POTENTIAL OF SESAME OIL *(Sesanum indicum)* BATH AS AN AUSTEMPERING MEDIUM FOR DUCTILE CAST IRON

BY

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AHMADU BELLO UNIVERSITY, ZARIA

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Declaration

I declare that this dissertation titled "Potential of sesame oil bath as an austempering medium for ductile cast iron" was carried by me at the Department of Metallurgical and Materials Engineering under the supervision of Prof. T. Ause and Prof. O. Aponbiede. The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this dissertation was previously presented for another degree or diploma at any university.

Abdulhamid, Shehu _____ _____

(Signature) **Date**

Certification

This dissertation entitled "POTENTIAL OF SESAME OIL BATH AS AN AUSTEMPERING MEDIUM FOR DUCTILE CAST IRON" by Shehu Abdulhamid meets the regulations governing the award of Master of Science Degree of the Ahmadu Bello University, Zaria and is approved for its contribution to knowledge and literary presentation.

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Dedication

This work is dedicated to my father, Alhaji Abdulhamid Waziri, for cherishing me and giving me the right upbringing.

Acknowledgement

I am ultimately grateful to Allah for making it possible for me to successfully complete this work. My profound and deepest gratitude goes to my supervisors Prof. T. Ause and Prof. O. Aponbiede who inspite of their tight schedule of work were able to spare time to go through my Dissertation in details and proffer relevant corrections and suggestions. Their persistent encouragement has been immensely useful.

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Lastly, I am indeed grateful to my mother, family and my caring friends for their supports and encouragements.

Abstract

The effect of hot-sesame oil bath as an austempering medium for ductile cast iron was investigated. Samples of ductile cast iron were machined to standard dimensions for tensile, impact and hardness/microstructural analysis test specimens prior to the austempering process. Sesame oil was used as hot quenching bath for the austempering process. The samples were normalized and thereafter, austempered in hot Sesame oil bath at 250^{o}C and 300^{o}C for 1-5hrs from austenitizing temperatures of 850^{o}C and 900^{o}C. Mechanical properties of the test samples were evaluated. The test results obtained showed that samples austempered at 250^{o}C and 300^{o}C for 3hrs exhibited superior mechanical properties than those at 1, 2, 4 and 5hrs. The tensile strength increased from 523N/mm^2 for the as-received samples to the maximum value of 1029N/mm^2 for the austempered samples with percent elongation of 14.6. Impact strength increased from 61kJ/m^2 (as-received) to 161kJ/m^2 (austempered). It was also observed that the hardness value increased from 324 HVN (as-received) to 418 HVN after austempering. Microstructures of the samples were analyzed using optical microscopy (OM) and scanning electron microscopy (SEM). In all the test samples, ausferrite (bainite) structures which consist of acicular ferrite and carbon enriched austenite was observed. The FTIR analysis of the oil after austempering heat treatment showedthat some peaks (functional groups/radicals) were re-configured.The research showed that, sesame oil produced ausferrite (bainite) structures, hence could be recommended for use as an austempering quenching medium for ductile cast iron.

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Abbreviations

%	-	Percentage
Mn	-	Manganese
Fe	-	Iron
P	-	Phosphorous
C	-	Carbon
Mg	-	Magnesium
Mo	-	Molybdenum
Ni	-	Nickel
Cu	-	Copper
S	-	Sulphur
SEM	-	Scanning Electron Microscopy
OM	-	Optical microscopy
Cp	-	Centipoises
X100	-	Magnification at 100 times
Si	-	Silicon
HT	-	Heat treatment
Aust.	-	Austempering
FTIR	-	Fourier transform infrared spectroscopy

CHAPTER ONE

INTRODUCTION

1.1Background of the Study

Heat treatment originated as an ancient art in man's attempt to improve the performance of materials in practical applications (Rajan*et al.*, 1988).Khanna (2002) pointed out that, in the middle of the 19th century, more attention was directed to develop the art and science of producing, working and treating of metals to suit various areas of applications to improve man's comfort. The final properties of an alloy are not only due to its chemical composition, but also due to its metallurgical history. It's possible to use different treatments so as to change the properties of an alloy; thus, finding the easiest way of heat treatment and optimum treatment conditions is of fundamental importance in achieving the desired properties (Fracasso, 2010).

Metals and alloys develop requisite properties by heat treatment which plays a critical role in achieving appropriate microstructure that imparts the desired characteristics in a given material. Hence, the study of heat treatment is of great significance.

Rajan *et al.* (1988) defined heat treatment as heating and cooling operation(s) applied to metals and alloys in solid state so as to obtain the desired properties. Formation of austenite is a preliminary step for any heat treatment process for ferrous alloys. Depending on various parameters (composition of the alloy, cooling rate, section size, etc), austenite may transform on cooling to lower temperature phases such as: ferrite, pearlite, bainite or martensite. The presence of these transformed phases and their structures is important in deciding the resultant properties in the metal/alloy (Ause, 2008). There are various types of heat treatment processes, namely; annealing, normalizing, hardening, tempering etc. In addition to these we have special heat treatments called interrupted quenching procedureswhich include;

ausforming, martempering and austempering (Khanna, 2002). Ause(2008) showed that these heat treatment processes are used to achieve the following: refine grains, soften the metal, increase hardness, remove residual stresses, improve machinability, improve toughnessand increase ductility. According to Hassan, (2009) austempering is a hardening process for metals/alloys which yields better mechanical properties including: higher ductility, resistance to shock and uniform hardness. The process consists of quenching the material from the proper austenitizing temperature directly into a hot bath at a temperature between 230°C – 450°C, and the bath temperature is maintained for a desired recommended time to transform the austenite into bainite. In the process of austempering, ductile cast iron undergoes a remarkable transformation when subjected to the heat treatment process. The resulting microstructure, known as "Ausferrite", consists of unique matrix of fine acicular ferrite with carbon enriched stabilized austenite while, the matrix of conventional ductile iron is a mixture of pearlite and ferrite (Amar *et al.,* 2013).

Generally, quenchants are grouped into two categories namely: those maintained at room temperature and those maintained at higher temperatures (250-450^{o}C). The room temperature quenchants are regarded as the most popular, common and conventional or traditional type of quenchants. Few examples include; water, brine, oil (mineral or vegetable), caustic soda solution, etc. Those quenchants maintained at higher temperaturesare used for interrupted quenching heat treatment processes such as martempering, austempering, and ausforming. Examples of these types of quenching media are molten salt bath, molten lead bath and hot oil bath (ASM, 1978).

According to Ause, (2008) for some heat treatment operations that requires hardening of components by quenching, those quenching media such as water, brine, oil, etc may sometimes be too drastic, hence resulting to inferior mechanical properties and defects such as cracking and distortion. Such defected components are eventually rejected, resulting to

economic waste. Therefore, in order to impart good mechanical properties (especially strength and toughness/ductility) and simultaneously avoid cracking and distortion, many researchers suggested that other heat treatment methods like martempering and austempering in molten salt bath or hot oil bath could be used. But unfortunately, salt bath furnace is known to be very costly.

Ductile iron is a family of cast iron with free carbon existing as spherical graphite. Compared to other kinds of cast irons it has the best mechanical properties which makes it a very attractive material. The heat treating of ductile iron by austempering produces the austempered ductile iron (ADI) with outstanding mechanical properties that can be varied over a wide range by changing the heat treatment parameters. Austenitizing and austempering temperatures and time are factors that affect the final properties of ADI (Akor and Tuleum 2014). Austempered ductile iron (ADI) offers a design engineer the best combinations of low cost, design flexibility, good machinability, high strength to weight ratio and good toughness, wear resistance and fatigue strength (Tun and Lwin 2009). The austempering process was first developed in the early 1930's as a result of work by Bainwhich was conducted on the isothermal transformation of steel. In the early1940's, Flinn applied this heat treatment to gray cast iron. In 1948 the invention of ductile iron was announced jointly by the British Cast Iron Research Association (BCIRA) and the international nickel company (INCO) (Kathy, 2002).

By the 1950's, both ductile iron and the austempering process had been developed. However, the technology to produce ADI on an industrial scale lagged behind (Kathy, 2002). By the 1990's ASTM A897-90 and ASTM A897M-90 specifications for austempered ductile iron castings were published in the US while other specifications were developed worldwide. In addition, a new term to describe the matrix microstructure of ADI as "ausferrite" was introduced (Kathy, 2002 and Tun and Lwin 2009). Replacing the conventional steel parts by

ADIs resulted in several advantages which strongly promoted the acceptance and use of these materials, for example in the automotive industry (Lawrence, 2011).

1.2 Statement of the Problem

Austempering is known to be one of the quench hardening methods which is applied to ferrous alloys in order to improve toughness and ductility. It is usually carried out in a bath. The three baths used for austempering are: molten salt bath, molten lead bath and hot oil bath. Perhaps molten salt bath is the commonest used for austempering heat treatment but it is very expensive and scarce in developing countries like Nigeria. Its acquisition requires importation and in addition has the following shortcomings: It may cause explosion as a result of instrument failure, it is very hazardous especially during cleaning and in maintaining the bath at elevated temperatures, it can be dangerous as it can produce vapours which are toxic to workers, the running costs are high and the surfaces of samples become corroded if for any reason they are left too long in the bath. Molten lead bath also is expensive and emits fumes which is a health threat to humans and general environment and even to the metal itself if lead or lead oxide adheres to the metal being quenched.

Based on the above aforementioned reasons, the research question here is; can the identified Sesame oil commonly called Beniseed oil (*sesanum indicum oil*), be suitable for use as an austempering medium for ductile cast iron? Especially that the preliminary investigation on the oil have shown that, it has high flash point, it is biodegradable and is locally available in abundance which could be cheaply obtained.

1.3 Justification

Austempering heat treatment is a high performance isothermal treatment that imparts superior properties to ferrous metals/alloys. The molten salt bath and lead bath have been the most popular quenchants used for the austempering process. However, their substantial

deficiencies justify the search for alternative replacement media. This will go a long way in assisting many local heat treatment industries in the area of austempering and conserve the limited foreign exchange due to the importation of salt bath furnace and associated consumables, since Nigeria is enriched with varieties of these oils.

1.4 Aim and Objectives

The aim of this research work is to explore the use of hot Sesame(Beniseed)oil*(Sesanum indicum oil)* bath as an austempering medium for ductile cast iron. The specific objectives targeted in this work are to:

i. carry out the physico-chemical characteristics of the sesame oil.

ii. carry out the FTIR analysis of the oil before and after austempering heat treatment

iii. determine the mechanical properties(tensile, impact and hardness)of austempered ductile cast iron and to compare with that of molten salt bath in the literature.

iv. examine and study the microstructure of the austempered ductile cast iron using OPM and SEM.

v. Correlate the microstructures obtained with themechanical properties of austempered ductile cast iron

1.5 Scope of the Work

Ductile cast iron has been used to evaluate the suitability of sesame oil bath as an Austempering medium. Mechanical properties (toughness, tensile strength, percent elongation and hardness), austempering temperatures(250^{o}C and 300^{o}C), austenitizing temperatures (850^{o}C and 900^{o}C) and austempering times (1-5hours) were the parameters used to assess the effectiveness of the Sesame oil as an austempering quenching medium.

CHAPTER TWO

LITERATURE REVIEW

2.1 Introduction

The discovery of ductile iron in 1948 gave a new lease of life to the cast iron family. The history of ductile iron has been influenced by a number of technological developments that have resulted in new business opportunities for the foundry industries, the latest of which was the introduction of Austempered ductile iron (Sandeep, 2012).

The interrupted quenching procedure employed to the ductile iron which is known as austempering heat treatment, involves heating the ductile iron to the austenitizing temperature, holding it for sometime at that temperature for complete homogenization to take place and then quenching into a molten salt bath maintained at a temperature just above 250^{o}C (i.e. M_S – martensite start temperature) (Hassan, 2009). Austempered ductile iron (ADI) has a microstructure containing spheroidal graphiteembedded in a matrix of bainite(ferrite and austenite). The resulting microstructure of ADI provides properties that are superior to conventional ductile iron, cast and forged aluminum and many cast and forged steels (Tun and Lwin 2009).

