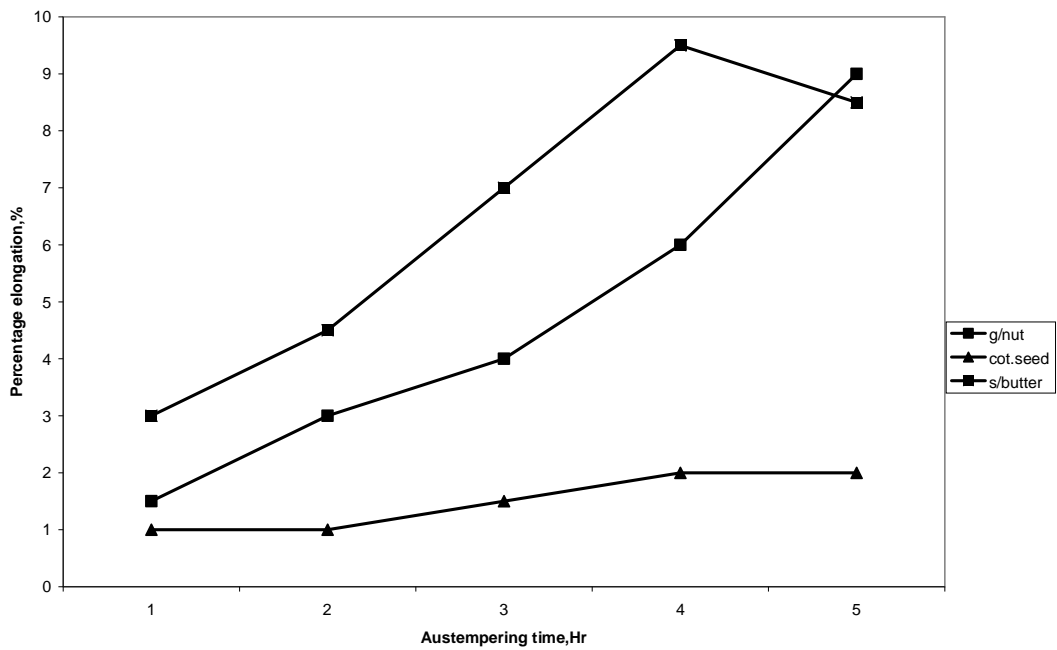


Fig.4.6. Effect of austempering time on percentage elongation of austempered ductile iron

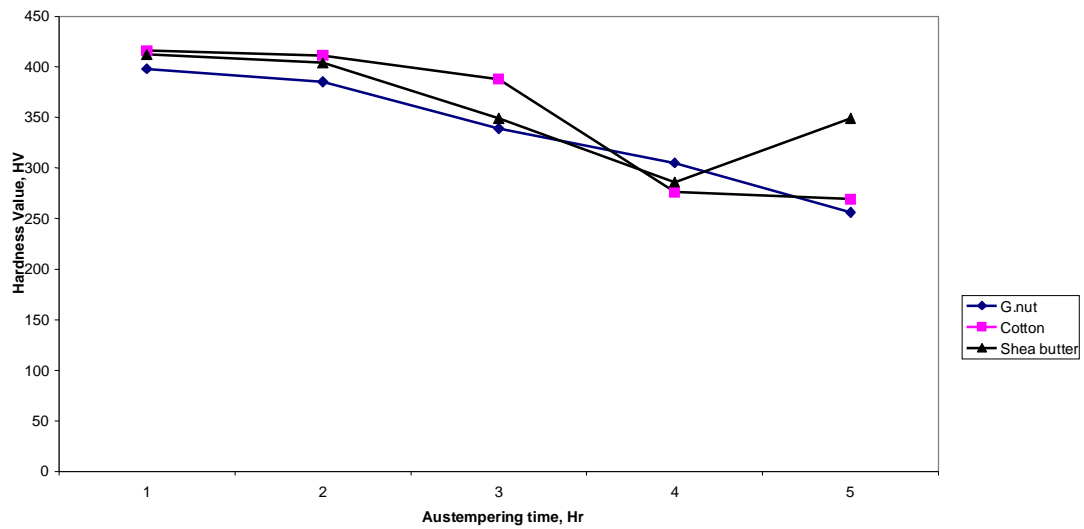


Fig 4.7 Effect of Austempering time on Hardness of nodular cast iron sample austenitized at 950⁰C and austempered at 250⁰C

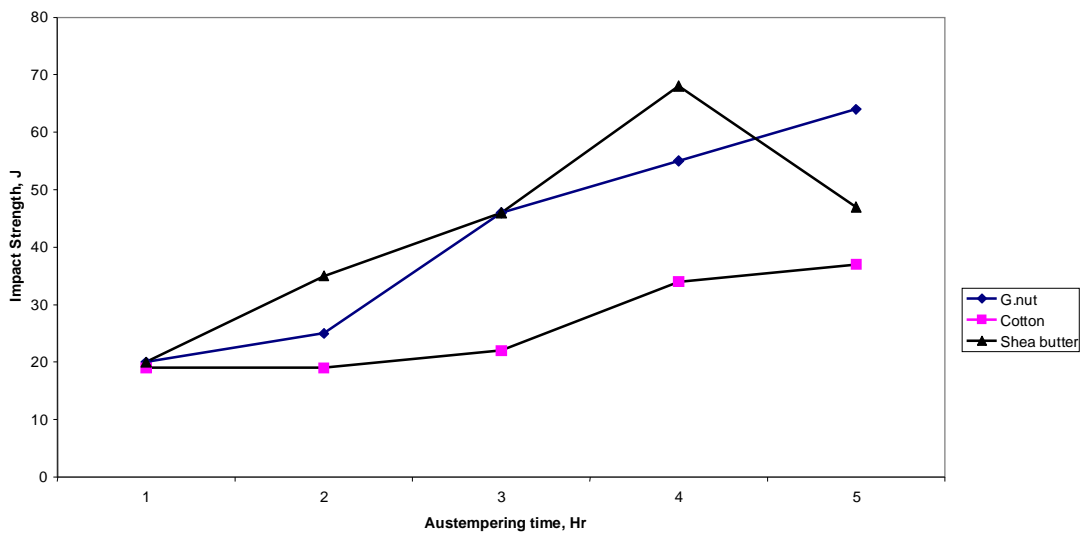


Fig 4.8 Effect of Austempering time on the Impact strength of nodular cast iron sample austenitized at 950⁰C and austempered at 250⁰C

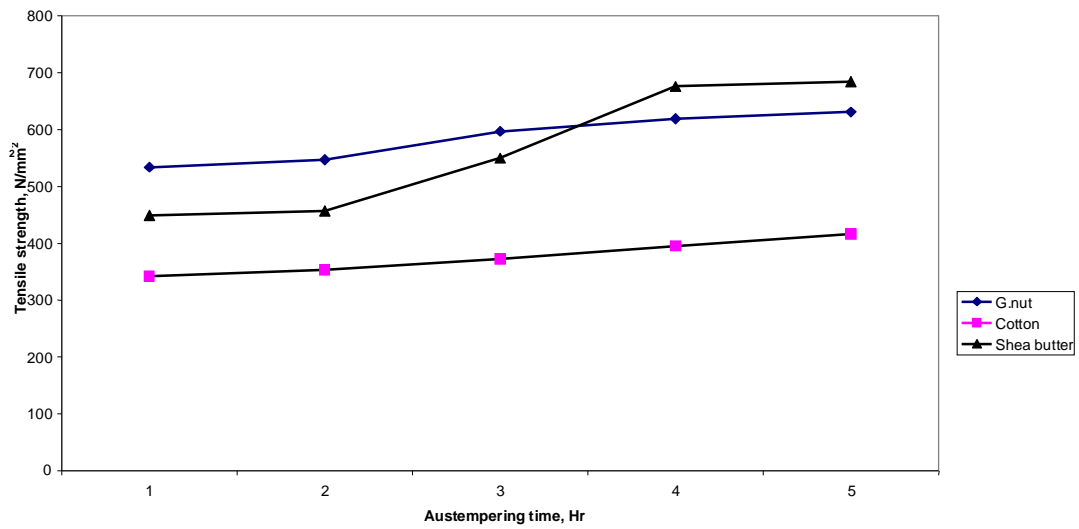


Fig 4.9 Effect of Austempering time on the tensile strength of gray cast iron sample austenitized at 950 °C and austempered at 250 °C

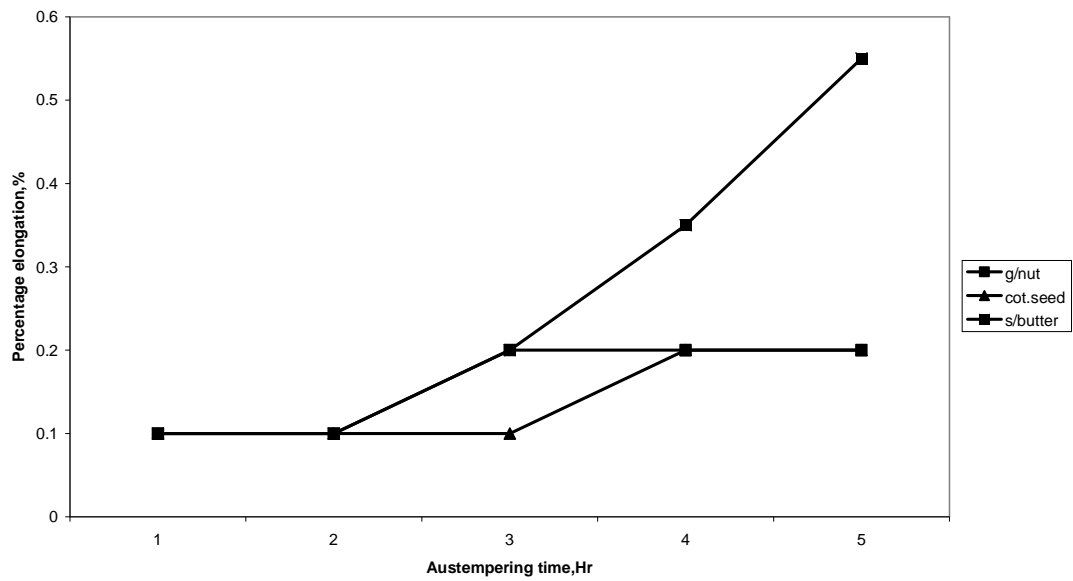


Fig.4.10 Effect of austempering on percentage elongation of austempered grey cast iron

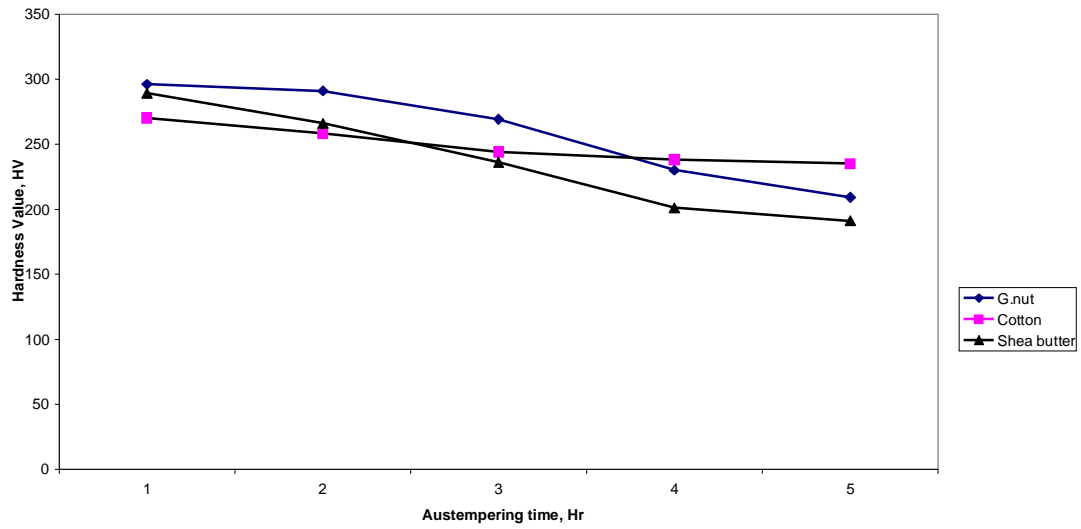


Fig 4.11 Effect of Austempering time on the Hardness of Gray cast iron sample austenitized at 950⁰C and austempered at 250⁰C

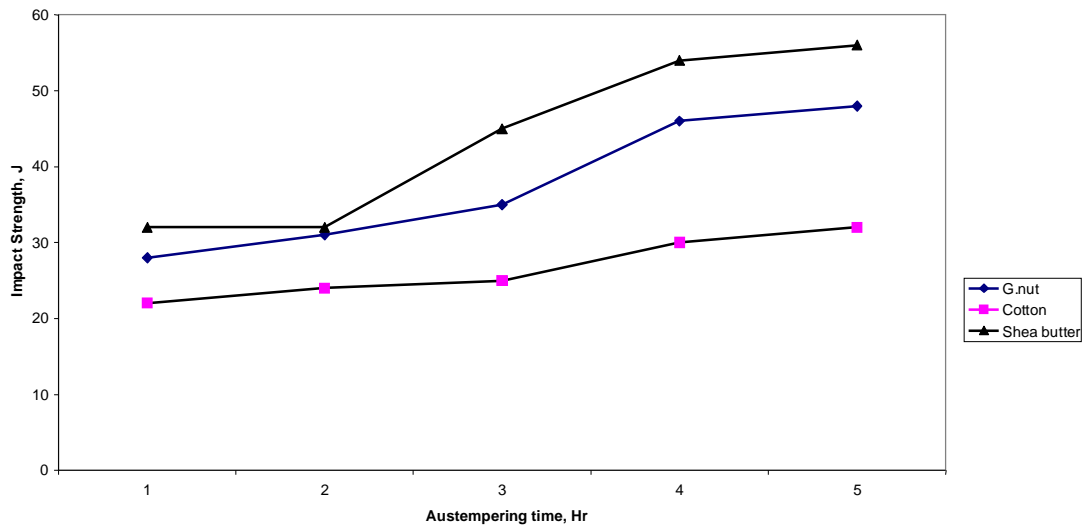


Fig 4.12 Effect of Austempering time on the Impact strength of gray cast iron sample austenitized at 950⁰C and austempered at 250⁰C

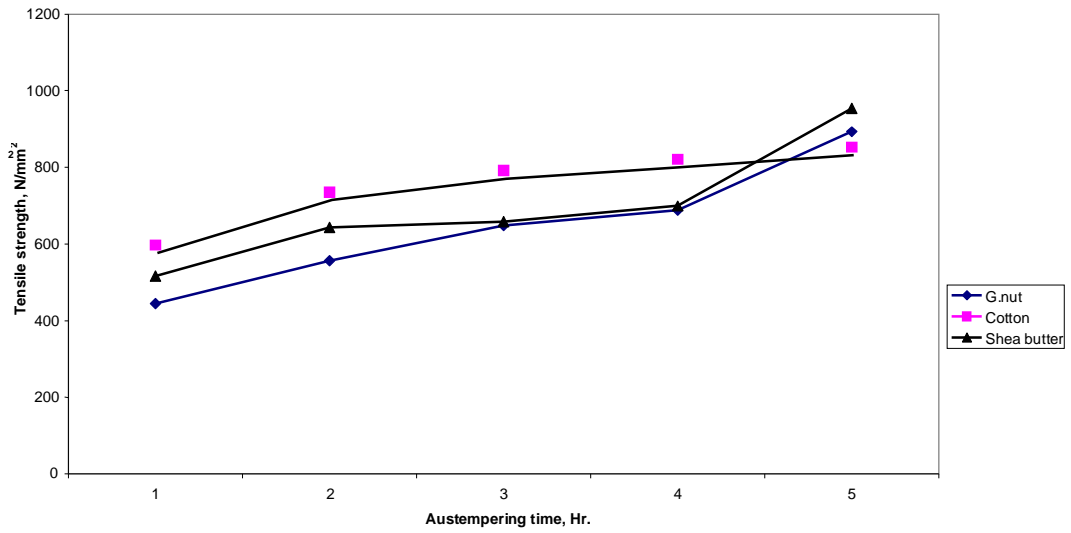


Fig 4.13 Effect of Austempering time on the tensile strength of low alloyed carbon steel sample austenitized at 950 °C and austempered at 250 °C

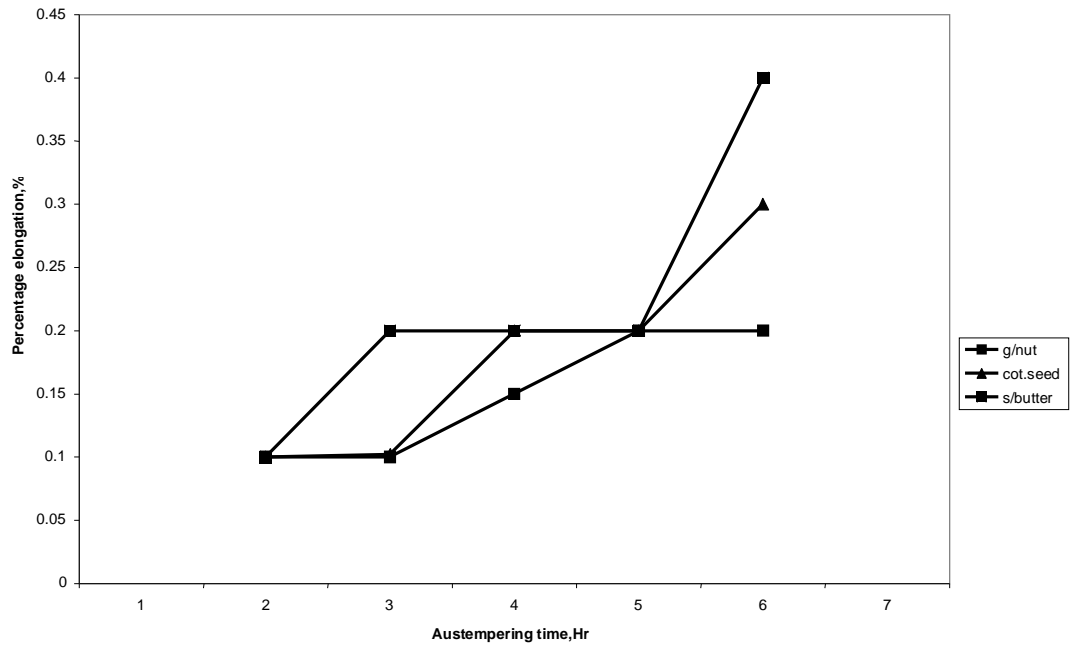


Fig.4.14. Effect of austempering time on percentage elongation of austempered low alloyed carbon steel

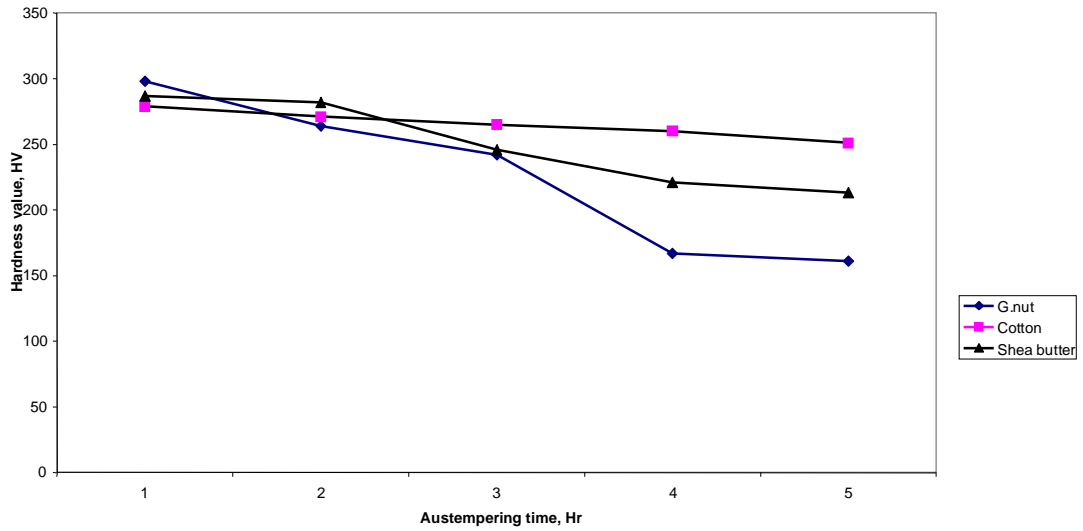


Fig 4.15 Effect of Austempering time on the Hardness of low alloyed carbon steel sample austenitized at 950^oC and austempered at 250^oC

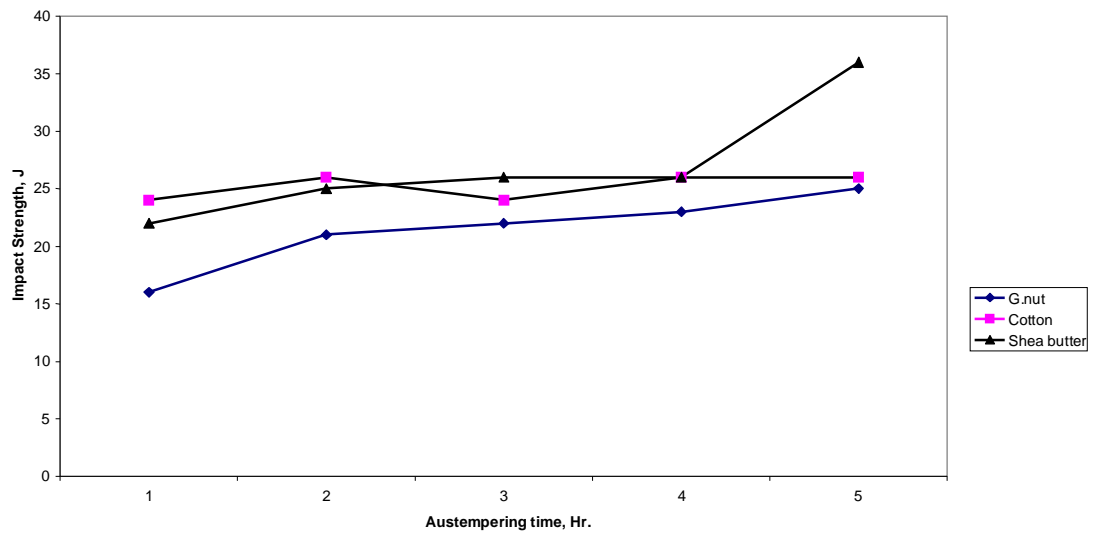


Fig 4.16 Effect of Austempering time on the Impact strength of low alloyed carbon steel sample austenitized at 950^oC and austempered at 250^oC

CHAPTER FIVE

5.0 DISCUSSION

5.1 MECHANICAL PROPERTIES OF AUSTEMPERED MATERIALS IN VARIOUS VEGETABLE OILS

The mechanical properties of austempered medium carbon steel, grey cast iron, ductile cast iron, and low alloyed carbon steel are shown in Tables B2, B3, B4 and B5 respectively. The results were compared favourably with those reported in the literature (Charles 1971, Bolton 2002 and ASM,1991). Higher hardness, tensile and impact values were observed in respect of austempered medium carbon steel and ductile iron than grey cast iron and low alloyed steel.

The maximum tensile strength values of 898N/mm², 866/mm² and 1020N/mm² at 5hours were obtained for samples austempered in groundnut, cottonseed and shear butter oils respectively while that of austempered ductile cast iron (ADI) recorded the highest tensile strength values of 1041N/mm², 733/mm² and 1030N/mm² at 5hours. These samples equally gave the highest impact and hardness values as compared to samples austempered in cottonseed oil. This is also reflected on the micro-structure as shown in Appendix A.

The higher strength, hardness and impact level obtained in austempered medium carbon steel and ductile cast iron is due to the formation of fine homogenous bainite structure in the austempered samples (see Micrographs A6, A7, A11, A12, A26, A29, A55, A57 and A61).The medium carbon steel and ductile iron samples austempered in hot shear butter and groundnut oils developed more bainite structure than samples austempered in cottonseed oil as quenchant.

5.2 EFFECT OF AUSTEMPERING TIME ON THE TENSILE PROPERTIES OF AUSTEMPERED SAMPLES IN VARIOUS VEGETABLE OILS.

Tables B2, B3, B4 and B5 show the values of tensile strength of austempered medium carbon steel, grey cast iron, ductile iron and low allowed carbon steel respectively in hot groundnut, cottonseed and shear butter oils. The load-elongation curves were used to determine the values of tensile properties of these materials. It has been observed that the tensile strength of these materials were dependent upon the austempering time.