2.2Classes of cast iron

The term 'cast iron' covers a wide range of iron – carbon alloys containing 2.0 to 4.0 percent carbon (Figure 2.1) and 0.5 to 3.0% Si. Theoretically however, the carbon content of cast iron can lie between 2.11% and 6.67%, as higher carbon contents tend to make the cast iron more brittle (Smith, 1993). The alloys typically contain varying percentages of Mn, S and P

together with other alloying elements such as Cr, Mo, Cu and Ti,these elements are specifically and closely controlled (Nelson, 1985)

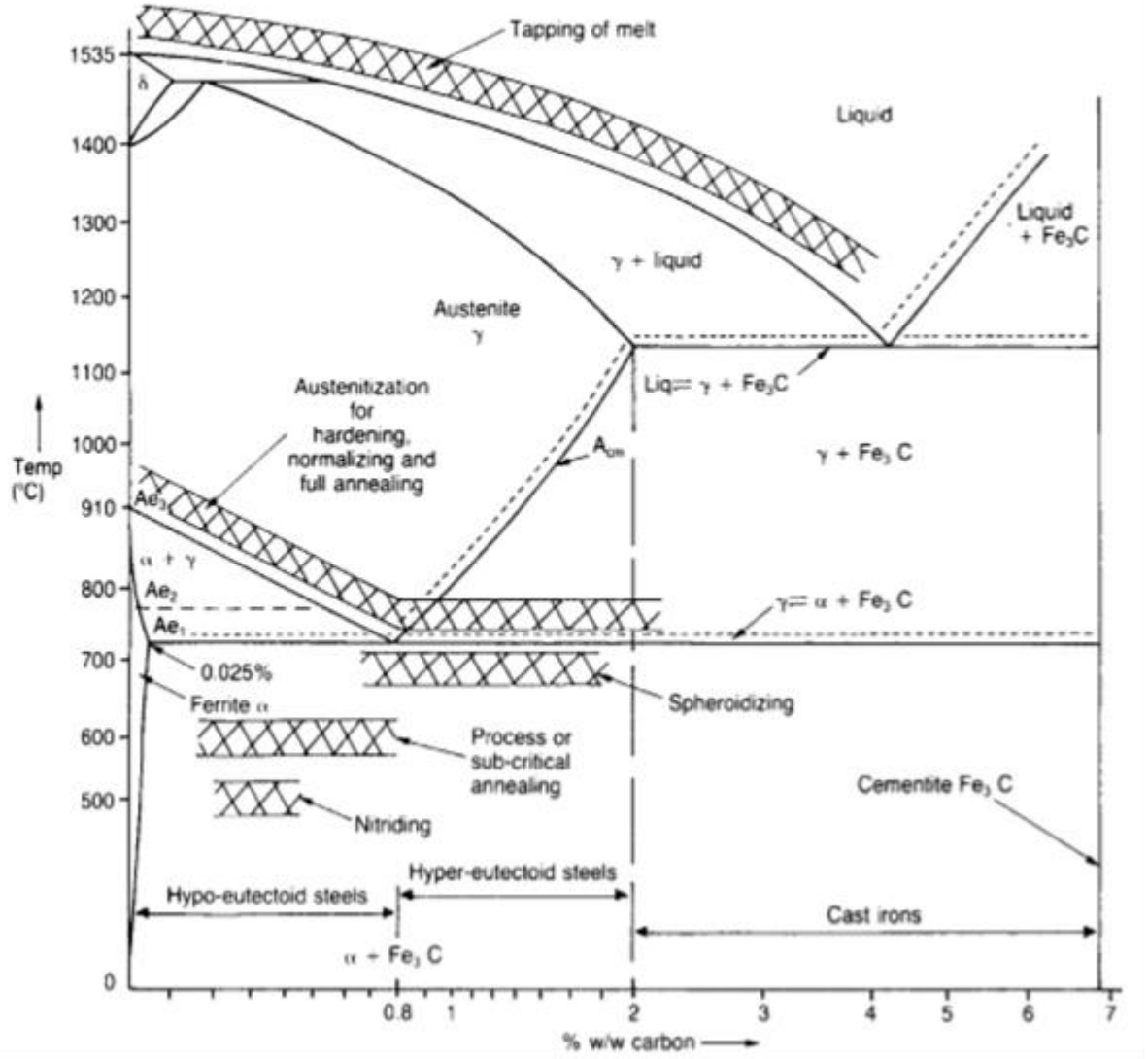


Figure 2.1: A section ofIron-Carbon Equilibrium Diagram*(Source*: Smallman and Bishop, 1999).
Note: Dotted lines represent iron-graphite equilibrium diagram

There are six basic types of cast iron which are classified depending on the shape, distribution and form of carbon phase in their microstructures which are: Grey cast iron, ductile cast iron, white cast iron, malleable cast iron, compacted graphite iron and alloy cast iron.

2.2.1 Grey cast iron

This type of cast iron is formed during solidification. When the composition and the cooling rate are appropriated, carbon will precipitate in the form of graphite flakes that are interconnected within each eutectic cell. The flakes appear black in the photography. Gray cast iron primarily fractures along the graphite and the fracture appears gray, hence the name gray iron (Figure 2.2). The graphite morphology established the mechanical properties. The applications of gray iron includes: block engines, compressor housing, transformer cases and valve bodies (Higgins, 1995).

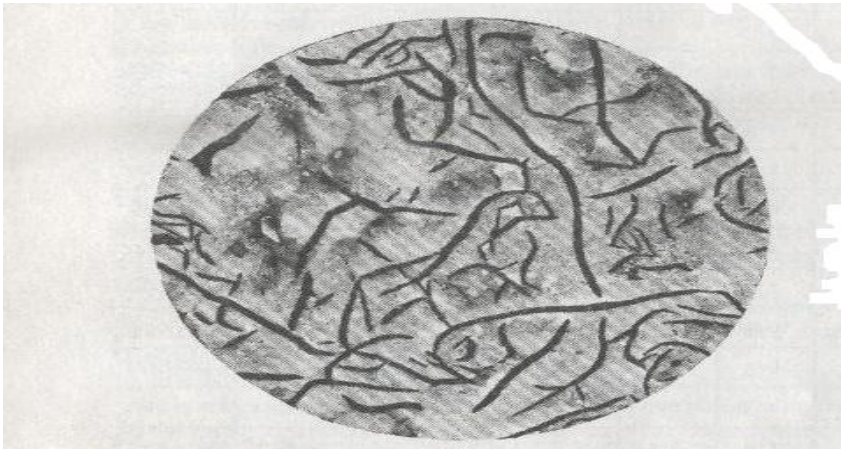


Figure 2.2: Microstructure of gray cast iron (*Source:* Alp *et al.,* 2005)

2.2.2 Ductile Cast Iron

Ductile irons have a wide range of mechanical properties which make them suitable for use in engineering components (Figure 2.3). Ductile cast iron consists of graphite spheroids dispersed in a matrix similar to that of steel (Lee *et al.,* 2003). The nodules are formed by addition of nodulizers like Magnesium, Yttrium or Cerium into the melt (Dommarco *et al.,* 2008). Ductile cast irons consisting of graphite nodules in a ferrite matrix will have high ductility and toughness whilst those consisting of graphite nodules in a pearlite matrix will be characterized by high strength (Kempster, 1979). The application of ductile iron include: Automobile construction, industrial machinery, wind turbine, electrical energy generation,

valves, air conditioning machinery, lawn and garden equipment, agricultural products (Ambooken,2006).

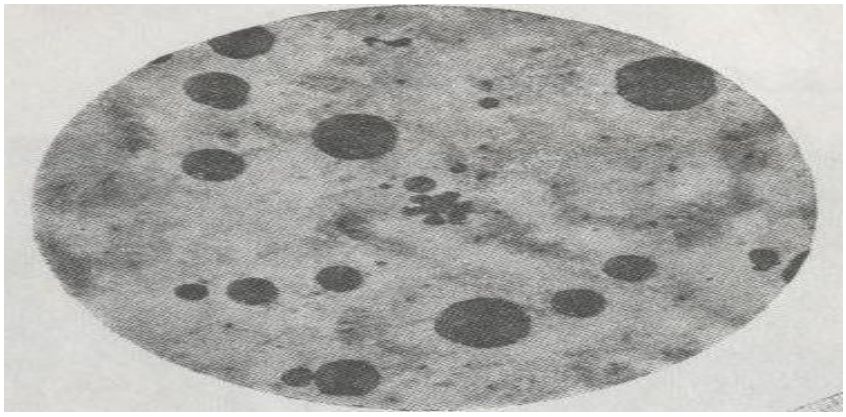


Figure 2.3: Microstructure of pearlitic ductile cast iron (*Source:* Henkel and Pense, 2002)

2.2.3White cast iron

White cast iron derives its name from the fact that its freshly broken surface shows a bright white fracture(Figure 2.4). White cast iron has almost all its carbon chemically combined in form of cementite, which is a very hard and brittle constituent (Khanna, 2002). The properties of white cast iron include: high hardness, excellent abrasive wear resistance hence not machinable, high strength in compression, very brittle. White cast iron is the first step in the production of malleable iron castings. Other uses include: crusher jaws and hammers, crusher balls, liners for ball mills, rolls for crushing ores and rolling mills (Ause, 2008).

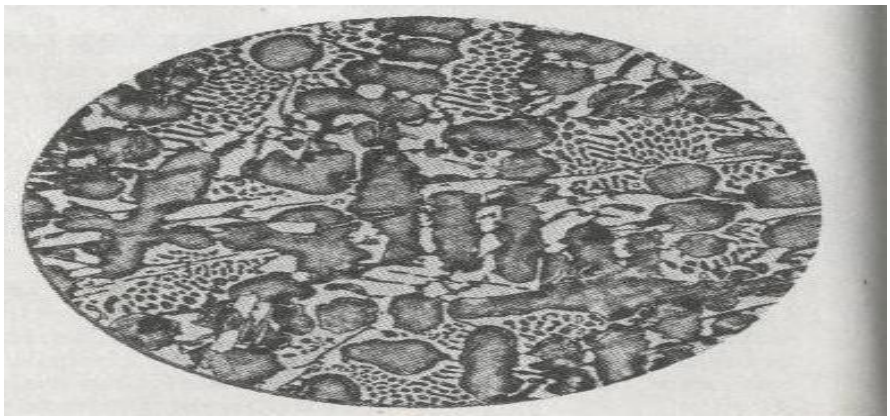


Figure 2.4: Microstructure of white cast iron(*Source:* Henkel and Pense, 2002).

2.2.4 Malleable Cast Iron

Malleable irons are produced by annealing the white iron of controlled composition. The iron is cast as white cast iron of suitable composition. The white cast iron castings are reheated to about 927°C for long periods of time in the presence of materials containing oxygen, such asiron oxide. At the elevated temperatures, cementite (Fe_3C) in the white iron decomposes into ferrite and free carbon. Upon cooling, the combined carbon further decomposes into small compact particles of graphite (instead of flake like graphite seen in grey cast iron). If the cooling rate is very slow, more free carbon is released. This free carbon is referred to as temper carbon, and the process is called malleableizing. Resulting properties are opposite to those of the white iron from which it is derived. Rather than being hard and brittle, it is malleable and easily machinable (Figure 2.5). The cost of malleable iron castings is slightly less than the ductile iron castings (Alp *et al.*, 2005).The microstructure of the malleable cast iron is shown in Figure 2.5.As their names imply, malleable cast iron have increased malleability and ductility, increased tensile strength and toughness.Malleable iron castings are often used for heavy duty bearing surfaces in automobiles, trucks, railroad rolling stock, farm and construction machinery (Alp *et al.*, 2005).

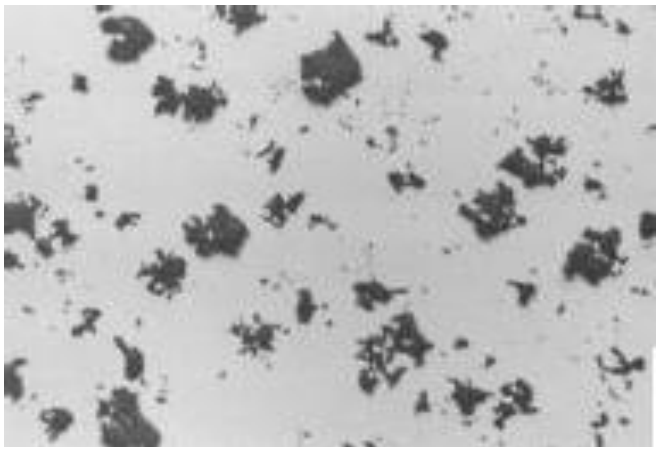


Figure 2.5: Microstructure of ferritic malleable cast iron (*Source:*Alp *et al.*, 2005).

2.2.5 Compacted Graphite Iron

Compacted graphite irons (CG) are the newest form of cast iron, recognized since 1965 having unique mechanical and physical properties determined in large part by the shape of the graphite. These irons are produced almost the same way as ductile cast iron, except the objective is to have the molten iron dissolve a lesser amount of Mg, usually 0.01-0.015Wt.%. This amount of Mg is enough to guarantee that no flakes are formed, yet not high enough to allow full nodularity (Rundman, 1990). Compacted graphite iron contains graphite in the form of thick, stubby flakes (Figure 2.6). Its mechanical properties are between those of gray and ductile iron (Ause, 2008).According to Kalpakjan,(1989) the graphite in the structure is in the form of short, thick and interconnected flakes with undulating surfaces and rounded extremities

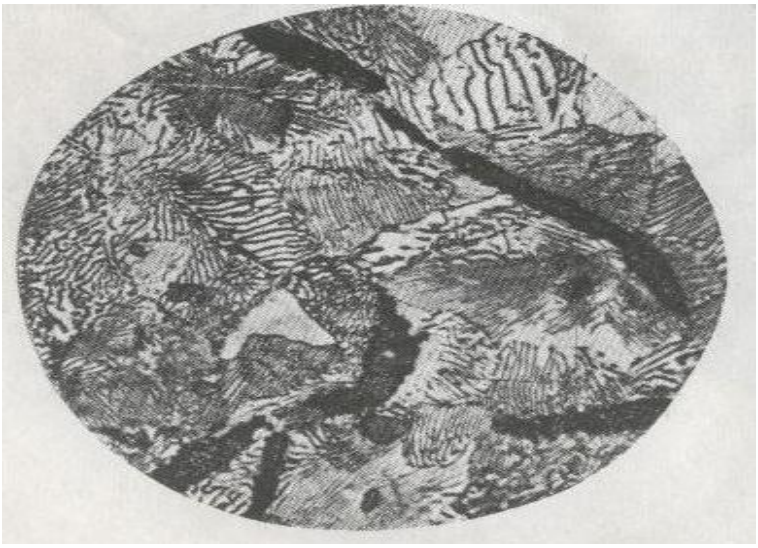


Figure 2.6:Microstructure of compacted graphite iron(*Source:* Henkel and Pense, 2002).