Figure 4.1 shows the effect of austempering time on the tensile strength of medium carbon steel austempered in hot groundnut, cottonseed and shear butter oils. It shows that sample austempered in hot shear butter oil had the highest percentage of 88% at 5hours while sample austempered in cottonseed oil had the lowest value of 78% at 5hrs. Figures 4.5 and 4.9 show the graphs of tensile strength of ductile cast iron and grey cast iron samples austempered at 250°C in groundnut, cottonseed and shear butter oils. As observed, with increase of austempering time, the tensile strength also increased. The values of austempered grey cast iron samples in these oils were 631N/mm², 416 N/mm² and 684 N/mm² at 5hrs.respectively. It is known that for ductile iron the matrix constituents control the tensile properties (Keough, 1998).

Figure 4.5 shows the effect of austempering time on the tensile strength of ductile iron samples austempered at 250°C in groundnut, cottonseed and shear butter oils. The highest % elongation on ductile iron in these oils was 9%, 2% and 9.5% respectively (Figure 4.6).

This shows that samples austempered in groundnut and shear butter oils have the highest ductility.

Elliot et al (1999) reported that the tensile strength values shows a decreasing trend with increasing austempering temperature, while elongation values shows an increasing trend up to 673 K, and thereafter, decreases. The decrease in elongation above 673K is attributed to the change in the morphology of bainite (from a stronger lower bainite to a relatively weaker upper bainite). However, the present results relate the effect of austempering time in vegetable oils as quenchants.

5.3 EFFECT OF AUSTEMPERING TIME ON THE HARDNESS OF AUSTEMPERED SAMPLES IN VARIOUS VEGETABLE OILS.

Figure 4.3 shows the effect of austempering time on the hardness of medium carbon steel austempered in various hot vegetable oils (cottonseed, groundnut and shear butter oils) at 250°C. It shows that with increase in austempering time, the hardness values decreased. This is attributed to the increase in the formation of bainite structure as austempering time increases from 1-5hrs. The drop in hardness at 2-4hrs using hot shear butter and groundnut oils is attributed to the formation of more bainite. At this time austenite has more time to transform to bainite.

Figure 4.7 shows the effect of austempering time on the hardness of austempered ductile cast iron. The hardness decreased slightly with increased austempering time. The higher hardness values obtained at the austempering time of 1hr may be due to the insufficient time for reasonable austempering reaction to have occurred before the sample was removed from the hot vegetable oils and cooled in air leading to high quantity of retained

austenite. The retained austenite then transformed to martensite on cooling to room temperature. The drop in hardness is attributed to the onset of the stage 1 reaction ($\gamma \rightarrow \alpha + \gamma_{hc}$). Austenite has more time to transform to ausferrite hence leading to a decrease in the quantity of retained austenite. As the austempering time was further increased, the hardness increased for sample austempered in shear butter and cottonseed oils due to formation of cementite from the stage ii austempering reaction ($\gamma_{hc} \longrightarrow \alpha + Fe_3C$).

Figures 4.11 and 4.15 show the effect of austempering time on the hardness values of austempered grey cast iron and low alloyed carbon steel samples. The graphs revealed similar decreasing trend of hardness values as austempering time increases in all the selected vegetable oils for quenching. This decrease is gradual for each vegetable oil.

5.4 EFFECT OF AUSTEMPERING TIME ON THE IMPACT STRENGTH OF AUSTEMPERED MATERIALS IN VARIOUS VEGETABLE OILS

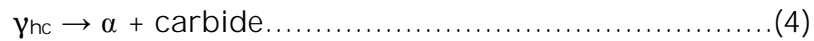
Appendix B (Tables B2 – B5) shows the values of impact energy of the 4 types of materials. Tables B2 shows the impact energy values of austempered medium carbon steel samples in groundnut, cottonseed and shear butter oils at 250°C. As observed, the impact values increases with increase of austempering time. This increase is attributed to the progression of austempering reaction which might have caused considerable quantity of austenite to transform to bainite. This is completely in agreement with the work of Tomita (1990) and Elliot (1999). The highest impact value was observed for sample austempered in shear butter oil at 5hrs while the least impact value was for austempered specimen in hot groundnut oil. The low impact strength obtained at 1hr is due to the insufficient austempering holding time to transform austenite to bainite

instead of retained austenite or martensite.

Figures 4.8, 4.12 and 4.16 show the effects of austempering time on the impact strength of austempered grey cast iron, ductile iron and low alloyed cast steel respectively. For austempered ductile and grey cast irons samples, there was progressive increase of impact strength with increase of austempering time. This increase is attributed to the progression of stage I austempering reaction which caused considerable quantity of austenite to transform to bainite. This reaction can be expressed as:



As the austempering temperature is held for too long, then the carbon enriched austenit (γ_{hc}) further decomposes in to ferrite and carbide.



This is also in agreement with Tomita (1990) and Elliot (1999). This refers to the increase in % elongation of the ductile iron if compared with grey cast iron and low alloyed steel. The low alloyed steel shows the lowest value of impact energy due to the presence of carbide forming elements such as Cr and Mo.

CHAPTER SIX

6.0 CONCLUSIONS AND RECOMMENDATIONS

6.1 CONSLUSIONS

This work is centered on potentials of using some vegetable oils as quenchants for austempering of steels and cast irons. From the results obtained, the following conclusions could be drawn:

1. The results indicate that after austempering of medium carbon steel, and ductile iron a relation between microstructure and mechanical properties exists.
2. Hot shear butter and groundnut oils were able to cause formation of "bainite structure" at 250°C with medium carbon steel, ductile cast iron and low alloyed steel.
3. A complete bainite structure was obtained from austempered medium carbon steel in hot shear butter oil at 3hrs and 4hrs while at 4hrs, complete bainite structure was formed using groundnut oil as quenchant for ductile cast iron.
4. The results of tensile strength, percent elongation, hardness and impact tests shows that the use of shear butter and groundnut oils as austempering quenching bath gave increase in tensile strength, ductility and impact toughness while the hardness values decreased marginally in the case of medium carbon steel and ductile cast iron.
5. The low impact and percentage elongation values obtained for austempered low alloyed steel and grey cast iron samples was due to the formation of martensite and retained austenite structure.
6. The austempering time of 3-4hrs were found adequate to

develop the necessary enhanced mechanical properties in medium carbon steel, ductile cast iron and low alloyed carbon steel in shear butter and groundnut oils.

7. The austempered grey cast iron samples showed little response to austempering process using any of the three vegetable oils as quenchants.
8. ADI resembled ASTM 897-06 grade 1050-700-7 because the mechanical properties are closed to this grade of ADI.

6.2 CONTRIBUTION TO KNOWLEDGE.

- 1) Bainite and Ausferrite structures were formed in steels and cast irons respectively.
- 2) Additional applications of vegetable oils in austempering process as quenchants in metallurgical engineering.
- 3) The optimum austempering time of four hours was established for the vegetable oils quenchants.

6.3 RECOMMENDATIONS FOR FURTHER STUDIES

The following recommendations are made for further studies.

1. The effect of austempering temperatures from 300°C and 400°C on these oils with addition of antioxidants is recommended.
2. The fatigue strength characteristics of steels and cast irons austempered in these selected vegetable oils and other available oils should be investigated.

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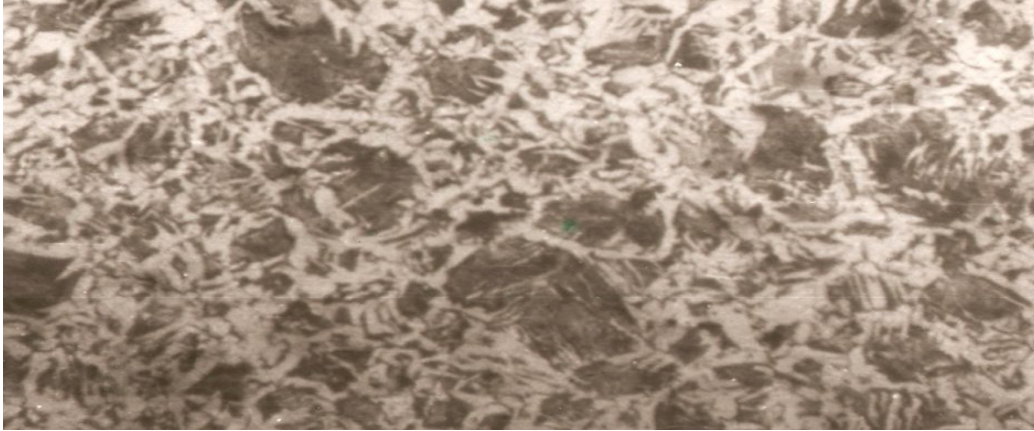
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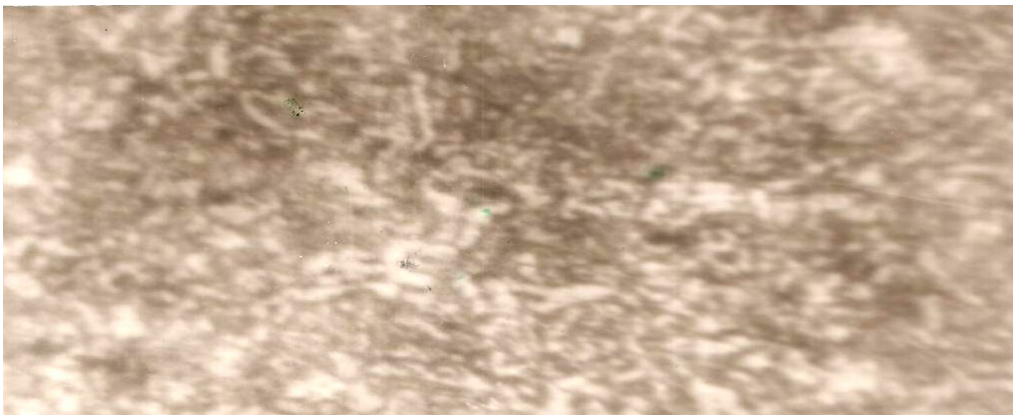
APPENDIX A



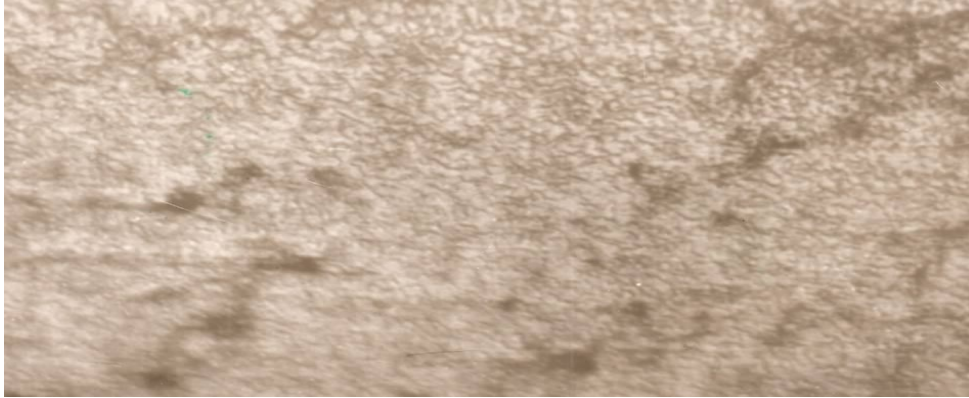
Micrograph A1: As-received structure of medium carbon steel. The structure consists of ferrite (white) and pearlite (dark). (x200)



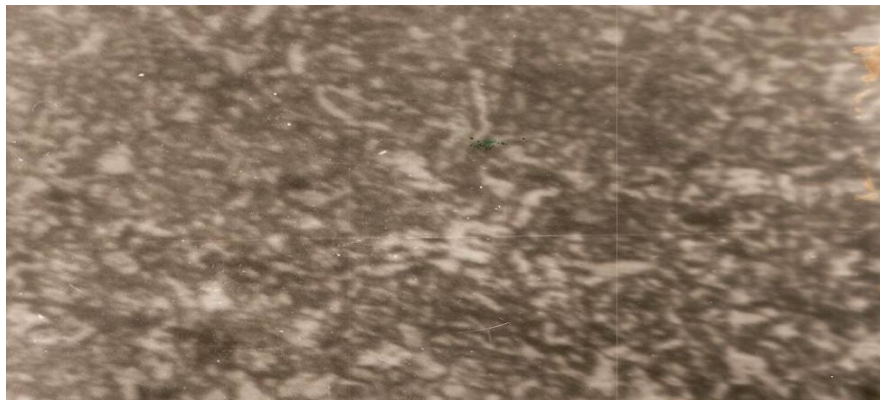
Micrograph A2: Annealed structure of medium carbon steel. The structure indicate ferrite (white) and the pearlite (dark). (x200)



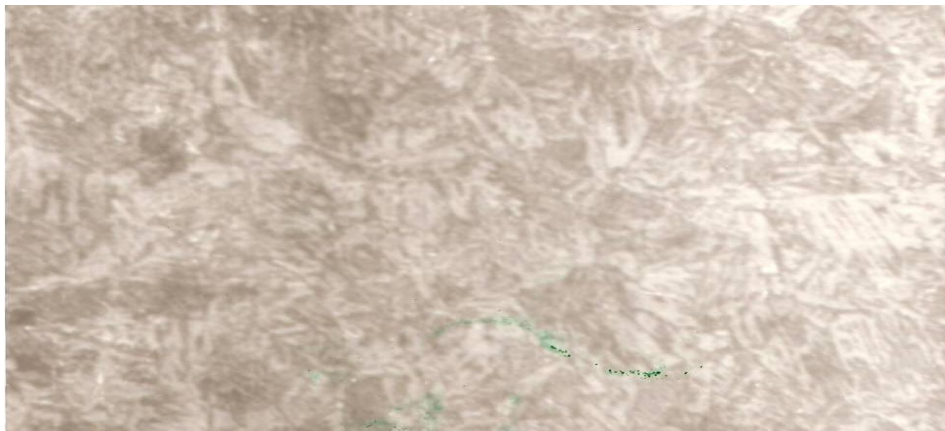
Micrograph A3: Normalized structure of medium carbon steel. The structure consists of pearlite (dark) in ferrite matrix (white). (x200).



Micrograph A4: Austempered structure of medium carbon steel in hot shea butter oil at 250⁰C for 1hr. The structure shows a mixture of martensite and retained austenite. (x200).



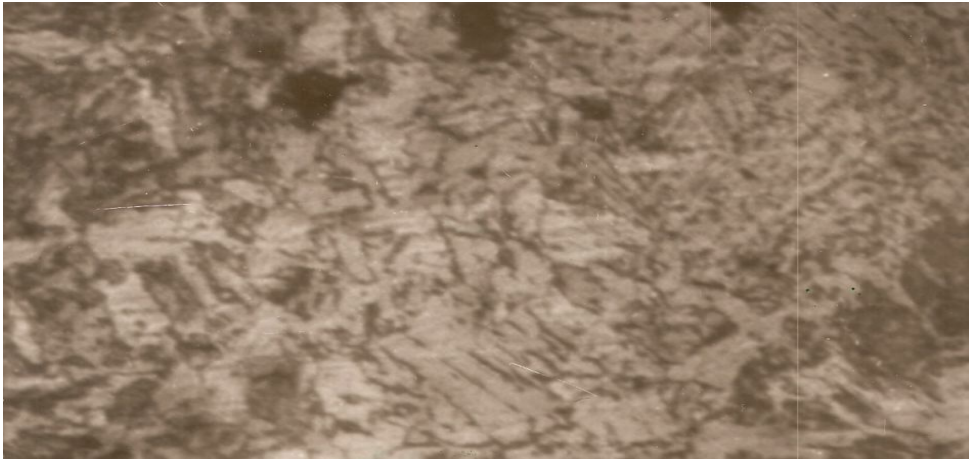
Micrograph A5: Austempered structure of medium carbon steel in hot shear butter oil at 250⁰C for 2hrs. The structure consists of bainite (white) and martensite (dark). (x200)



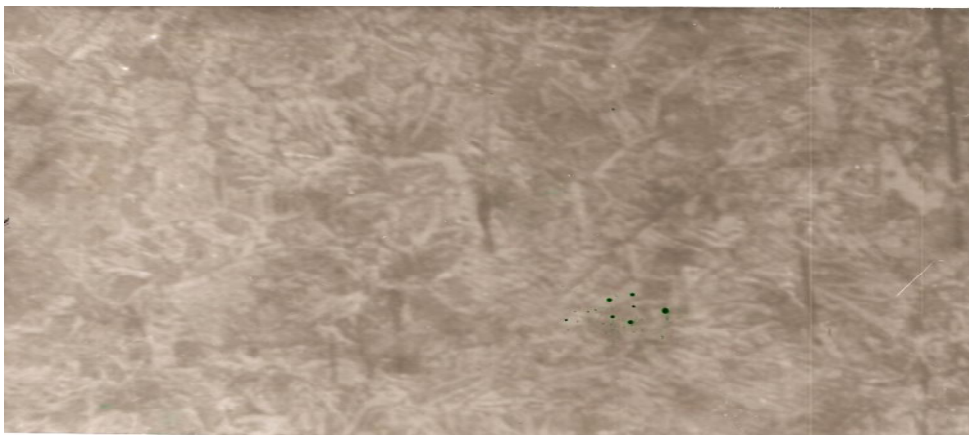
Micrograph A6: Austempered structure of medium carbon steel in hot shear butter oil at 250⁰C for 3hrs. The structure indicates full bainite structure (white feathery). (x200)



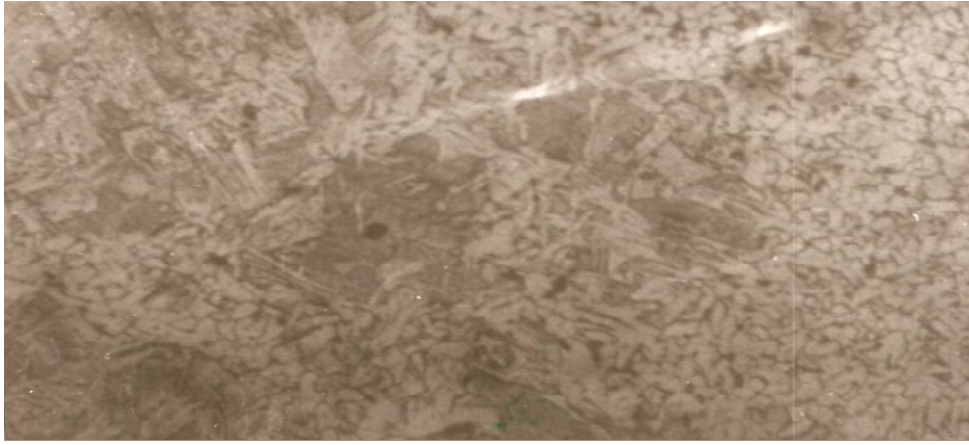
Micrograph A7: Austempered structure of medium carbon steel in hot shear butter oil at 250°C for 4hrs. The structure consists of complete bainite structure (white feathery). (x200)



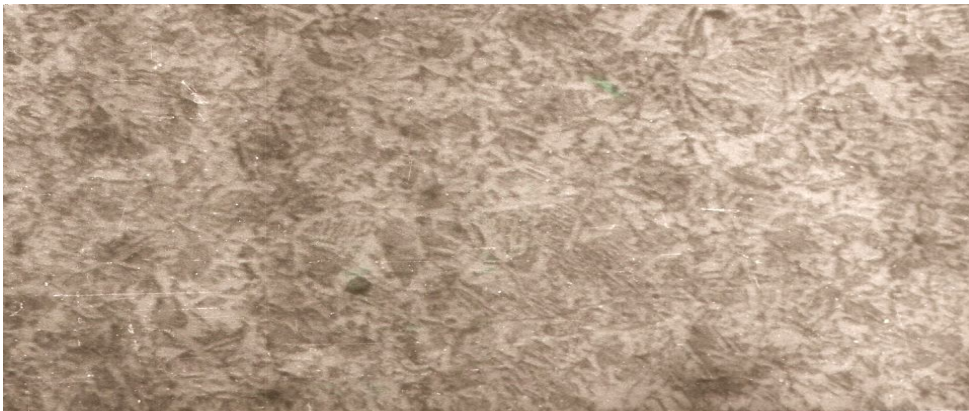
Micrograph A8: Austempered structure of medium carbon steel in hot shear butter oil at 250°C for 5hrs. The structure shows same as A7 but more bulky. (x200)



Micrograph A9: Austempered structure of medium carbon steel in hot groundnut oil at 250°C for 1hr. The structure consists of bainite (white) and retained austenite (dark). (x200)



Micrograph A10: Austempered structure of medium carbon steel in hot groundnut oil at 250°C for 2hrs. The structure shows a mixture of bainite (white) and martensite (dark). (x200)



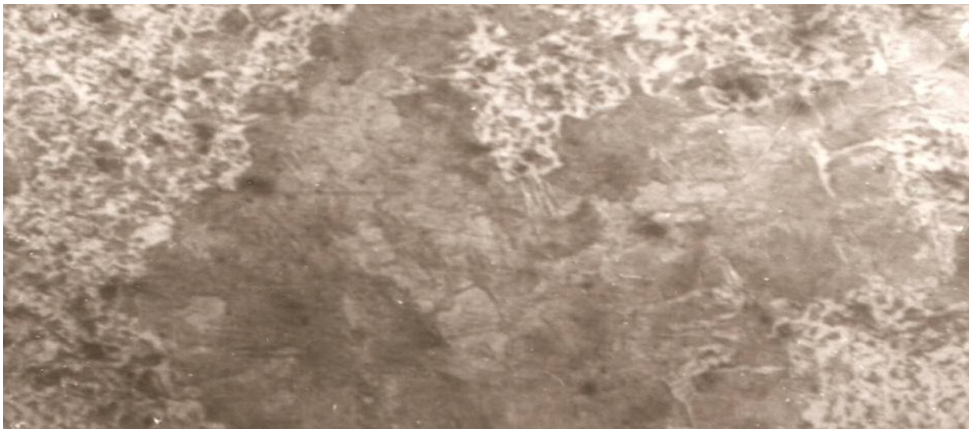
Micrograph A11: Austempered medium carbon steel in hot groundnut oil at 250°C for 3hrs. The structure reveals bainite (white). (x200)



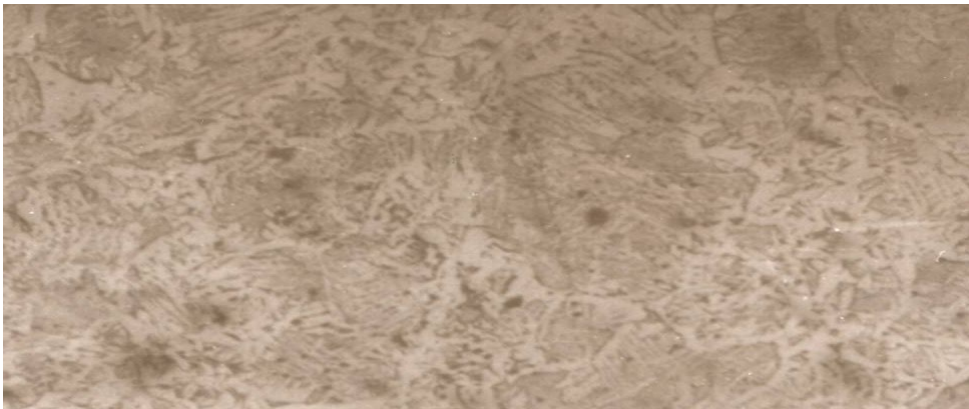
Micrograph A12: Austempered medium carbon steel in hot groundnut oil at 250°C for 4hrs. The structure shows a more bainite (white) structure. (x200)



Micrograph A13: Austempered medium carbon steel in hot groundnut oil at 250⁰C for 5hrs. The structure consists of bainite (white) and martensite (dark).(x200).



Micrograph A14: Austempered medium carbon steel in hot cotton seed oil at 250⁰C for 1hr. The structure shows abnormal growth of retained austenite (dark) and bainite (white). (x200).



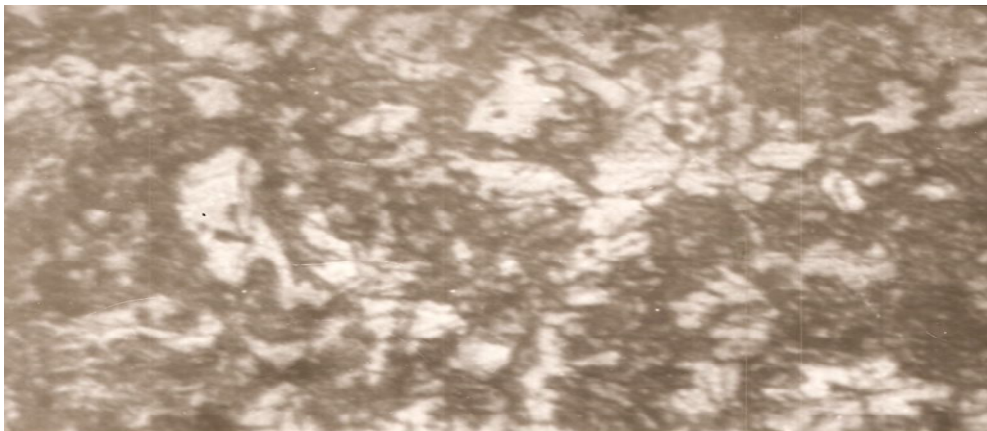
Micrograph A15: Austempered medium carbon steel in hot cotton seed oil at 250⁰C for 2hrs. The structure shows on set of bainite (white) and retained austenite (dark) . (x200).