2.2.6 Alloy Cast Iron

Alloy cast iron is a general group where we add sufficient amount of element or elements to produce a measurable modification in the physical or mechanical properties. By doing so we

can achieve an improvement in properties like resistance to corrosion, resistance to heat or wear or to improve mechanical properties like fatigue strength and tensile strength (Ambooken, 2006).

2.3 Heat Treatment of Ferrous Alloys

Metals and alloys develop requisite properties by heat treatment which plays a critical role in achieving appropriate microstructure that impart the desired mechanical properties in a given metal. Thus, the study of heat treatment is of great significance (Rajan *et al.,* 1988).Heat treatment can be defined as heating and cooling operations applied to metals/alloys in solid state so as to obtain the desired properties. The heating and cooling sequence may involve temperatures above, below and at the ambient. Controlled heating and cooling rates and a variety of furnace atmospheres and heating media may be used. (Rajan *et al.,*1988). There are various types of heat treatment processes in practice some of which include:

2.3.1 Stress Relief Heat Treatment

As the name implies, this heat treatment process is employed to relieve internal stresses. The process of stress relieving consists of heating cast iron or steel uniformly to a temperature below the lower critical temperature, holding at this temperature for sufficient time, followed by cooling. As a general rule, stress relief treatment should not change the microstructure. These internal stresses also called residual stresses or locked in stresses that are developed during different operations like solidification of casting, welding, machining, grinding, precipitation and phase transformation during quench hardening. These stresses if not relieved, enhance the tendency of cast iron or steels toward cracking and dimensional instability (Ause, 2008).

2.3.2 Annealing Heat Treatment

AsRajan *et al.* (1988) explained, annealing involves heating cast iron or steel component to a sufficiently high temperature within the austenite region and slowly cooled in the furnace to room temperature. The temperature at which cast iron or steel is heated, and the holding time are determined by various factors such as the chemical composition, size and shape and final properties desired. Ause,(2008) showed that, annealing can be subdivided into three classes depending on heat treatment temperature as full, partial and sub-critical annealing and the purpose of annealing include:

i. To soften the component to enhance machinability

ii. To relieve internal stresses developed during solidification, machining, forging and welding

2.3.3Normalizing Heat Treatment

This is the heat treatment process whereby a steel or cast iron is heated to a temperature above the upper critical point, soaked at this temperature for the necessary homogenization to occur and then cooled in air. Normalizing treatment usually produce grain refinement as a pre-hardening condition and also removes unfavorable structures such as widmanstatten and cementite network present in the as-cast component (Khanna, 2002).

2.3.4 Hardening (quenching)

High hardness or strenght can be obtained by a process known as hardening. Hardening treatment consists of heating to a sufficiently high temperature, holding at that temperature followed by rapid cooling such as quenching in water, oil, or salt baths. The high hardness developed by this process is due to the phase transformation accompanying rapid cooling (Rajan *et al.*, 1988). The factors affecting as- quenched hardness are austenitization temperature, composition, section size, severity of the quenching medium and presence of alloying elements (Wilson, 1975).

2.3.5 Tempering Heat Treatment

According to Hassan, (2009) tempering heat treatment is a process of heating martensitic steel or cast iron at a temperature below the eutectoid transformation temperature in order to make the steels and cast irons softer and more ductile. It is a process whereby quench hardened steelsand cast irons in the martensitic condition are heated to some predetermined temperature between room temperature and lower critical temperature, holding for an hour then followed by air or oil cooling (Alexey and Arnord 2006). Tempering therefore reduces hardness, strength and wear resistance marginally. However, this marginal loss is adequately compensated byadvantages gained by relieving of internal stresses, restoration of ductility,toughness and transformation of retained austenite (Khanna,2002). Tempering may be classified into low temperature tempering (150-250^{o}C) medium temperature tempering (350-450^{o}C), and high temperature tempering (500-600^{o}C).

2.4 Interrupted Quenching Processes.

These are processes normally applied during quenching to take care of problems during quenching (Hassan, 2009). Common interrupted quenching processes include:

2.4.1 Austempering

Austempering is one of the heat treatment methods used for hardening offerrous metals. It is an isothermal heat treatment that produces a structure that isstronger and tougher than comparable structures produced by conventional heattreatment methods (e.g. water quenching and tempering).The austempering process consists of austenitizing a ferrous component (steel or cast iron) to a temperature between 825-950^{o}C, soaking at this temperature for 1 to 2 hoursdepending on the component thickness, quenching from this temperature into ahot liquid medium maintained at a pre-selected temperature between 250-450^{o}Cthen removed and cooled to room temperature in air (Ause, 2008). Austempering cycle

is shown schematically on the isothermaltransformation diagram in (Figure 2.7). When austenite is cooled rapidly and held isothermally at a temperature between 250-450^{o}C, the austenite is transformed not to pearlite but to bainite (Tun and Lwin 2008). The bainite consist of an intimate mixture of ferrite andcementite but differs in structure. According to Singh *et al.*,(2001), cementite in bainite appears asfinely dispersed precipitates.

According to Ause, (2008) the austempered microstructure in cast ironwas documented by Flinn, and was alternately misnamed as Bainite and acicular ferrite. Years later, the structure was officially classified as ausferrite by ASTMand identified as a mixture of acicular ferrite and austenite. The onemajor area of application of austempering is in the production of austemperedductile iron (ADI) components which are used forautomotive part, machine tools, railroad construction and militaryapplications.

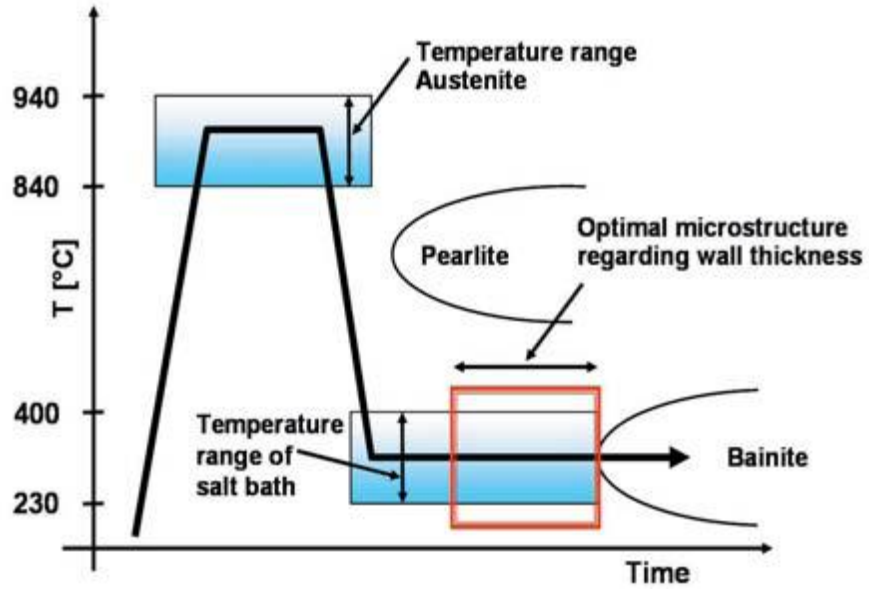


Figure 2.7: A Schematic Diagram of the Austempering Heat Treatment Process
(*Source*: Tun and Lwin 2008)

2.4.2 Martempering

This is similar to austempering, in that the cooling is interrupted just above the martensite start(Ms)temperatureand held for some time (Figure 2.8). Cooling is resumed before austenite

starts transforming to bainite and at a much slower rate. The holding time is shorter here. The structure formed is called tempered martensite (Hassan, 2009).

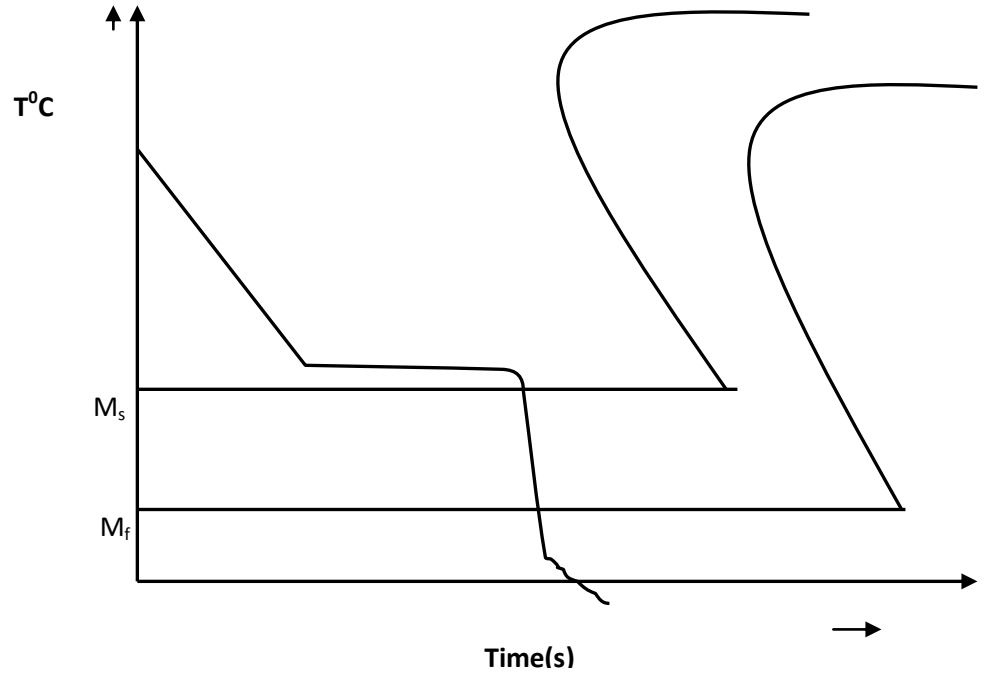


Figure 2.8: A schematic diagram of the Martempering heat treatment process.
(S*ource:*Khanna, 2010)

2.4.3Ausforming

Like austempering, the steel is quenched in a salt bath at a temperature just above the Ms temperature (250^{o}C). This temperature is maintained until bainite is about to form and the steel is mechanically worked as it cools to room temperature(Figure 2.9) (Hassan, 2009).

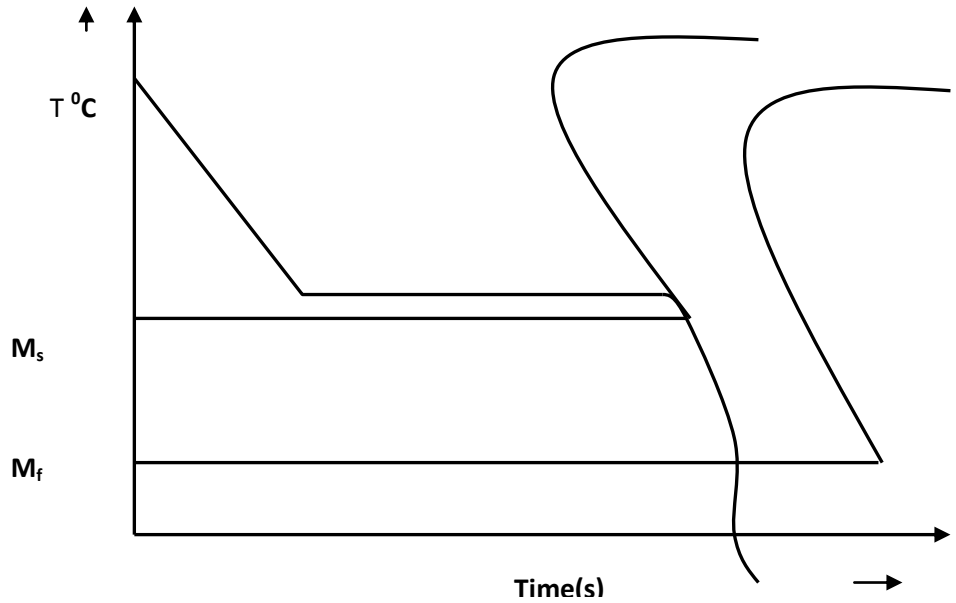


Figure 2.9: A schematic diagram of the Ausforming heat treatment process (S*ource:* Khanna, 2010)

2.5Austempered Structure in Steel andDuctile Cast Iron

Austempered Ductile Iron (ADI) is a group of ferrous materials whose mechanical properties can be varied over a wide range by a suitable treatment such as austempering. Singh *et al.*,(2001) reported that during austempering, two simple types of bainite are formed: upper and lower bainites. Metallographic observation reveals that upper bainite is feathery and the lower bainite is acicular. The Microstructure of upper bainite consists of ferrite lath with carbides precipitated on the surface of the laths, whereas lower bainite consists of ferrite lath with precipitates of carbide within the laths at an angle to the major axis of the ferrite lath (Khanna, 2010). The upper and lower bainites are shown schematically in Figures 2.10 and 2.11. Both the upper and lower bainites are nucleated by ferrite. According to Rajan *et al.*, (1988), the nucleated ferrite grows into bainitic plate on cooling and holding at a bainitic temperature region provided for by the molten bath. The bainite plate so formed is surrounded by austenite which is enriched in carbon (high carbon austenite). The mechanical properties of ductile Iron and ADI are primarily determined by the metal matrix. The

properties of ADI are due to its unique matrix of acicular ferrite and carbon stabilized austenite called ausferrite.