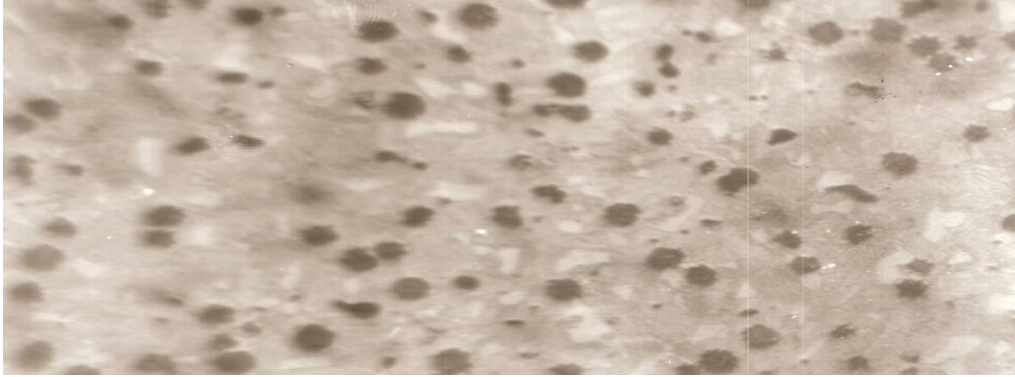
Micrograph A16: Austempered medium carbon steel in hot cotton seed oil at 250⁰C for 3hrs. The structure shows bainite plates (white) and martensite (dark). (x200)



Micrograph A17: Austempered medium carbon steel in hot cotton seed oil at 250⁰C for 4hrs. The structure indicates Widemanstatten plates (white). (x200)



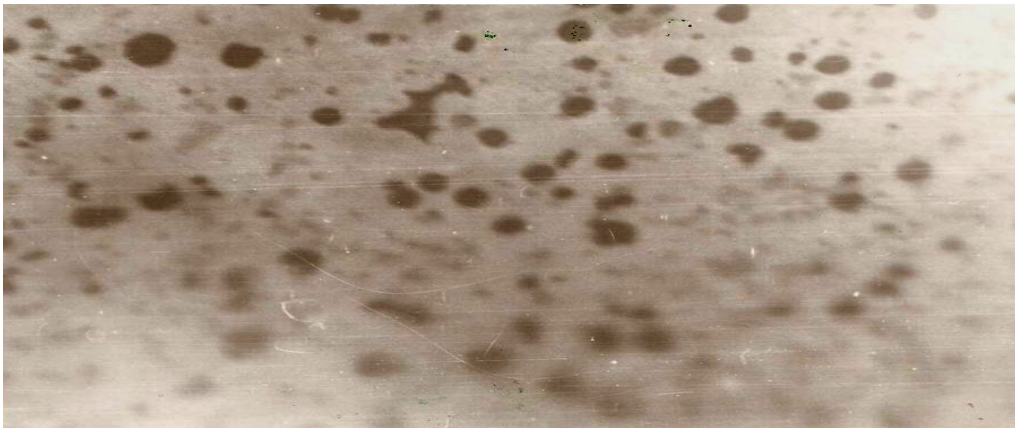
Micrograph A18: Austempered medium carbon steel in hot cotton seed oil at 250⁰C for 5hrs. The structure shows bainite (white) and stable austenite (dark). (x200)



Micrograph A19: As-received structure of ordinary nodular cast iron. The structure consists of graphite nodules [black] in pearlite matrix (white). (x250)



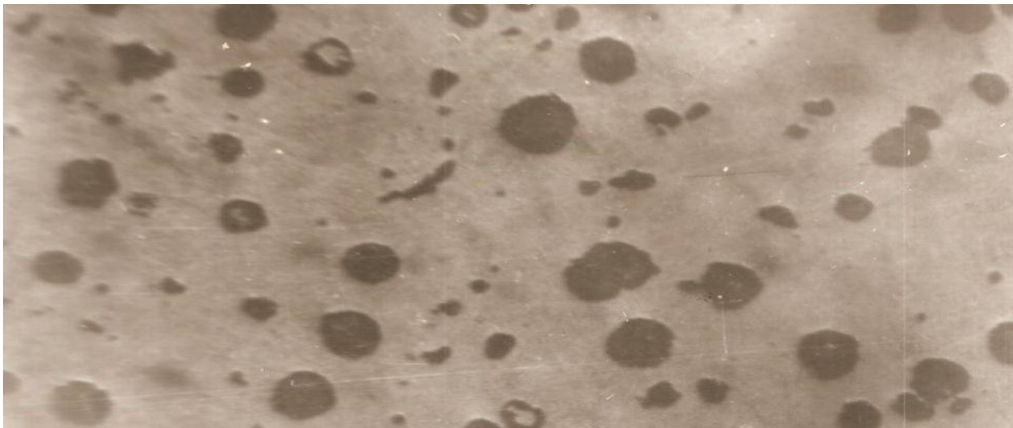
Micrograph A20: Annealed structure of ordinary nodular cast iron. The structure shows graphite nodules [black] in ferrite matrix (white areas). (x250)



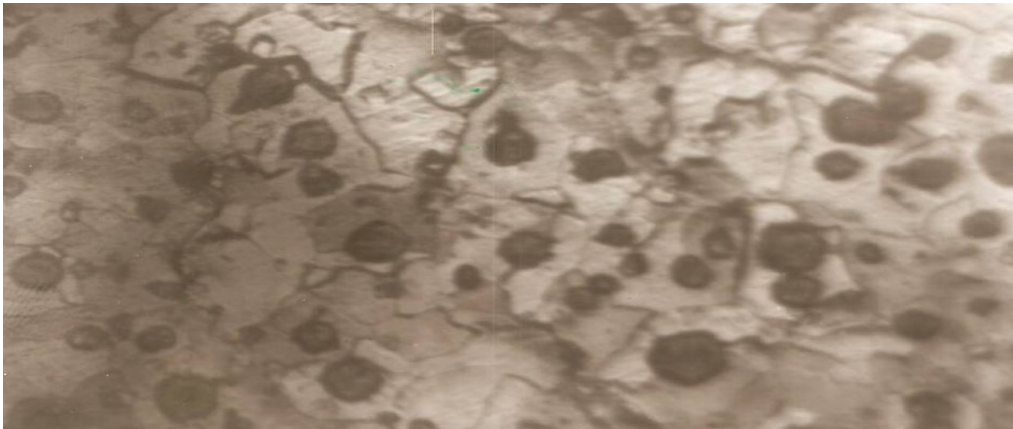
Micrograph A22: Austempered structure of ordinary nodular cast iron in hot shea butter oil at 250°C for 1hr. This structure reveals graphite nodules (black) in pearlite matrix (x200)



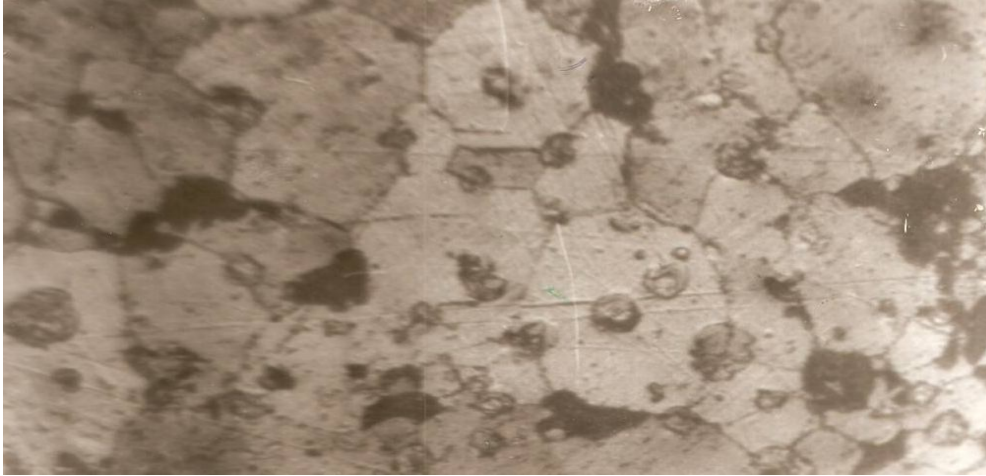
Micrograph A23: Austempered structure of ordinary nodular cast iron in hot shea butter oil at 250°C for 2hrs. The structure shows graphite nodules (black and grey balls) in ausferrite .(x200)



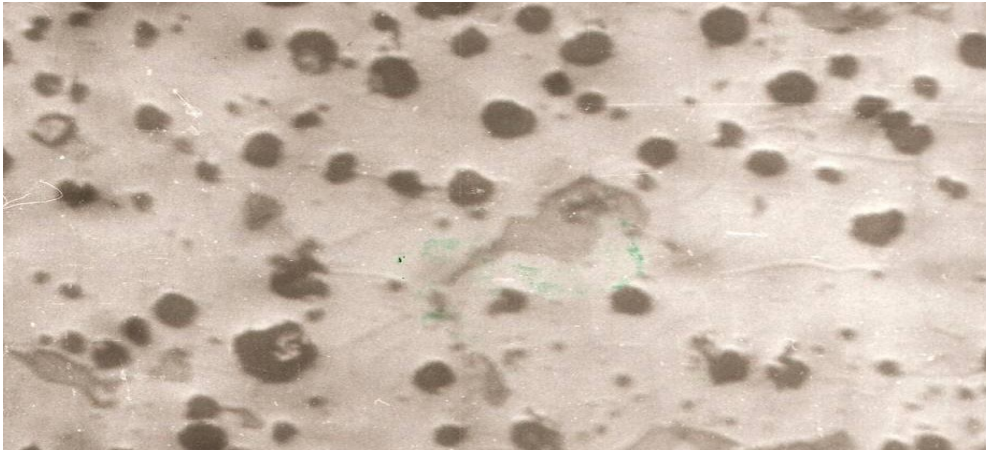
Micrograph A24: Austempered ordinary nodular cast iron in hot shea butter oil at 250°C for 3hrs. The structure consists of graphite nodules (black balls) in ausferrite matrix. (x200)



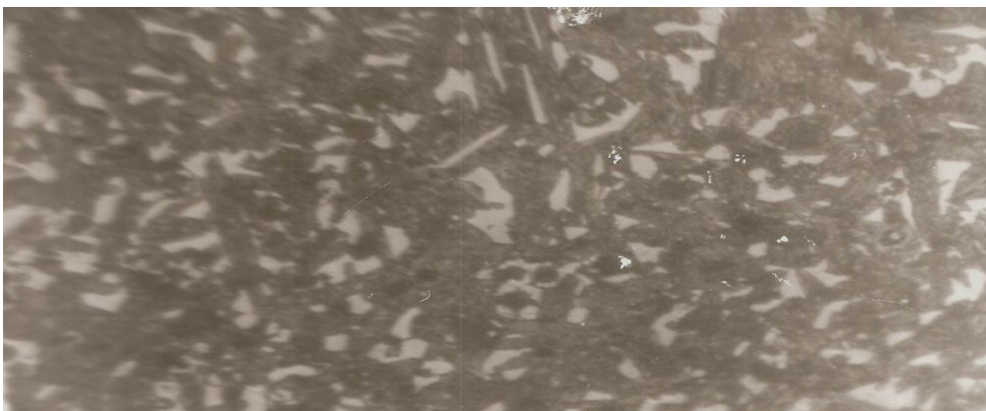
Micrograph A26. Austempered ordinary nodular cast iron in hot groundnut oil at 250°C for 1hr. The structure reveals Micrograph A26:graphite nodules (black and grey balls) in ausferrite matrix. (x200).



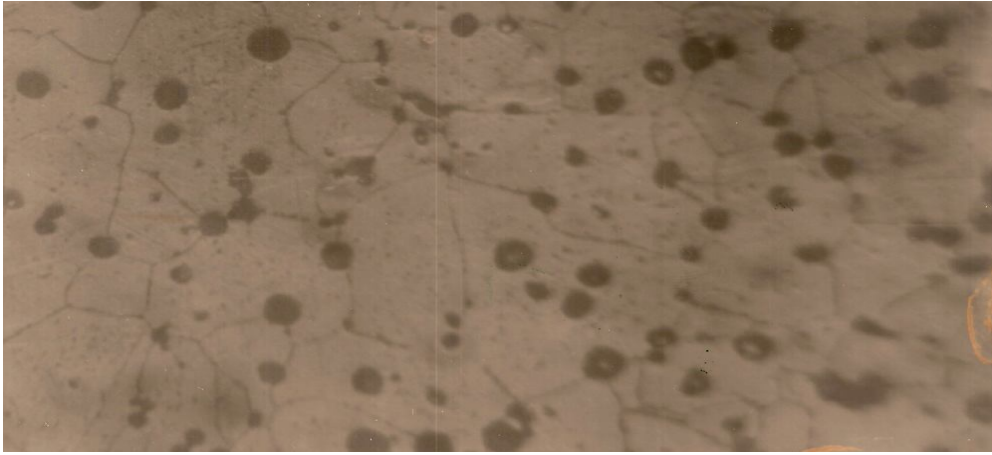
Micrograph A27: Austempered structure of ordinary nodular cast iron in hot groundnut oil at 250°C for 2hrs. The structure shows graphite nodules (black and grey balls) in a matrix of ausferrite. (x200).



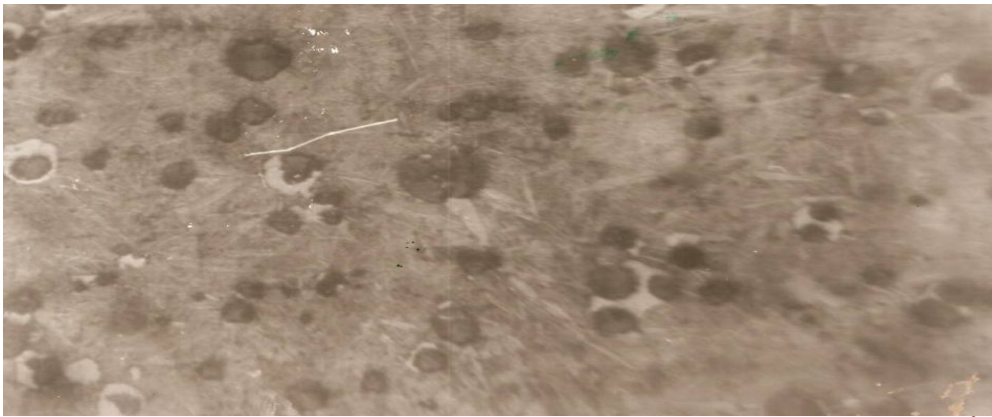
Micrograph A28: Austempered structure of ordinary nodular cast iron in hot groundnut oil at 250°C for 3hr. The structure reveals graphite nodules (black balls) in ausferrite. (x200).



Micrograph A29: Austempered nodular cast iron in hot groundnut oil at 250°C for 4hrs. The structure shows graphite nodules (black balls) in ausferrite and martensite (dark) matrix. (x200).



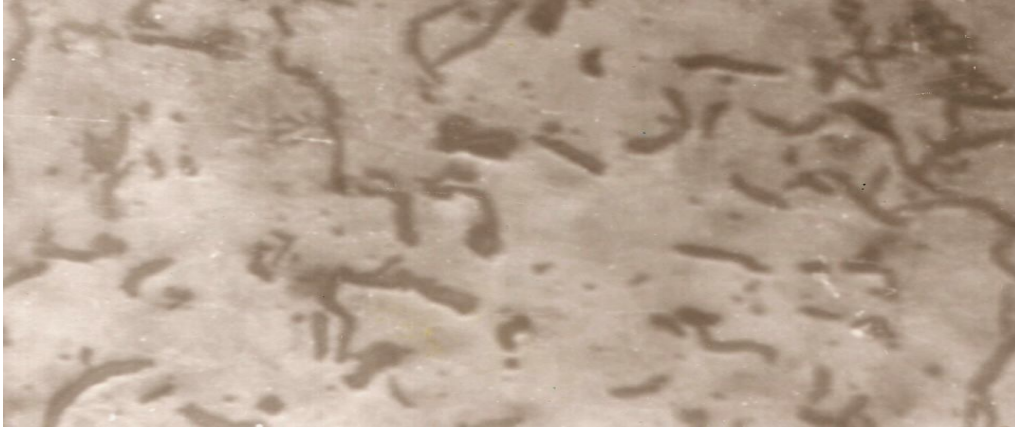
Micrograph A31: Austempered nodular cast iron in hot cotton seed oil at 250⁰C for 1hr. The structure shows graphite nodules (black balls) in retained austenite. (x200).



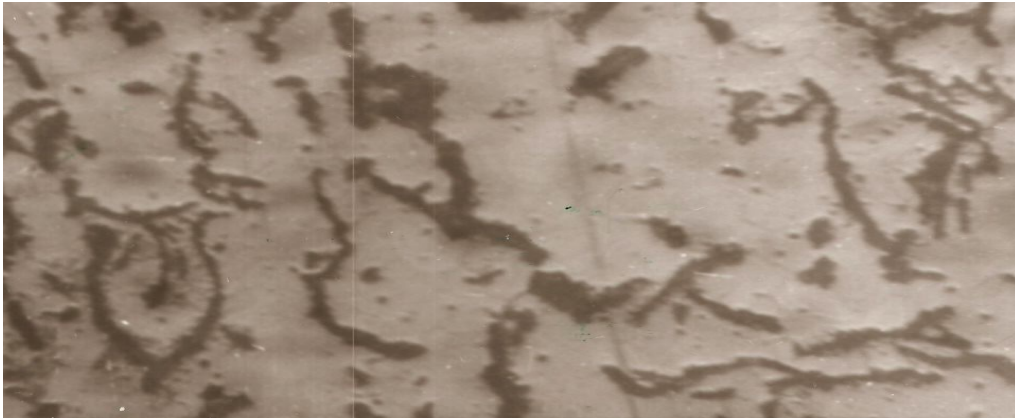
Micrograph A32: Austempered nodular cast iron in hot cotton oil at 250⁰C for 3hrs. The structure shows graphite nodules (back balls) with carbide particles and few Bull-eyes. (x200).



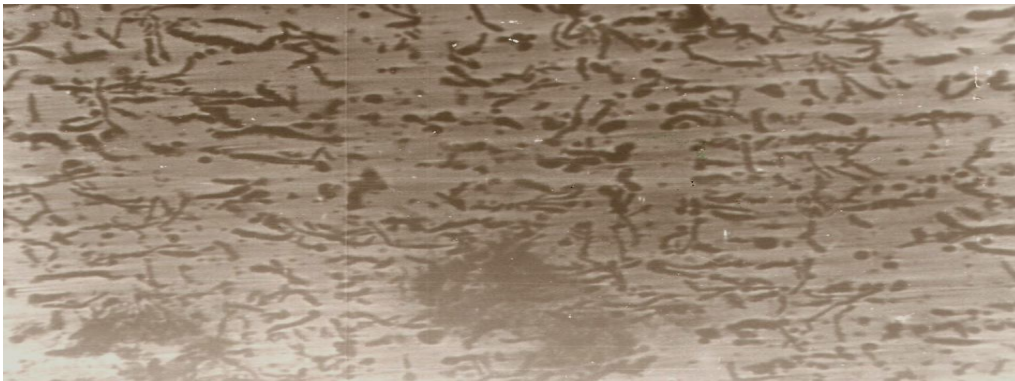
Micrograph A33: Austempered nodular cast iron in hot cotton seed oil at 250⁰C for 5hrs. The structure reveals graphite nodules (back balls) and some particles of secondary graphite (black plate) in ausferrite matrix. (x250).



Micrograph A34: As-received structure of grey cast iron. The structure consists of graphite (dark flakes) and ferrite (light-colored grains). (x200).



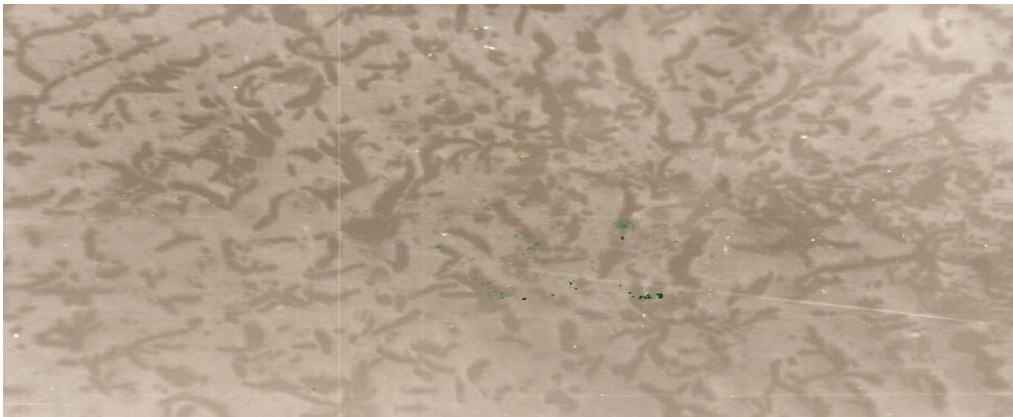
Micrograph A35: Annealed structure of grey cast iron. The structure consists of graphite (dark flakes) and ferrites (light-colored grains). (x200)



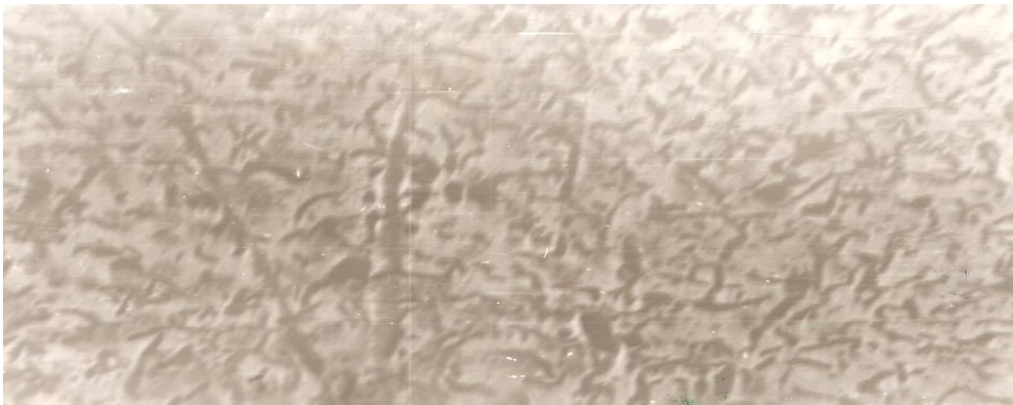
Micrograph A36: Austempered grey cast iron in hot groundnut oil at 250°C for 1hr. The structure shows graphite (dark flakes) and retained austenite. (x200)



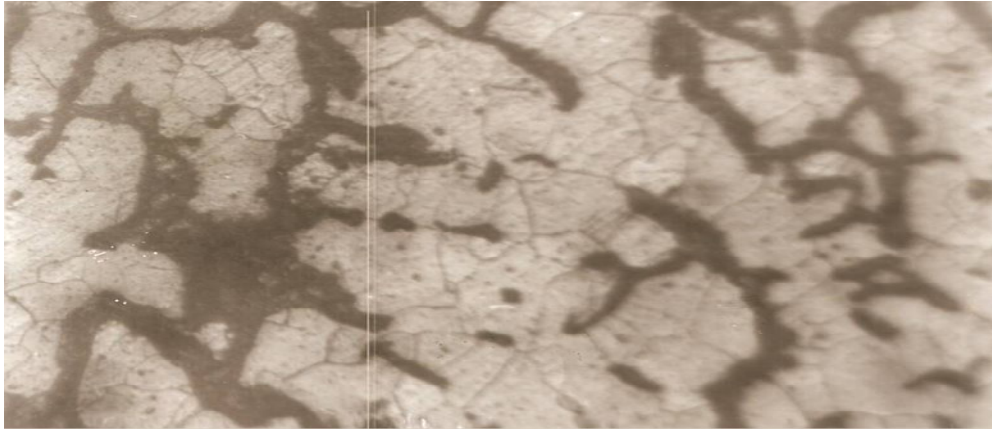
Micrograph A37: Austempered grey cast iron in hot groundnut oil at 250°C for 2hrs. The structure reveals graphite (dark-flakes) and martensite. (x200)



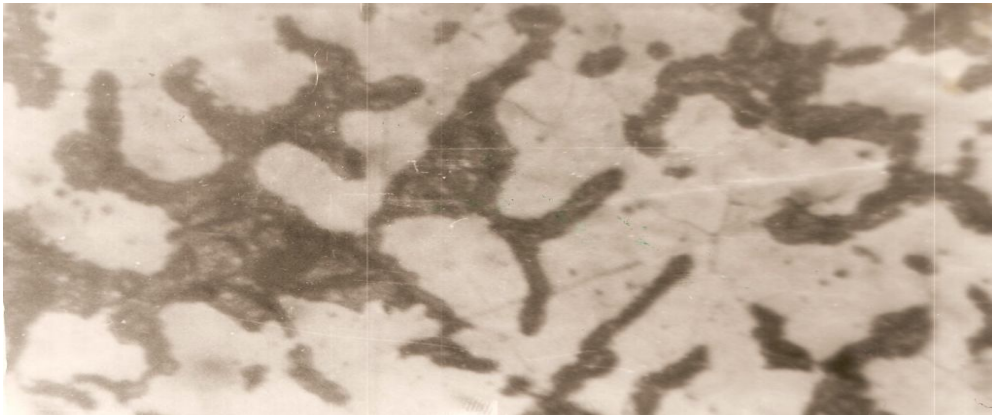
Micrograph A38: Austempered grey cast iron in hot groundnut oil at 250°C for 3hrs. The structure shows graphite (dark flakes) in martensite. (x200).