Ause (2008) pointed out that austempering takes place in two stages as follows:

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

Stage II: ($\gamma_{hc} \rightarrow \alpha + Fe_3C$) (high carbon austenite further decomposes to ferrite and carbide, a product known as bainite. Eric *et al.,* (2004) showed that, the austempering process in cast iron (especially ductile cast iron) differs from that in steel because, in ductile cast iron, the above two stages of reaction take place, whereas, for steels only the second stage occurs. The good combination of mechanical properties obtained in austempered ductile cast iron could be attributed to the unique ausferrite structure ($\alpha + \gamma$ hc) produced in stage I. The stage II reaction is however undesirable because it leads to embrittlement due to formation of Fe_3C (Cementite) (Ause, 2008).One of the variable known to interfere with the stability of the austenite/ferrite is the chemical composition of the cast iron or steel. It is generally known that, in the chemical composition of ductile cast iron, silicon content is always higher than in steel. Silicon is known to be ferrite stabilizer and a graphitizer (Lee *et al.*, 2003). The higher amount of silicon in ductile cast iron enables the formation of ferrite and high carbon austenite matrix of the stage I reaction. This high silicon content retards the formation of Fe_3C which is usually associated with bainitic structure in steels. Therefore, during austempering of steels, only the stage II reaction occurs because in the chemical composition of steel, silicon content is usually low, hence formation of cementite becomes easier. It has been reported that in ductile cast irons, the presence of 2.4 to 2.8 wt% silicon prevents the rapid formation of cementite (Fe_3C), but in steel the low percentage of silicon makes the condition favorable for cementite to form. The formation of cementite in cast iron

microstructures due to longer austempering times leads to lower toughness and ductility of cast iron (Lee *et al.*,2003).

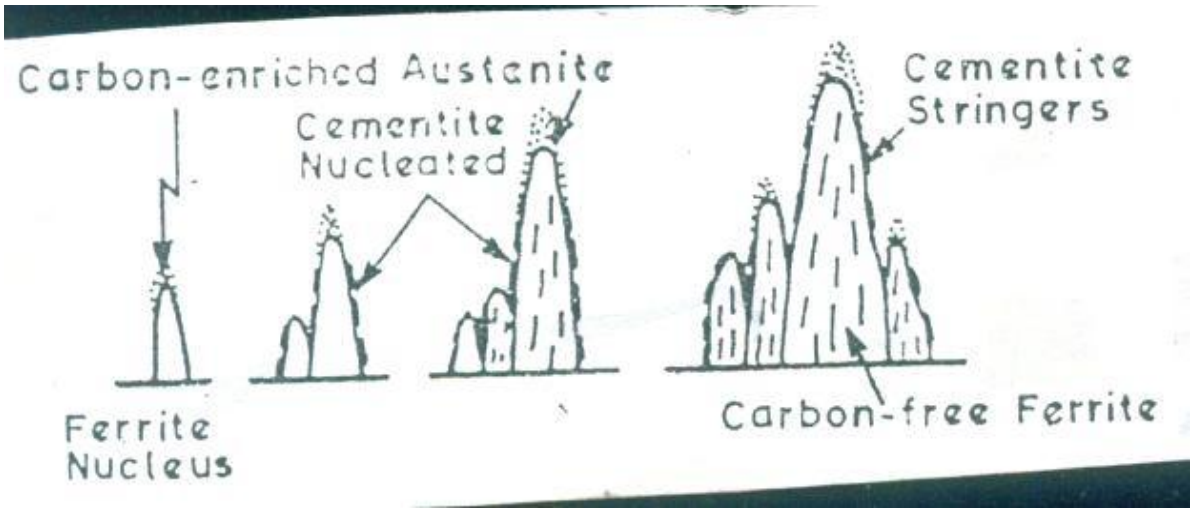


Figure 2.10: Upper Bainite (Feathery)*(Source*: Rajan *et al.*, 1988)

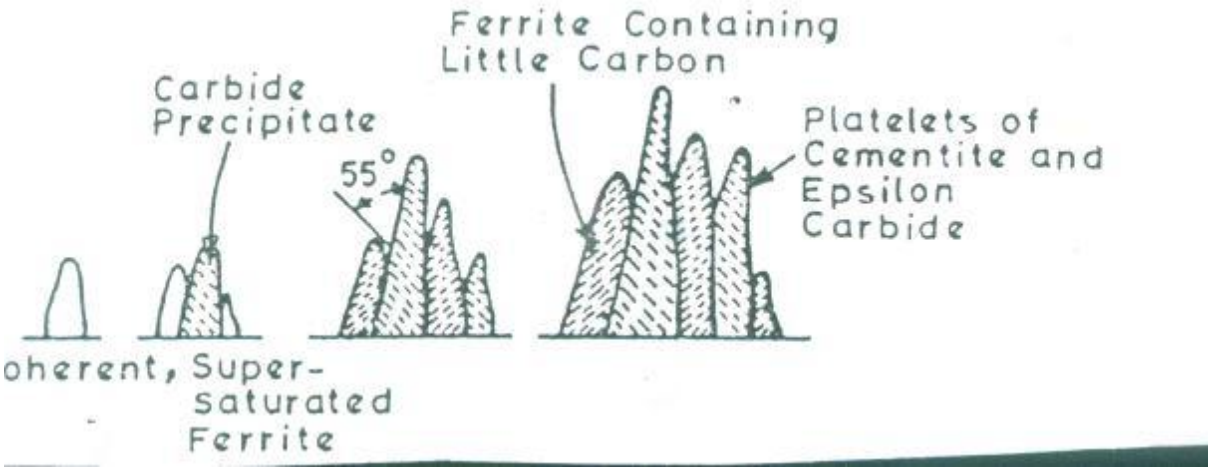


Figure 2.11: Lower Bainite (Acicular)*(Source*: Rajan *et al.*, 1988)

2.6Factors Controlling Austempering Reactions

There are certain conditions which enable the austempering reaction to proceed spontaneously. Branka and Branko (2004) reported that, for austempering process to be properly carried out, the following conditions must be adhered:

i Austenitizing Temperature and Time

ii Austempering Temperature and Time

iii Section Size and Alloying

2.6.1Austenitizing Temperature and Time

Austenitizing is the process of heating steel/cast iron components to austenite phase. Heating components to austenitizing temperature is the first step of almost all heat treatment processes applicable to ferrous alloys. Full austenitization usually ensures good quality heat treatment. In general, alloy content, the original microstructure and section size determine the time requiredfor austenitizing (Ause, 2008). The austenitizing temperature controls the carbon content of the austenite which in turn, affects the structure and properties of the austempered component (Rajan *et al.,* 1988).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^{o}C andaustenitizing times of approximately 2 hours are normal and have been shown to be sufficient to recarburize the matrix fully (Sani, 2008).Ause, (2008) in his work reported that, Kazerooni *et al.,* (1997) used salt bath furnace and investigated the influence of austenitizing temperatures of 950, 920, 870, 840, and 800^{o}C on austempering kinetics, austempered microstructure, mechanical properties and hardenability of Mn-Mo-Cu alloyed ductile iron, and found that, decreasing theaustenitizing temperature accelerates the stage I reaction and the introduction of ferrite into the austempered structure.

2.6.2 Austempering Temperature and Time

Austempering temperature is that temperature at which transformation of austenite to bainite occurs isothermally (Rajan *et al.,* 1988). The austempering temperature is the primary determinant of the final microstructure in any austempering heat treatment process. As the

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

2.6.3 Alloying andSection Size

The primary aim of alloying elements is to provide sufficient hardenability to the matrix of the metal intended to be austempered. Alloying additions are also usually made to ensure sufficient hardenability when thicker section castings are to be austempered otherwise, pearlite will form during quenching from the austenitizing temperature to austempering temperature (Kazerooni *et al.,* 1997).Ause, (2008) showed that, the major elements that play significant roles in enhancing properties during austempering are: carbon, molybdenum, manganese, nickel, cobalt, boron, silicon and copper. These elements are required and used singly or in combination in varying amounts to prevent/delay pearlite formation in given section sizes. Several researchers have been concerned with the influence of alloying elements on the mechanical properties of austempered ductile iron and steels. Lee *et al.,* (2003) investigated various ADI with high Si and Mn content, austenitized at 800^{o}C and 900^{o}C. They concluded that the optimum Si/Mn ratio of ADI was approximately 1.5 for the best combination of hardness, tensile, impact and wear property.

2.7Advantages of Austempering over Conventional Quench and Temper Heat

Treatment

Austempering process produces a structure that is stronger and tougher than comparable structures produced by conventional heat treatment. Ause, (2008) reported that, accordingto ASM metal handbook, (1970) Austempering is usually substituted for conventional quench and temperheat treatment for either one or both of two reasons:

i. To obtain improved mechanical properties

ii. To reduce the likelihood of cracking and distortion

In most applications, austempering is less expensive than conventional water quenching and tempering because, in austempering, the component is simultaneously hardened and tempered in the hot bath, hence time and cost of operation are reduced. The benefits of austempering also includes less distortion of the parts in quenching and a microstructure with certain mechanical advantages compared to oil or water quenching. Such mechanical advantages include higher tensile strength, improved impact toughness, high ductility, better wear resistance and lighter components (Sani, 2008).

2.8 Quenching Media for Austempering

As applied to the heat treatment of steels and cast irons, quenching is a process of rapid cooling from austenitizing temperature. A medium that is used for quenching is known as quenchant. The most commonly used quenching media for austempering are molten salt, molten lead and hot oil bath (Ause, 2008).

i. Molten salt bath: Is the quenching medium most commonly used in austempering because, it virtually eliminates the problem of a vapour phase barrier during the initial stage of quenching. Its viscosity is uniform over a wide range of temperatures and low at austempering temperature (near that of water at room temperature). This minimizes drag out losses, it remains stable at operating temperatures and is completely soluble in water. Thus, facilitating subsequent cleaning operation. The

molten salt commonly used has a composition of 50-60% potassium nitrate and 37 – 50% sodium nitrate (Dauda,2005).

ii. Molten lead bath: it is also used as a medium for interrupted quenching, although its use has been restricted because of its high initial cost. Furthermore, lead requires protection against oxidation and may give problem when lead or lead oxide adheres to the metal being quenched (Dauda, 2005).

iii. Hot oil also has some associated disadvantages. ASM-metal handbook (1970) pointed out that, oils exhibit chemical instability at high temperatures. Due to persistent vapour phase in oils, they are slower quenchants than molten salt at elevated temperatures(Ause, 2008).

2.9 Vegetable Oils

Vegetable oils are triglycerides, made up of 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-carbon double bonds. In saturated fatty acids, all the carbon atoms are linked to two hydrogen atoms and there are no double bonds (Sani, 2008).

According toLauralice *et al.*, (2005) the promising alternative fluids to petroleum oils as quenchants are vegetable oils, which are typically biodegradable and non-toxic.

Hassan *et al.*, (2007) reported that local vegetable oil such as palm kernel, soya bean, groundnut and shea butter have potentials as quenching media for hardening of grey cast iron.

2.9.1 Sesame Oil

Sesame oil *(Sesanum indicum)*is an edible vegetable oil derived from sesame seeds. It has a distinctive nutty aroma and taste. Historically, sesame was cultivated more than 5000 years ago as a drought-tolerant crop and was able to grow where other crops failed (Raghar *et*

al.,1990). In general, sesame oil contains oleic (35.9-47%), linoleic (35.6-47.6%), palmitic (8.7-13.8%), stearic (2.1-6.4%), as well as arachidic acids (0.1-0.7%) (Borchani *et al.*,2010). In Nigeria, the notable colours for sesame seeds are white, yellow and black (Fariku *et al.*, 2007).

Nigeria is richly endowed with different types of vegetable oils found in various parts of the country. Most of the vegetable oils produced in the country are mainly used in food, soap, candles and pharmaceutical industries. The varieties of vegetable oils includes: cottonseed oil, groundnut oil, palm kernel oil, palm oil, coconut oil, castor oil, shear butter oil and Sesame 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 (Sani, 2008).

2.10Review of Previous Works

Because of the undesirables side effects of molten salt and lead baths as austempering media, researchers over the years have been searching for alternatives. Among others are:

Ause (2008) investigated the use of hot bitumen bath as a quenching medium for austempering of steel and ductile cast iron. It was concluded that bainitic- ferritic structures could be formed from hot bitumen bath.Bitumen adheres to the surfaces of component which requires nonpolar solvent such as kerosene to wash it out. This ofcourse add cost to the finishing operations.

.Akor and Tuluem (2014) studied the effect of austempering time on the mechanical properties of ductile iron austempered in rubber seed oil. It was found that rubber seed oil could be used as hot bath for the austempering of ductile cast iron. But because of the toxicity of the oil limit the oil for use in heat treatment processes.

Ademoh (2015) worked on the possibility of using Nigerian neem seed oil as an austempering quenchant for locally recycled steels. It was reported that neem seed oil could be used as an austempering medium. The limitation of the neem seed oil for use as an austempering heat treatment is that, it is expensive and is not always available.

Aigbodion *et al.,* (2005) evaluated the use of engine oil as austempering medium for the austempering of ductile iron for the production of agricultural machines and farm implements.It was concluded that bainitic- ferritic structures could be formed. The engine oil has low flash point (260 ^{o}C.) which cannot be used at higher austempering temperatures above 260 ^{o}c. Lawrence, (2011) investigated the use of Khaya Senegalensis (Mahogany) seed oil as quenching medium in austempering process of ductile iron. . It was concluded that ausferric structures could be formed from Khaya Senegalensis (Mahogany) seed oil as austempering medium.The limitation of the Khaya Senegalensis (Mahogany) seed oil for use as an austempering heat treatment is that, it is expensive and is not available.

Sani, (2008) investigated the potential of using some vegetable oils as quenchants for austempering of steel and cast irons. He observed that hot shea butter and groundnut oils were effective as austempering quenchants for medium carbon steel and ductile iron.The limitation of shea butter and groundnut oils is that upper bainite structure could be attained because of thelow flash points.

Hamza and Mohammed (2008) studied the physico-chemical properties of oil extract from Sesanum Indicum L. seed and his findings reflected that, the oil was found to benon-toxic, cyanide free and suitable for human usage.

In summary, it can be seen from the reviewed studies and researches, it is evident that little attention has been given ontoxicity, availability, cost and possibilities of the quanchant to be

operated at upper bainite temperature ranges have not been investigated. Therefore present study was carried out in order to address the aforementioned research gaps.

CHAPTER THREE

MATERIALS AND METHODS

3.1 Materials and Equipment

3.1.1 Materials

Thematerials used in this research work include: ductile cast iron samples (which were obtained from National Automotive Design and Development Council Zaria, Kaduna State), Sesame oil (Beniseed oil)*(Sesanum indicum oil)*(which were obtained from Kurmi Market Kano)polishing powder, water, grinding papers, polishing cloth, cotton wool, copper piece, nitric acid and ethanol.

Table 3.1: Chemical composition of the as-received ductile cast iron

Element	C	Si	Mn	P	S	Cu	Ni	Mo	Mg	Fe
Content (%)	3.81	2.70	0.26	0.06	0.042	0.32	1.85	0.2	0.04	90.72

3.1.2 Equipment

The equipment used include: Thermocouple, Optical Pyrometer,oil bath, electrically heated muffle furnace, Optical Metallurgical Microscope (OM), Scanning Electron Microscope (SEM), lathe machine, polishing machine, Vickers hardness tester, Hounsfield tensometer, Hounsfield balance impact testing machine.

3.2 Methods

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

3.2.1 Machining of test samples

The ductile cast irons (rod form) were machined to standard impact, tensile test and hardness/microstructural analysis samples.

3.2.2 Normalizing of Samples

135 samples were heated to 900^{o}C and soaked for one hour for the necessary transformation to occur and the attainment of homogenization after which the samples were air cooled. This normalizing heat treatment was conducted purposely to remove the effects of undesirable structures due to machining and also to restore original condition of the structure and induce homogeneity in the samples.

3.2.3 Quench Hardening and Tempering

30 samples were loaded into the furnace, heated to 900°C, soaked for one hour and then quenched in water, Brine and Sesame oil at room temperature. Thereafter 15 samples in the as quenched conditions from all the quenching media mention were tempered by reheating to 350^{o}C, soaked for one hour and air cooled.

3.2.4 Austempering in Hot-Sesame oil Bath

Normalized tensile, impact and hardness test/microstructural analysis samples of ductile cast iron were austempered as follows: 25 samples were loaded into the furnace, heated to 850°C,

soaked for one hour, then removed and quenched in hot Sesame oil bath at 250°C. After one hour, the first set of samples (one hardness, two impact and two tensile) were removed from the hot Sesame oil bath, cooled in air. Another set of samples were removed after 2, 3, 4 and 5 hours respectively, and cooled in air. The same process was repeated as described above for austenitizing temperature of 900^{o}C. Similarly, the above process was repeated for samples austempered in hot sesame oil bath at 300^{o}C.

3.2.5Physicochemical characteristics of sesame oil

(a) Iodine value (IV)

Oil sample was dissolved in 30 ml of 3:2 v/v CH3COOH and cyclohexane solution. After that, 50 ml of Wijs solution were added and kept undisturbed in the dark. Subsequently, it was titrated with a 0.1M $Na_2S_2O_3$ (sodium thiosulfate) solution. A blank test was carried out performed at the same time with the same quantities of the same reagents. The iodine value was computed using the relationship

$IV = [Ar \times (V_B - V_S) \times N]/(10 \times W)$

Ar = atomic weight of iodine, 126.90 g

V_B = volume of 0.1 M $Na_2S_2O_3$ consumed by the blank test (ml)

V_S= volume of 0.1 M $Na_2S_2O_3$consumed by the actual test (ml)

N = molarity of the $Na_2S_2O_3$

W = mass of the oil sample (g)

(b) Peroxide value (PV)

5 g of oil sample dissolved in a 30 ml of mixture of chloroform and acetic acid (2:3). 0.5 ml of saturated potassium iodide (KI) solution was also added. The mixture was shaken for 1 minute followed by addition of 30 ml of water. The mixture then titrated with 0.01 M sodium thiosulfate, until the yellow color is almost discharged. 5 ml of starch was added and the

titration continued, until the blue color disappear.A blank test was carried out under the same conditions.

P V= [1000 (V_T− V_B) × N]/W

V_T= volume of 0.01 M $Na_2S_2O_3$ consumed in the actual test (ml)

V_B= volume of 0.01 M $Na_2S_2O_3$ consumed in the blank test (ml)

N = molarity of the $Na_2S_2O_3$

W = mass of the oil sample (g)

(c) Saponification value (SV)

0.1 M KOH (potassium hydroxide) solution was prepared with 95 % ethanol and distilled water. 5 g of oil sample was weighed in a conical flask, the flask was connected to an air condenser and boiled until the oil is completely saponified, cooled and then titrated with 0.5 M HCl (hydrochloric acid) using phenolphthalein as indicator. A blank test was carried out under the same conditions (without oil sample in the conical flask). Saponification Value was calculated using the following relation:

SV = [Mr × (V_B– V_T) × N]/W

Mr = molecular weight of KOH, 56 g

V_B= volume of 0.5 M HCl consumed in the blank test (ml)

V_T= volume of 0.5 M HCl consumed in the actual test (ml)

N = molarity of the HCl

W = mass of the oil sample taken for the test (g)

(d) Acid value

1 g of oil sample was weighed in a conical flask. 25 ml of propan-2-ol was added to the sample to dissolve the oil. Then two drops of phenolphthalein were added to the mixture. 0.1 M of KOH was poured in the burette and titrated against the mixture of the oil samples. Yellow colour changed to pink, the initial and final readings were recorded, thereafter, the

total average titer value also recorded. The acid value was calculated using the following equation

Acid value = (molarity of KOH * molar mass of KOH * titer value)/mass of oil

(e) Density

Mass and volume of oil were accurately measured using weighting balance and cylinder, the density was computed using relationship

Density= mass / volume

(f) Viscosity

The spindle of the viscometer was inserted into the sesame oil and it was run after which the viscosity was indicated on the scale.

(g) Boiling point

The Thiele tube was half full filled with sesame oil and was heated evenly. The tube was attached to the lower end of thermometer with two small rubber bands and the capillary was inserted into the tube. The bubbles of air was observed in the capillary indicating that the oil was boiling and the boiling temperature was taken.

(h) Smoke point

The evaporation pan was half filled with sesame oil and the test flame was lighted and adjusted in such a way that the temperature of oil increases by $5^{o}C$ to 10 per minute. When the smoke started emanating,the temperature was noted by means of optical pyrometer which represent the smoke point.

(i) Flash point

The oil cup was filled with the test oil up to the mark in thePensky Marten's apparatus. The lids on the top through which the thermometer and a stirrerwere inserted to ensure that the flame exposure device was fixed on the top. The test flame was lighted and adjusted to about 4 mm in diameter, the apparatus was heated in such a way that the temperature of oil

increases by 5 to 60 per minute. At every 10 ^{o}C rise of temperature test flame was introduce into the oil vapor. This was done by operating the shutter. On moving knob of shutter, test flame was lowered in oil vapors through opening. When the test flame caused a distinct flame in the interior cup, the temperature was notedwhich represent the flash point

The austempered samples were then subjected to tensile, impact and hardness tests**.** The distribution of samples for the various tests is present in Table 3.2.

Table 3.2: Number of samples used for various heat treatment operations.

Sample condition	No. of samples required for mechanical property tests and metallographic analysis			Total
	Tensile test	Impact test	Hardness and metallographic analysis	
As-received	2	2	1	
Normalized	2	2	1	
Quenched and Tempered	2x6	2x6	6	
Austempered	2x20	2x20	20	
				140

3.3 Mechanical Properties Tests

3.3.1 Tensile Test

Samples were machined to the standard dimensions prior to the heat treatment operationas specified in ASTM A370 (2007).The test method adopted is ASTM E8. The test sample was mounted at it ends onto the holding grips of the testing apparatus. The maximum loads were recorded directly from the Hounsfield tensometer. Following the fracture, the two broken ends of the specimens were fitted together and final gauge length was measured. The readings obtained were used to calculate the tensile strength and percentage elongation. The dimensionsof the tensile test sample used is shown in Figure 3.1.

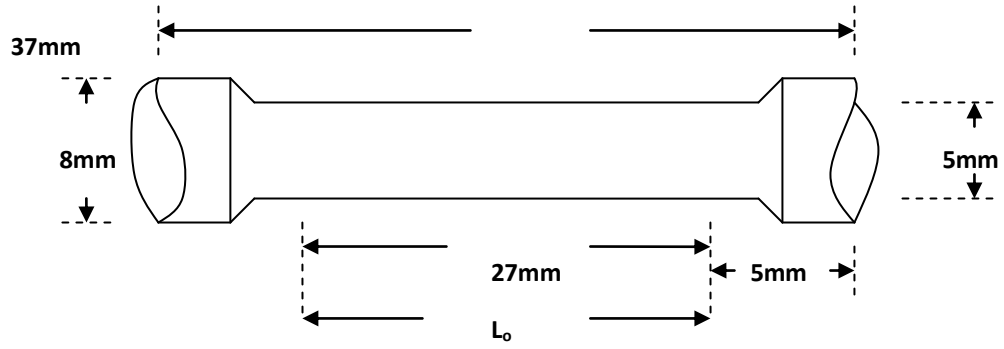


Figure 3.1: Standard tensile test piece (Source: ASTM, 2007)

3.3.2 Hardness Test Measurement

The hardness values of untreated and heat treated samples were obtained using a Vickers hardness tester. The hardness test samples were prepared to standard dimension as specified in ASTM A370 (2007). The test method adopted is ASTM E18. A diamond indenter was used to indent the surface of the test specimen by the application of static load of 30kgf, which was maintained for fifteen minutes. Both diagonals of the impression were measured using a lower power graduated microscope. The mean value of the diagonal length was used in the determination of the hardness number.The procedure was repeated for 3 different points of the test piece and the average value was recorded.

3.3.3 Impact Test

The impact test samples were machined to standard dimensions as specified in ASTM A370 (2007).The test method adopted is ASTM E23.The samples were machined to 8mm diameter and 45mm length with a 1mm deep V-notch. Before the test, the pendulum was set to a potential energy position of 162.75J. The test specimen was griped vertically in a vice, the trigger was released and the registering pointer of the quadrant scale were indicated the energy absorbed in Joules to break the specimen. The energy absorbed in breaking the sample was recorded and the same was repeated for other test pieces.

3.4 Microstructural examination

All the specimens (both untreated and heat treated) were prepared for optical microscopy (OPM)/scanning electron microscope(SEM) using the standard metallographic techniques. The specimens were ground on a water lubricated silicon carbide abrasive papers of 180, 240, 320, 400 and 600 grit sizes. Polishing was carried out on two 15cm rotating discs of a METASERV universal polishing machine with synthetic velvet polishing clothes impregnated with 1-micron Alumina paste. The specimens were etched with 2% Nital solution using the swabbing method with cotton wool soaked in the etchant.

The microscopic examinations were carried out on M100 optical metallurgical microscope and the microstructures obtained were captured with the aid of an in built camera. SEM werealso carried out on most of the austempered samples. The surface of the specimens prepared for examinationwere polished and then etched.They were then scanned with an electron beam, and the reflected (or back-scattered) beam of electrons were collected, displayed at the same scanning rate on a cathode ray tube (similar to a TV screen) and the images on the screen were photographed.