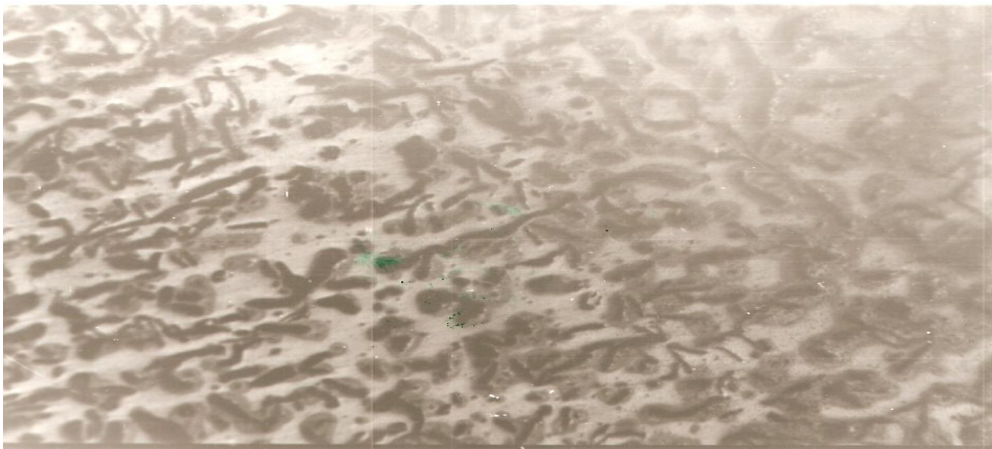
Micrograph A39: Austempered grey cast iron in shear butter oil at 250°C for 4hrs. The structure consists graphite (dark flakes) in network of ausferrite and martensite. (x200).



Micrograph A40: Austempered grey cast iron in hot shear butter at 250°C for 1hr. The structure consists of graphite (dark flakes) and retained austenite. (x200).



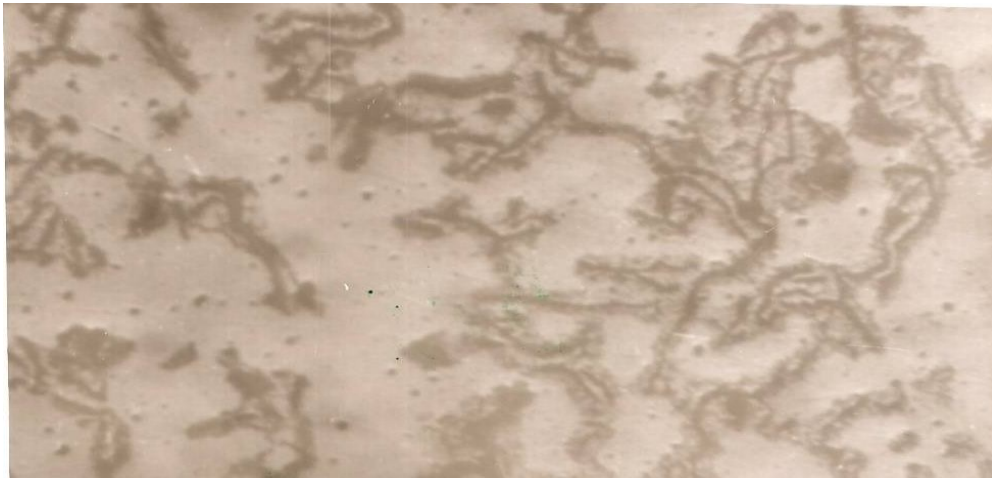
Micrograph A41: Austempered grey cast iron in hot shear butter oil at 250°C for 2hrs. The structure shows graphite (dark flakes) in martensite matrix. (x200).



Micrograph A42: Austempered grey cast iron in hot shear butter oil at 250°C for 3hrs. The structure reveals graphite dark flakes (dark) transformed to ausferrite and martensite (dark). (x200)



Micrograph A43: Austempered grey cast iron in hot groundnut oil at 250°C for 4hrs. The structure shows graphite flakes (dark) transformed to ausferrite and martensite (dark). (x200)



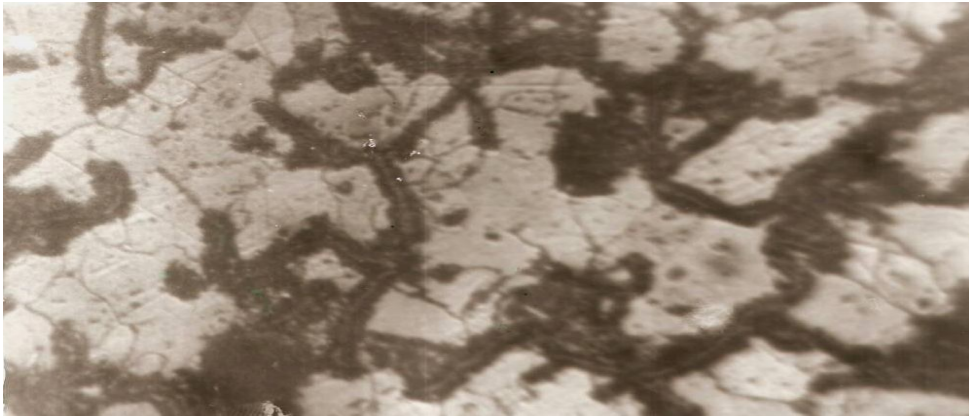
Micrograph A44: Austempered grey cast iron in hot cotton seed oil at 250°C for 1hr. The structure consists of graphite flakes (dark) in and martensite. Particles of secondary graphite also present (black dots). (x200)



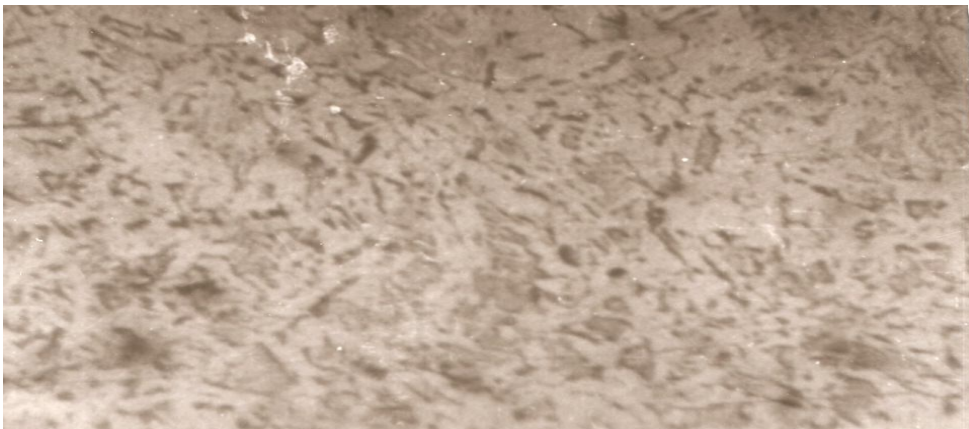
Micrograph A45: Austempered grey cast iron in hot cotton seed oil at 250°C for 2hrs. The structure reveals graphite flakes (black) in martensite. More particles of secondary graphite (black dots) were formed. (x200)



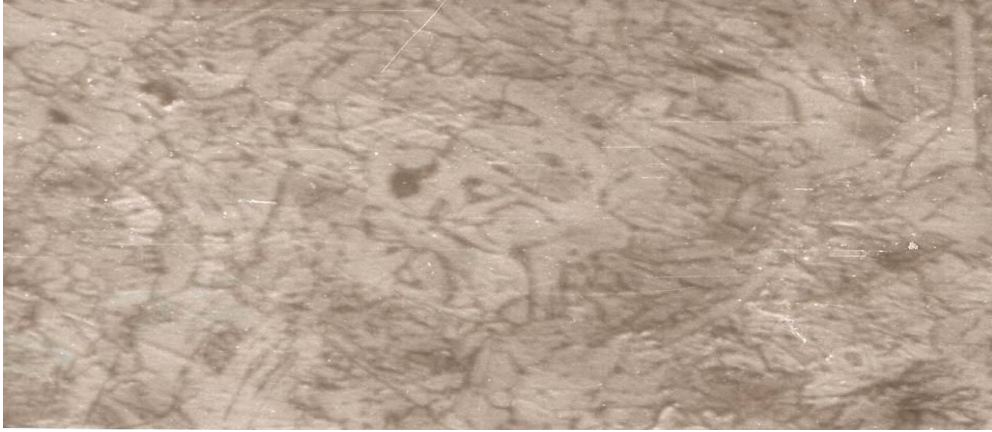
MicrographA46. Austempered grey cast iron in hot cotton seed oil at 250⁰ C for 3hrs. The structure consists of graphite flakes (black) in martensite matrix. Particles of secondary graphite also present (black dots). (x200)



Micrograph A47: Austempered grey cast iron in hot cotton seed oil at 250⁰C for 5hrs. The structure shows graphite flakes (black) in ausferrite matrix. (x200).