CHAPTER FOUR

RESULTS AND DISCUSSIONS

4.1 Introduction

This chapter presents mechanical properties tests result, optical micrographs, SEM images, and FTIR analysis of the austempering oil used; alongside with the discussion of such results.

Table 4.1: Physico-chemical properties of the Sesame oil used

Iodine Value (g/100g)	Acid value (mg KOH)	Peroxide value (m_{eq} KOH/g)	Saponification value (mg KOH)	Density (g/cm^3)	Viscosity at 25.5°C(cp) before Aust. HT	Viscosity at 25.5°C(cp) after Aust. HT
Sesame Oil 113.2	0.5	7.7	189	0.914	40.2	48

Smoke point(°C)	Boiling point(°C)	Flash point (°C)
223304	391	

4.2 Mechanical Properties of Ductile Cast Iron in the as-Received, Normalized, Quenched, and Quenched -Tempered Conditions.

Figures 4.1, 4.2, 4.3 and 4.4 depicted results obtained from mechanical tests of the quenched and quenched -tempered samples in the various media.

4.2.1 Tensile properties

a. Tensile strength

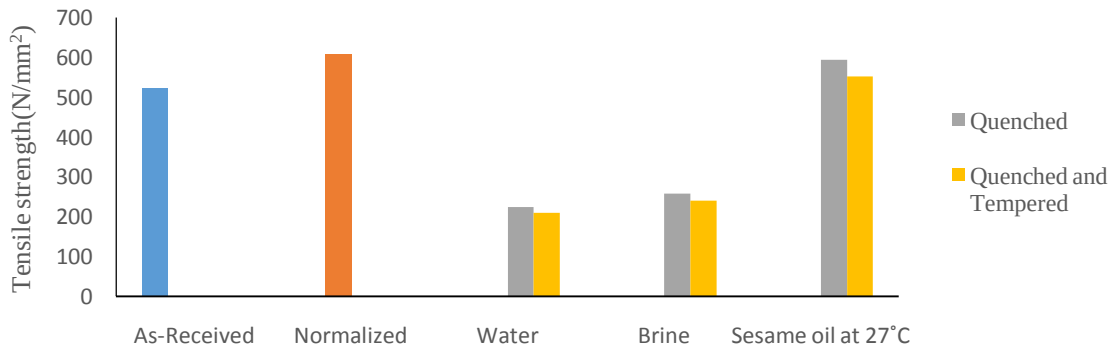


Figure 4.1: Profile of tensile strength of the ductile cast iron samples quenched in different quenching media.

b. Percent Elongation

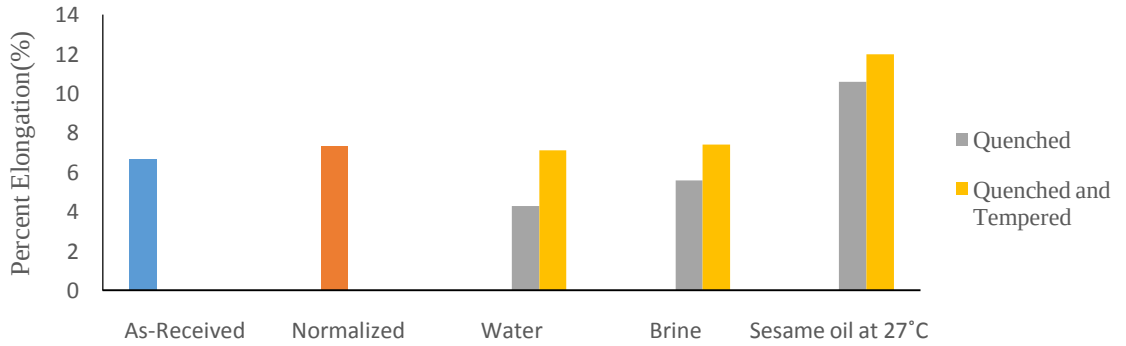


Figure 4.2: Profile of percent elongation of the ductile cast iron samples quenched in different quenching media.

Figures 4.1 and 4.2 show the tensile strength and percent elongation respectively for samples quenched in different media. Samples quenched in Water and brine showed lower tensile strength compared with those quenched in Sesame oil. This could be attributed to the high thermal and transformation stresses developed after quenching. These results are similar to the earlier observation of (Hassan *et al.*, 2009). Samples quenched in Sesame oil (at room temperature) showed highest percent elongation compared with those in water and brine which could be attributed to the fact that water and brine have higher quenching severity than Sesame oil.However, tempering resulted in decrease of tensile strength and increment of the percent elongation. This is in line with the earlier observation by Dodo (2015).

4.2.2 Hardness

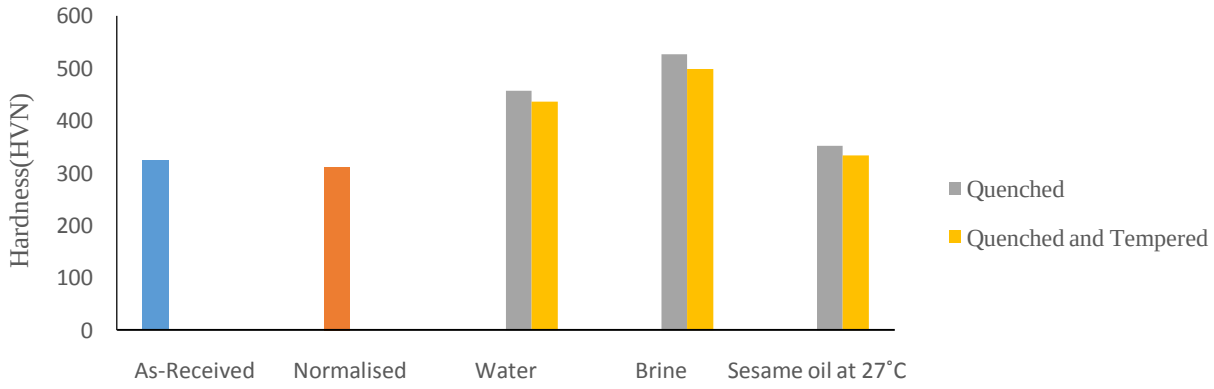


Figure 4.3: Hardness profile of the ductile cast iron samples quenched in different quenching media.

Figure 4.3 shows the hardness values for samples quenched in water, brine and Sesame oil. It can be observed that brine quenching resulted in maximum hardness. The reason might be due to Na^+ and Cl^- ions destabilizing the vapour blanket that is formed around the samples during the first stage of cooling and hence resulting in maximum heat removal which eventually led to highest hardness value. Water gave intermediate hardness value while Sesame oil gave the least hardness and hence has the lowest quenching severity.Tempering resulted in decrement of the hardness values that were obtained after quenching. These results are in agreement with earlier observation of Vivek *et al.*, (2014).

4.2.3 Impact Strength

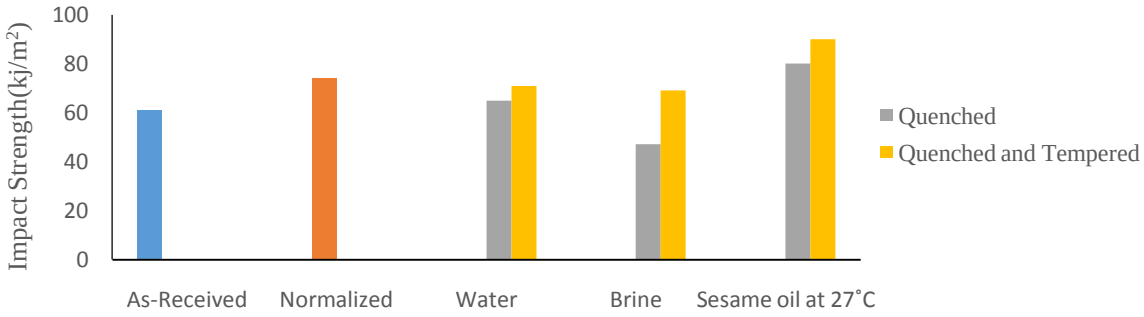


Figure 4.4: Profile of impact strength of the ductile cast iron samples quenched in different quenching.

Figure 4.4 shows the impact strength (energy absorbed per unit area under the notch) results for samples quenched in Water, Brine and Sesame oil. It showed that Sesame oil quenched samples produced the highest impact strength, while brine quenched samples produced the least impact strength. Tempering resulted in increment of the impact strength that were obtained after quenching the samples.These observationsare in line with what was obtained in literature (Balogun, 2008).

4.3 Mechanical Properties of Austempered Ductile Cast Iron

4.3.1 Tensile properties

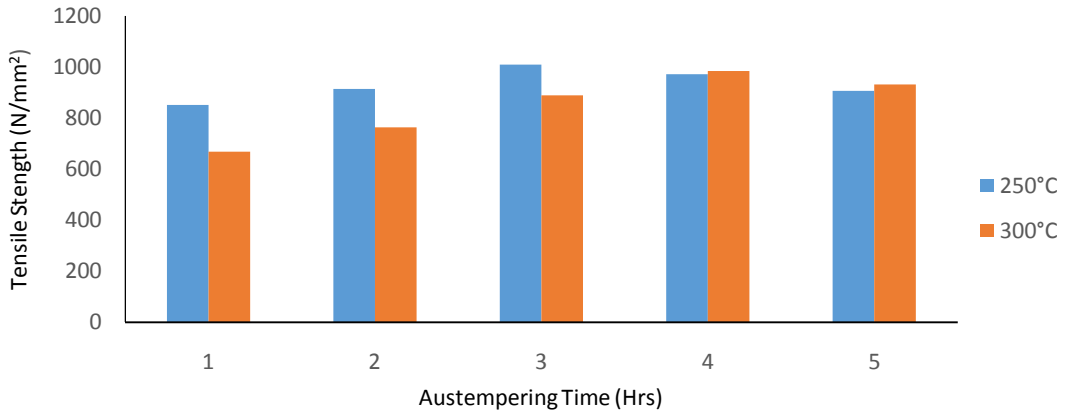


Figure 4.5: Profile of tensile strength of the ductile cast iron samples austenitized at 850°C and austempered in Sesame oil

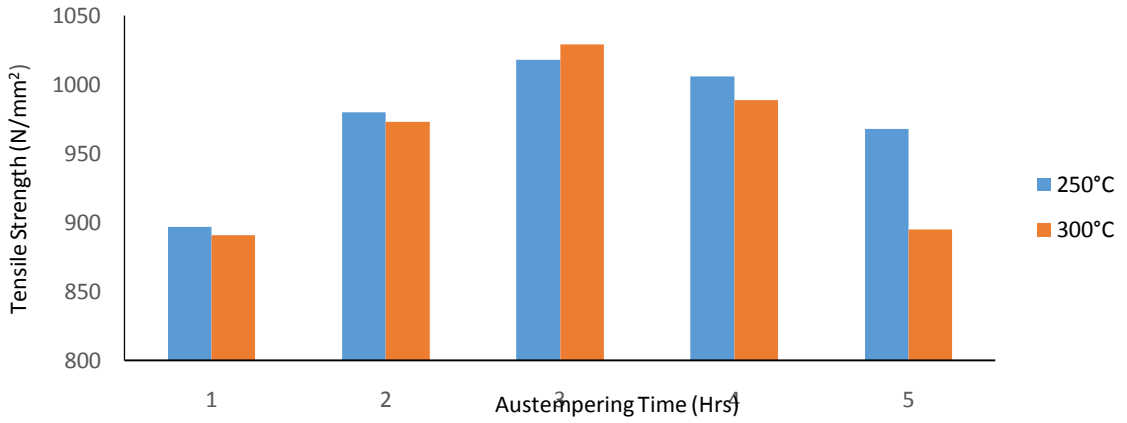


Figure 4.6: Profile of tensile strength of the ductile cast iron samples austenitized at 900°C and austempered in Sesame oil.

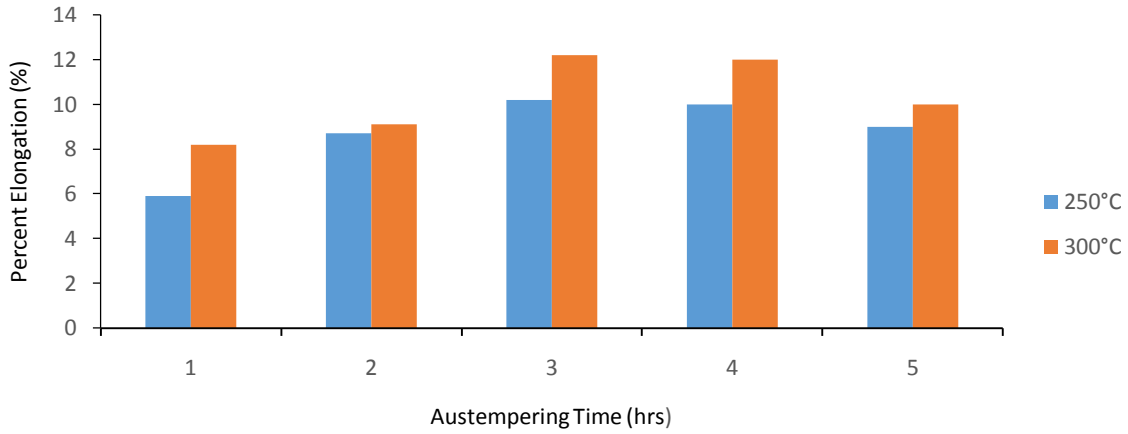


Figure 4.7: Profile of percent elongation of the ductile cast iron samples austenitized at 850°C and austempered in Sesame oil.

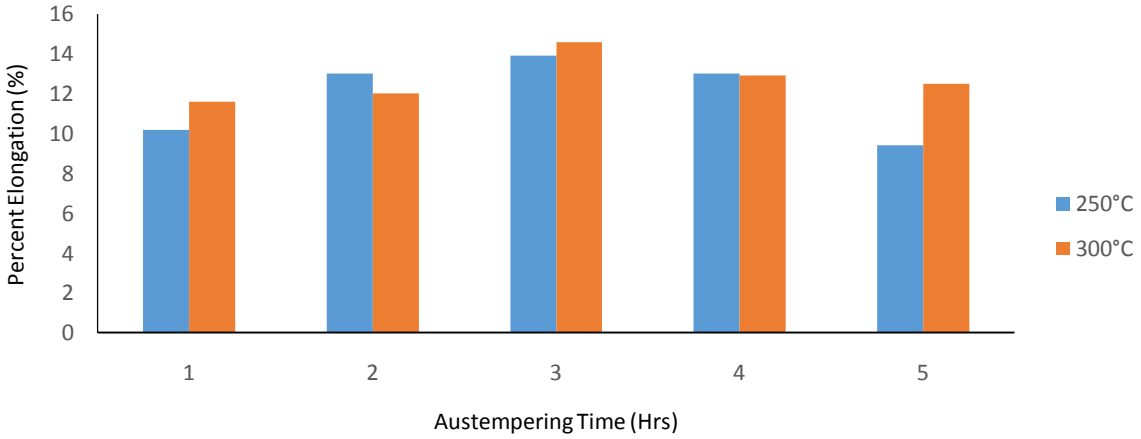


Figure 4.8: Profile of percent elongation of the ductile cast iron samples austenitized at 900^{o}C and austempered in Sesame oil.

Figures 4.5 and 4.6 show the effect of austempering time on the tensile strength of ductile cast iron samples austempered in hot Sesame oil bath at 250^{o}C and 300^{o}C for 1-5hours from austenitizing temperatures of 850^{o}C and 900^{o}C respectively. It was observed that as the austempering time increased the tensile strength increased, reaching a peak at 3 hours and then decreased for both austenitizing temperatures. This increase may be due to formation of stage 1 austempering reaction which might have caused large fraction of austenite to transform to ausferrite structure. At the austempering times of 4 and 5 hours, slight decrease of tensile strength was observed. This decrease could be attributed to the setting in of stage II austempering reaction. In the stage II reaction, stabilized high carbon enriched austenite transformed to bainitic ferrite plus ε-carbide. The brittle ε-carbide formed in this stage is responsible for the decrease, of the tensile strength at higher austempering times. The sample austenitized at 900^{o}C had the highest tensile strength value of 1029N/mm^{2} austempered at 300^{o}C for 3hours. This is in agreement with the earlier researches carried out by Sandeep, (2012) and Lawrence, (2011). Subsequently, a progressiveincrease oftensile strength values as theaustenitizing temperaturesincreases from 850^{o}C to 900^{o}C were also observed. This is because enlargement of austenite grains at higher austenitizing temperaturesimproves hardenability (Totten, 2006). However, the tensile strength values (Figure 4.5 and 4.6) show

a decreasing trend with increasing austempering temperature from 250^{o}C to 300^{o}C This may be attributed to the progressive decrease in carbon contents in the bainitic ferrite, which is similar to that reported by Elliot and Yazdani (1999).

Figure 4.7 and 4.8 represent the effect of austempering time on the percent elongation of ductile cast iron samples austempered in hot Sesame oil bath at 250^{o}C and 300^{o}C from austenitizing temperatures of 850^{o}C and 900^{o}C. The percentage elongation of the samples for both austenitizing temperatures increased as the austempering time increased, reaching a maximum at 3 hours then decreased. This increase may be due to progression of stage 1 austempering reaction which might have caused considerable quantity of austenite to transform to ausferrite structure. At the austempering times of 4 and 5 hours, the percent elongation decreased. This decrease could be attributed to the setting in of stage II austempering reaction.This is in agreement with Ause (2008). The samples austenitized at 900^{o}C proved to have the highest percent elongation compared to those austenitized at 850^{o}C. From the values (percentage elongation) obtained,shows that increasing austenitizing temperature from 850^{o}C to 900^{o}C had effect on the percentage elongation. This is similar to the report of Bisht, (2009).

4.3.2 Hardness

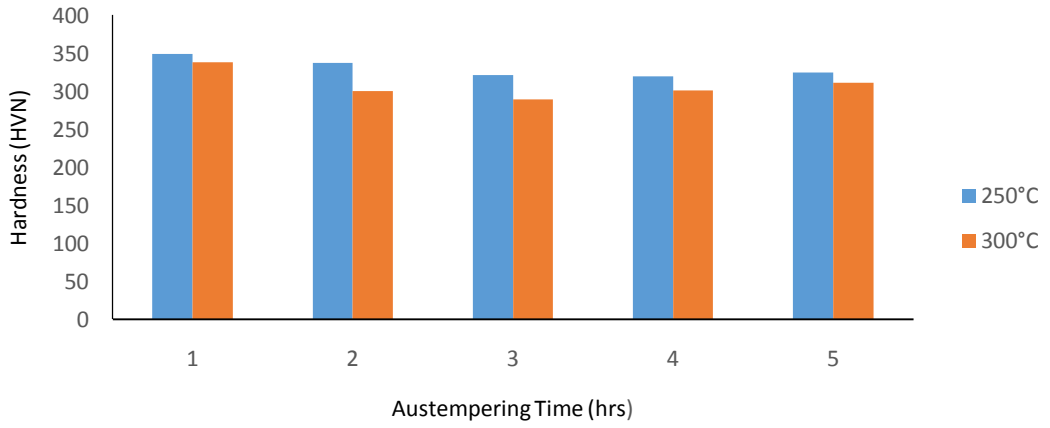


Figure 4.9: Hardness profile of the ductile cast iron samples austenitized at 850^{o}C and austempered in Sesame oil.

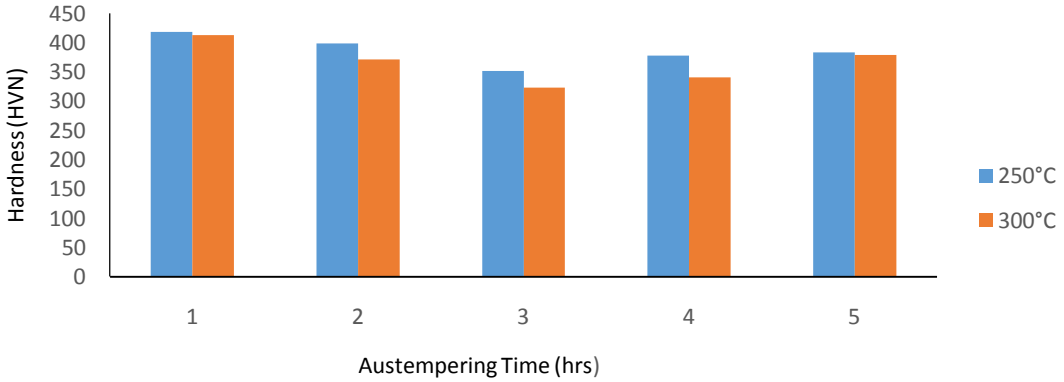


Figure 4.10: Hardness profile of the ductile cast iron samples austenitized at 900^{o}C and austempered in Sesame oil.

Figures 4.9 and 4.10 illustrate the effect of austempering time on hardness of ductile cast iron samples austempered in hot Sesame oil bath at 250^{o}C and 300^{o}C for 1-5hours from austenitizing temperatures of 850^{o}C and 900^{o}C.Hardness values were found to be high at initial austempering times but decreased to a minimum at 3 hours and then increased as the austempering time was increased. The higher hardness values obtained at the austempering time of 1hour may be due to the insufficient time for reasonable austempering reaction to have occurred before the sample was removed from the hot sesame oils bath, leading to high proportion of retained austenite. The increase in hardness at austempering times of 4 and 5 hours may be attributed to the presence of ε-carbide formed during stage II reaction. This observation is similar to the samples austempered in molten salt bath as reported by Almaida and Hasan (2009). An increase in hardness as the austenitization temperature was increased from 850^{o}C to 900^{o}C was observed, and reason for this could be attributed to the enlargement of austenite grains which improves hardenability (Totten, 2006). It was also observed that, the hardness showed a decreasing trend with increasing austempering temperature from 250^{o}C to 300^{o}C. This is attributed to the progressive decrease in high carbon contents in the bainitic ferrite. This is in line with what Adewuyi and Afonja (2000) established.

4.3.3 Impact Strength

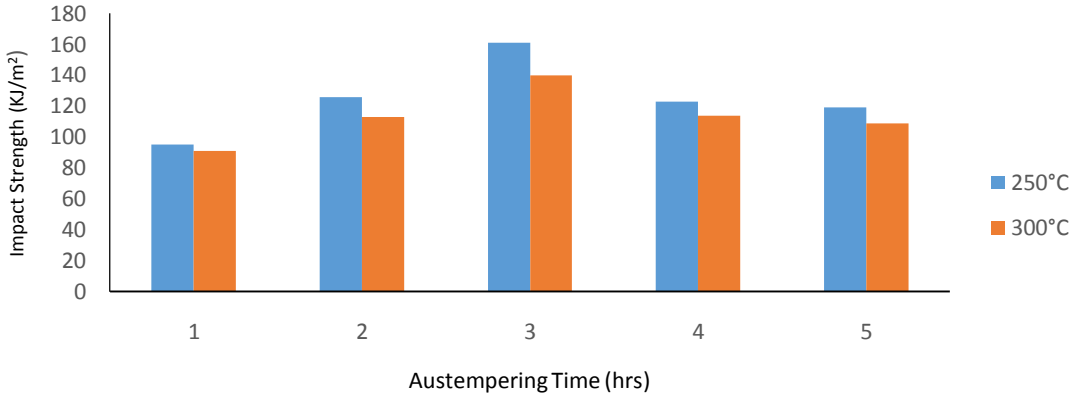


Figure 4.11: Profile of impact strength of the ductile cast iron samples austenitized at 850^{o}C and austempered in Sesame oil.

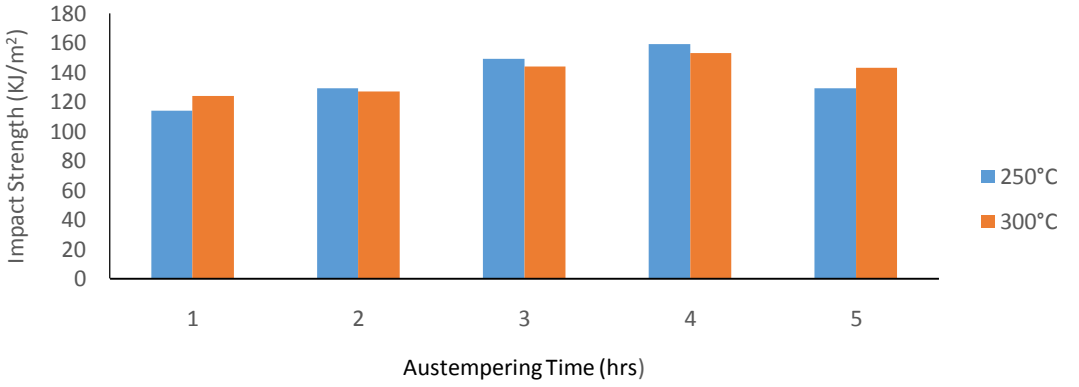


Figure 4.12: Profile of impact strength of the ductile cast iron samples austenitized at 900^{o}C and austempered in Sesame oil.

Figures 4.11 and 4.12 indicate the effect of austempering time on the impact strength of ductile cast iron samples austempered in hot Sesame oil bath at 250^{o}C and 300^{o}C for 1-5hours from austenitizing temperatures of 850^{o}C and 900^{o}C.The results clearly show that, as the austempering time increase, impact strength of the samples increased until it reached 3 hours. The decrease in impact strength by samples austempered at 4 and 5 hours, could be attributed to the brittleness set into the samples because of the start of stage II austempering reaction. These observations are in line with Ause, (2008).

4.4 Microstructures

The microstructures of the as-received, normalized, quenched and quenched-tempered samples are shown in Plates 1 to 3.