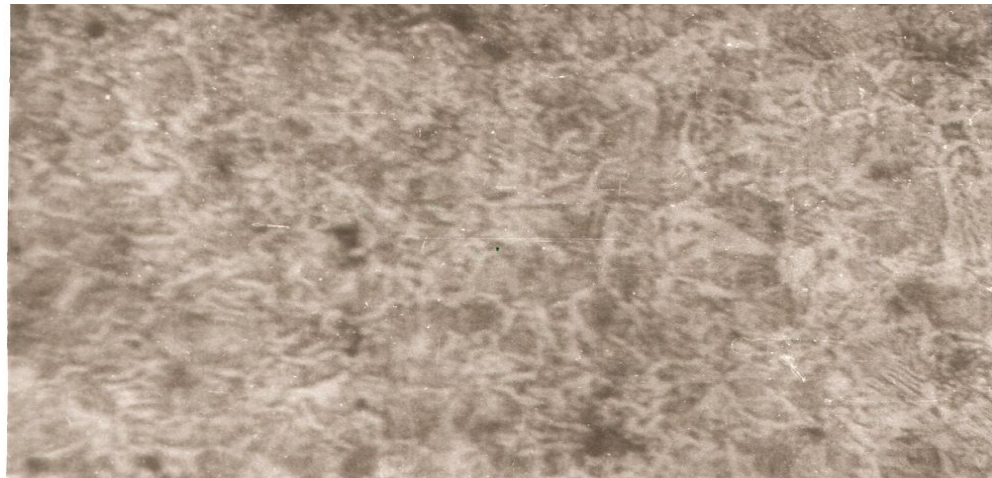
Micrograph A48: As- cast structure of low alloyed carbon steel. The structure consists of ferrite (white) and pearlite (dark). (x200)



Micrograph A49: Annealed structure of low alloyed carbon steel. The structure consists of ferrite (white) and pearlite (dark). (x200)



Micrograph A50: Normalized structure of low alloyed carbon steel. The structure indicates fine ferrite (white) and pearlite (dark). (x200)



Micrograph A51: Austempered low alloyed carbon steel in hot cotton seed oil at 250°C for 1hr. The structure shows on set of bainite (white) and martensite (dark). (x200)



Micrograph A52: Austempered low alloyed carbon steel in hot cotton seed oil at 250°C for 3hrs. The structure consists of bainite structure. (x200)



Micrograph A53: Austempered low alloyed carbon steel in hot cotton seed oil at 250°C for 4hrs. The structure shows more bainite . (x200)



Micrograph A54: Austempered low alloyed carbon steel in hot cotton seed oil at 250°C for 5hrs. The structure consists of massive bainite structure. (x200)



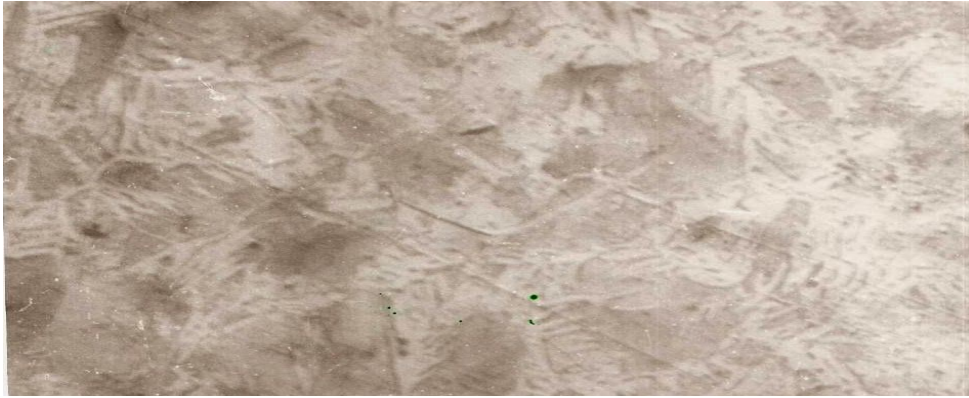
Micrograph A55: Austempered low alloyed carbon steel in hot groundnut oil at 250°C for 1hr. The structure shows Widmanstätten plates (white). (x200)



Micrograph A56: Austempered low alloyed carbon steel in hot groundnut oil at 250°C for 2hrs. The structure consists of bainite and martensite. (x200)



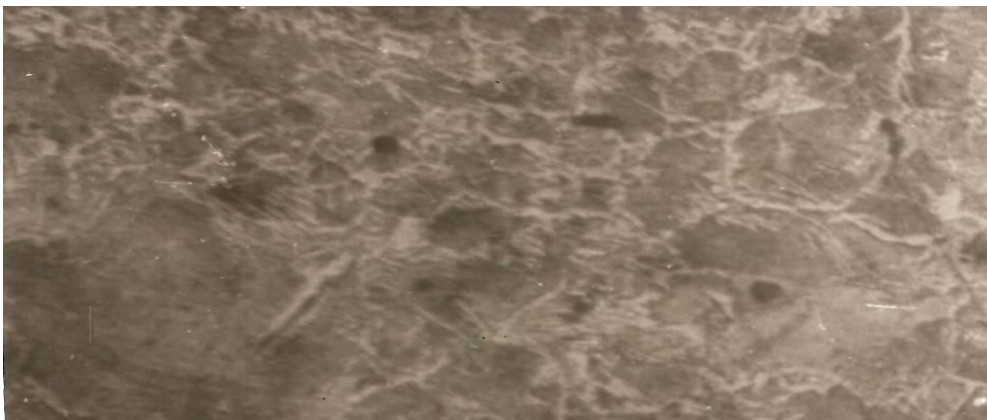
Micrograph A57: Austempered low alloyed carbon steel in hot groundnut oil at 250°C for 3hrs. The structure consists of more bainite structure. (x200)



Micrograph A58: Austempered low alloyed carbon steel in hot groundnut oil at 250°C for 4hrs. The structure shows massive network of bainite (white). (x200)



Micrograph A59: Austempered low alloyed carbon steel in hot shear butter oil at 250°C for 2hrs. The structure consists of bainite (white) and martensite (dark). (x200)



Micrograph A60: Austempered low alloyed carbon steel in hot shear butter oil at 250°C for 3hrs. The structure shows network of bainite and martensite (dark). (x200)



Micrograph A61: Austempered low alloyed carbon steel in hot shear butter oil at 250°C for 4hrs. The structure shows a network of bainite (white) and martensite (dark). (x200)

APPENDIX B

Table B1: Results of Hardness, Tensile and Impact Tests for As-Received, Annealed and Normalized samples.

| Grade of Alloy | Hardness (HV) | | | Tensile (N/mm ²) | | | Impact (J) | | |
|-----------------------------------|---------------|----------|------------|------------------------------|----------|------------|-------------|----------|------------|
| | As-received | Annealed | Normalized | As-received | Annealed | Normalized | As-received | Annealed | Normalized |
| Medium carbon steel 0.366%C | 200 | 154 | 264 | 610 | 620 | 600 | 49 | 51 | 44 |
| Low alloyed cast steel 0.492%C | 236 | 228 | 280 | 520 | 607 | 594 | 11 | 23 | 17 |
| Nodular cast iron | 196 | 169 | 251 | 570 | 761 | 632 | 31 | 46 | 41 |
| Grey cast iron | 235 | 220 | 240 | 375 | 227 | 427 | 25 | 33 | 24 |

Table B2: Mechanical properties of medium carbon steel samples, austenitized at 950°C and austempered at 250°C.

| Medium | Austempering time (Hrs) | Hardness (HV) | Tensile Strength (N/mm ²) | 0.2% Proof Stress (N/mm ²) | % Elongation (%) | % reduction in area (%) | Impact (charpy) strength (J) |
|------------------|-------------------------|---------------|---------------------------------------|--|------------------|-------------------------|------------------------------|
| Ground-nut oil | 1 | 269 | 606 | 386 | 65 | 36 | 35 |
| | 2 | 261 | 648 | 418 | 70 | 42 | 39 |
| | 3 | 205 | 684 | 450 | 82 | 41 | 43 |
| | 4 | 200 | 787 | 529 | 82 | 78 | 45 |
| | 5 | 196 | 898 | 536 | 86 | 80 | 58 |
| Cotton seed oil | 1 | 294 | 725 | 420 | 64 | 70 | 42 |
| | 2 | 281 | 729 | 424 | 66 | 70 | 54 |
| | 3 | 275 | 828 | 524 | 69 | 74 | 56 |
| | 4 | 265 | 833 | 550 | 70 | 75 | 59 |
| | 5 | 261 | 866 | 624 | 78 | 85 | 67 |
| Shear Butter Oil | 1 | 353 | 725 | 456 | 72 | 30 | 52 |
| | 2 | 269 | 730 | 475 | 78 | 31 | 56 |
| | 3 | 251 | 760 | 502 | 80 | 34 | 59 |
| | 4 | 250 | 870 | 663 | 81 | 35 | 61 |
| | 5 | 239 | 1020 | 859 | 88 | 42 | 101 |

Table B3: Mechanical properties of nodular cast iron samples austenitized at 950°C and austempered at 250°C.

| Medium | Austempering time (Hrs) | Hardness (HV) | Tensile Strength (N/mm ²) | Proof Stress (N/mm ²) | % Elongation (%) | % reduction in area (%) | Impact Energy (J) |
|------------------|-------------------------|---------------|---------------------------------------|-----------------------------------|------------------|-------------------------|-------------------|
| Ground-nut oil | 1 | 398 | 566 | 306 | 1.5 | 1.0 | 20 |
| | 2 | 385 | 699 | 456 | 3.0 | 2.0 | 25 |
| | 3 | 339 | 813 | 459 | 4.0 | 3.0 | 46 |
| | 4 | 305 | 918 | 752 | 6.0 | 2.0 | 55 |
| | 5 | 256 | 1041 | 776 | 9.0 | 6.0 | 64 |
| Cotton seed oil | 1 | 416 | 480 | 232 | 1.0 | 0.5 | 19 |
| | 2 | 411 | 501 | 259 | 1.0 | 0.5 | 19 |
| | 3 | 388 | 531 | 269 | 1.5 | 1.5 | 22 |
| | 4 | 276 | 650 | 386 | 2.0 | 1.0 | 34 |
| | 5 | 269 | 733 | 481 | 2.0 | 1.5 | 37 |
| Shear Butter Oil | 1 | 412 | 690 | 430 | 3.0 | 2.0 | 20 |
| | 2 | 404 | 660 | 403 | 4.5 | 2.5 | 35 |
| | 3 | 347 | 820 | 625 | 7.0 | 3.0 | 46 |
| | 4 | 286 | 1030 | 768 | 9.5 | 14.0 | 68 |
| | 5 | 349 | 860 | 684 | 8.5 | 3.5 | 47 |

Table B4: Mechanical properties of grey cast iron samples austenitized at 950°C austempered at 250°C

| Medium | Austempering time (Hrs) | Hardness, (HV) | Tensile strength, (N/mm ²) | 0.2% proof stress, (N/mm ²) | % Elongation, (%) | % reduction in area, (%) | Impact (Charpy) strength,(J) |
|-----------------|-------------------------|----------------|--|---|-------------------|--------------------------|------------------------------|
| G/nut oil | 1 | 296 | 543 | 256 | 0.1 | 0.1 | 28 |
| | 2 | 261 | 567 | 278 | 0.1 | 0.1 | 31 |
| | 3 | 269 | 579 | 316 | 0.2 | 0.1 | 35 |
| | 4 | 230 | 613 | 360 | 0.2 | 0.15 | 46 |
| | 5 | 209 | 631 | 371 | 0.2 | 0.2 | 48 |
| Cottonseed oil | 1 | 270 | 342 | 160 | 0.1 | 0.1 | 22 |
| | 2 | 258 | 353 | 172 | 0.1 | 0.1 | 24 |
| | 3 | 244 | 371 | 185 | 0.1 | 0.1 | 25 |
| | 4 | 238 | 395 | 212 | 0.2 | 0.1 | 30 |
| | 5 | 235 | 416 | 219 | 0.2 | 0.15 | 32 |
| Shea butter oil | 1 | 289 | 449 | 221 | 0.1 | 0.1 | 32 |
| | 2 | 266 | 457 | 242 | 0.1 | 0.1 | 32 |
| | 3 | 236 | 550 | 268 | 0.2 | 0.1 | 45 |
| | 4 | 201 | 676 | 393 | 0.35 | 0.2 | 52 |
| | 5 | 191 | 684 | 413 | 0.55 | 0.2 | 56 |

Table B5: Mechanical properties of low alloyed carbon steel samples austenitized at 950°C and austempered at 250°C.

| Medium | Austempering time (Hrs) | Hardness, (HV) | Tensile strength N/mm ² | Proof stress, N/mm ² | % Elongation, % | % Reduction in area % | Impact energy (J) |
|-----------------|-------------------------|----------------|------------------------------------|---------------------------------|-----------------|-----------------------|-------------------|
| G/nut oil | 1 | 298 | 444 | 260 | 0.1 | 0.1 | 16 |
| | 21 | 264 | 556 | 354 | 0.1 | 0.1 | 21 |
| | 3 | 242 | 648 | 436 | 0.15 | 0.1 | 22 |
| | 4 | 167 | 688 | 495 | 0.2 | 0.1 | 23 |
| | 5 | 161 | 893 | 653 | 0.2 | 0.15 | 25 |
| Cottonseed oil | 1 | 279 | 597 | 362 | 0.1 | 0.1 | 24 |
| | 2 | 271 | 735 | 472 | 0.1 | 0.2 | 24 |
| | 3 | 265 | 791 | 525 | 0.2 | 0.2 | 26 |
| | 4 | 260 | 821 | 547 | 0.2 | 0.2 | 26 |
| | 5 | 241 | 852 | 568 | 0.3 | 0.2 | 27 |
| Shea butter oil | 1 | 287 | 516 | 312 | 0.1 | 0.1 | 22 |
| | 2 | 282 | 643 | 436 | 0.2 | 0.1 | 22 |
| | 3 | 246 | 658 | 448 | 0.2 | 0.2 | 25 |
| | 4 | 221 | 699 | 489 | 0.2 | 0.2 | 25 |
| | 5 | 213 | 954 | 696 | 0.4 | 0.2 | 36 |