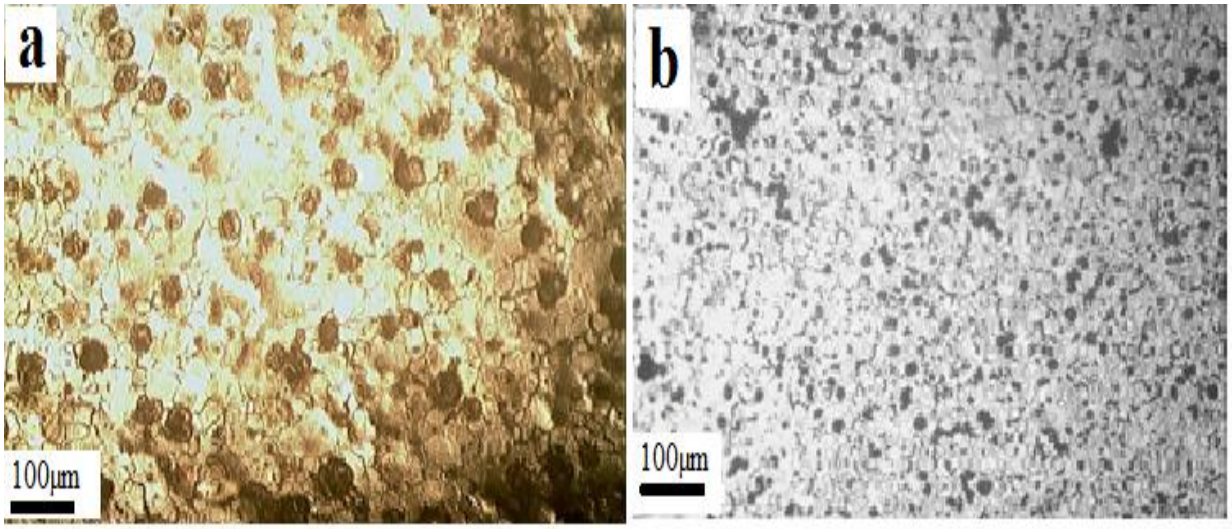


Plate 1: Optical micrograph of ductile cast iron. (a) as-received; the structure consists of graphite nodules with grain boundary ferrite in pearlite matrix; (b) after normalizing; the structure consists of graphite nodules in pearlite matrix, 2% Nital etch. (x100).

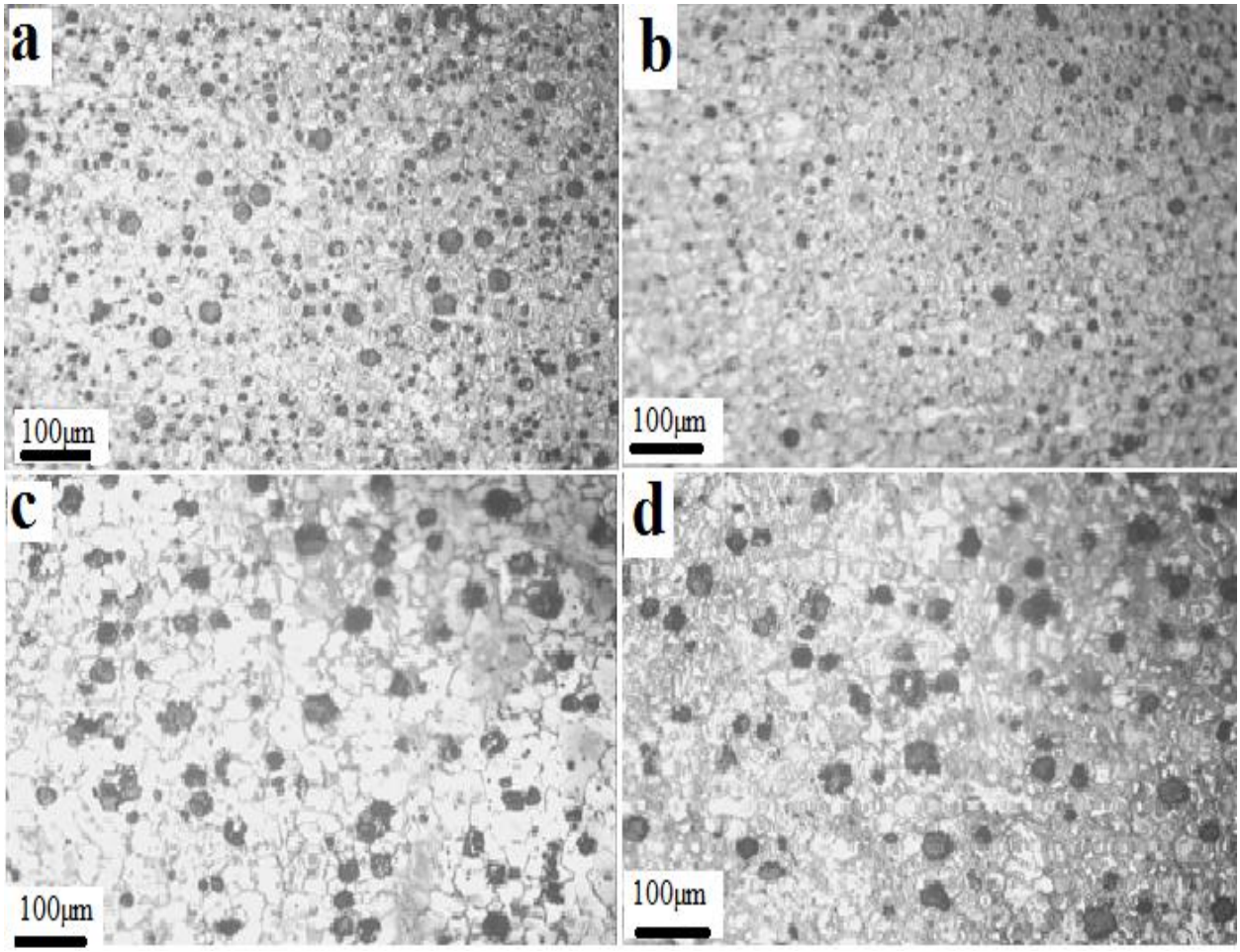


Plate 2: Optical micrograph of ductile cast iron quenched (a) in water; a structure of martensite (dark) and retained austenite (light-coloured areas), (b) in water and tempered at 350^{o}C; the structure consists of tempered martensite (c) in Brine; a structure of martensite (dark) and retained austenite (light-coloured areas) (d) in Brine and tempered at 350^{o}C; the structure consists of tempered martensite, 2% Nital (x100).

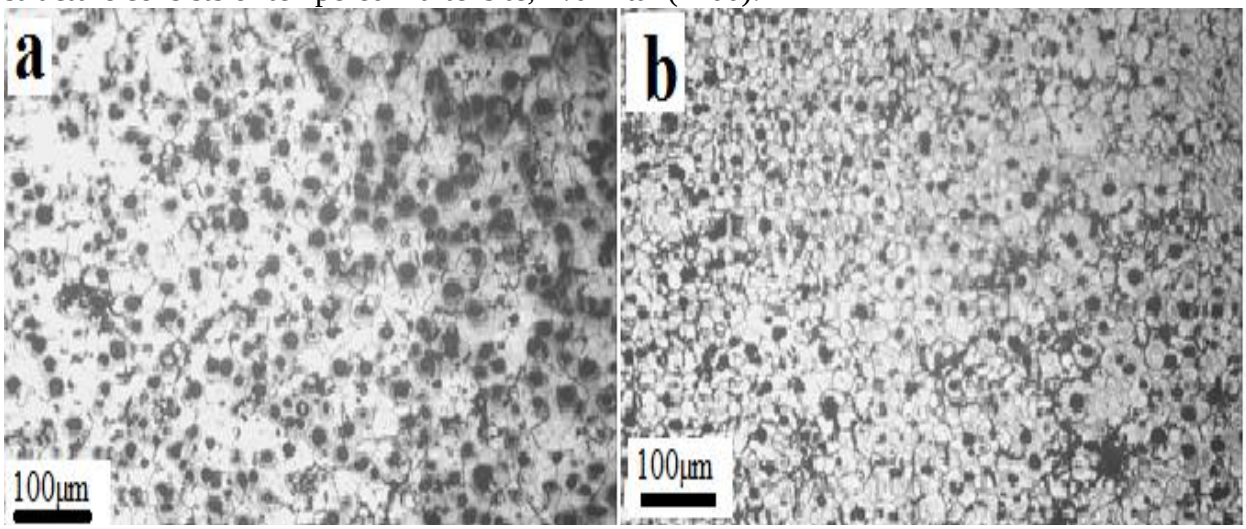


Plate 3: Optical micrograph of ductile cast iron quenched (a) in Sesame oil at room temperature; a structure of martensite (dark) and retained austenite (light-colored areas), (b) in Sesame oil at room temperature and tempered at 350^{o}C; the structure consists of tempered martensite, 2% Nital (x100).

The as-received structure of the test sample is shown in Plate 1(a); the structure consists of graphite nodules with grain boundary ferrite in pearlite matrix. The normalized structure of the ductile cast iron used is shown in Plate 1(b); the structure consists of graphite nodules in pearlite matrix. The microstructures of the test samples quenched in water, brine and sesame oil at room temperature are shown in Plates 2a, 2c and 3a, respectively. Brine quenched samples showed high proportions of martensite (because of the high hardness value obtained from the sample). Sesame oil (at room temperature) quenched samples (plate 3) showed martensite proportion lower than that of Brine and water quenched samples. All microstructures are associated with retained austenite. This is in line with the findings of Vivek *et al.,* (2014).

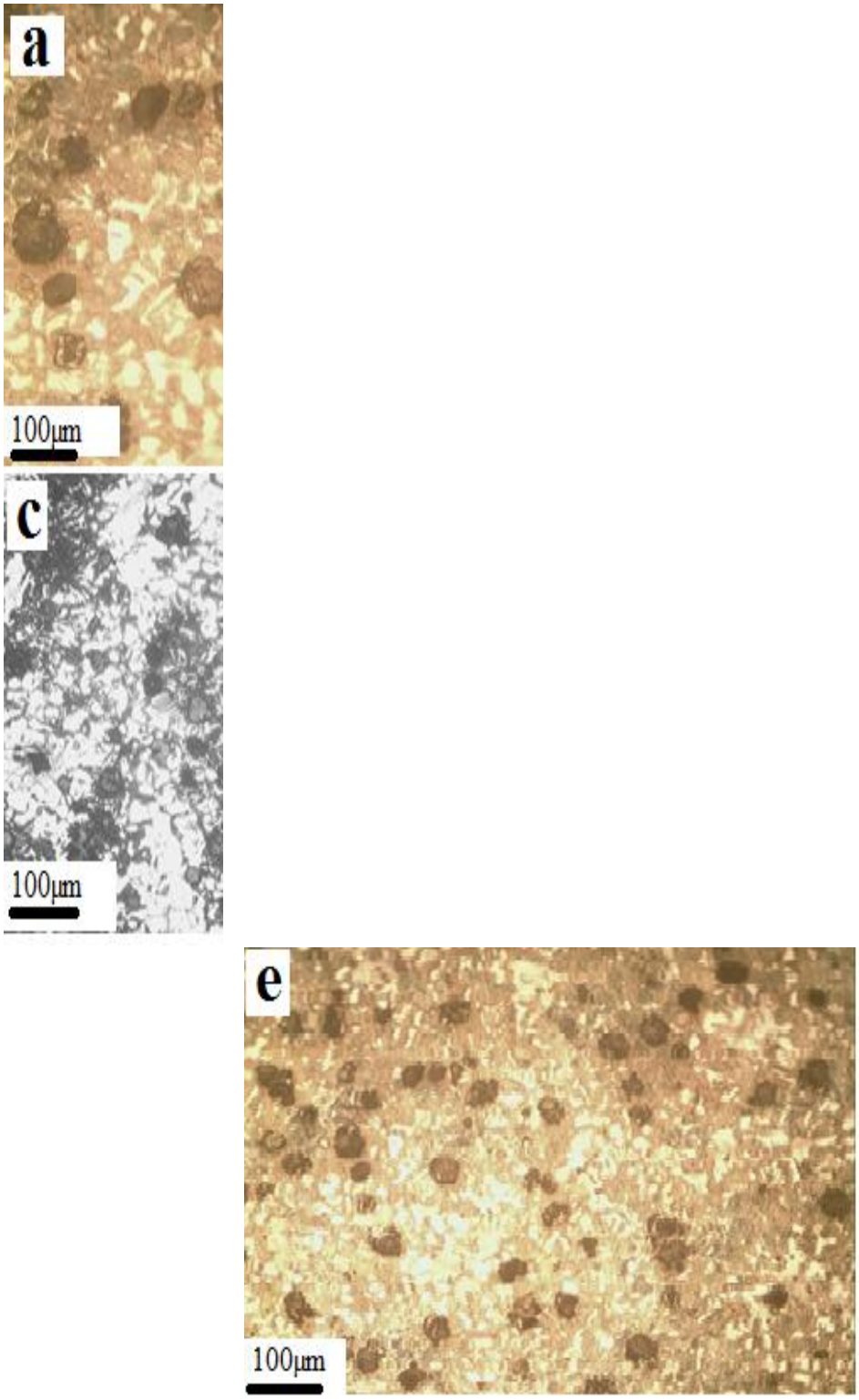


Plate 4: Optical micrograph of ductile cast iron austenitized at 850^{o}C, austempered at 250^{o}C in Sesame oil and etched in 2% Nital for (a) 1 hour, (b) 2 hours, (c) 3 hours, (d) 4 hours, (e) 5 hours. The structure consists of graphite nodules (dark balls) and retained austenite in ausferrite (x100).

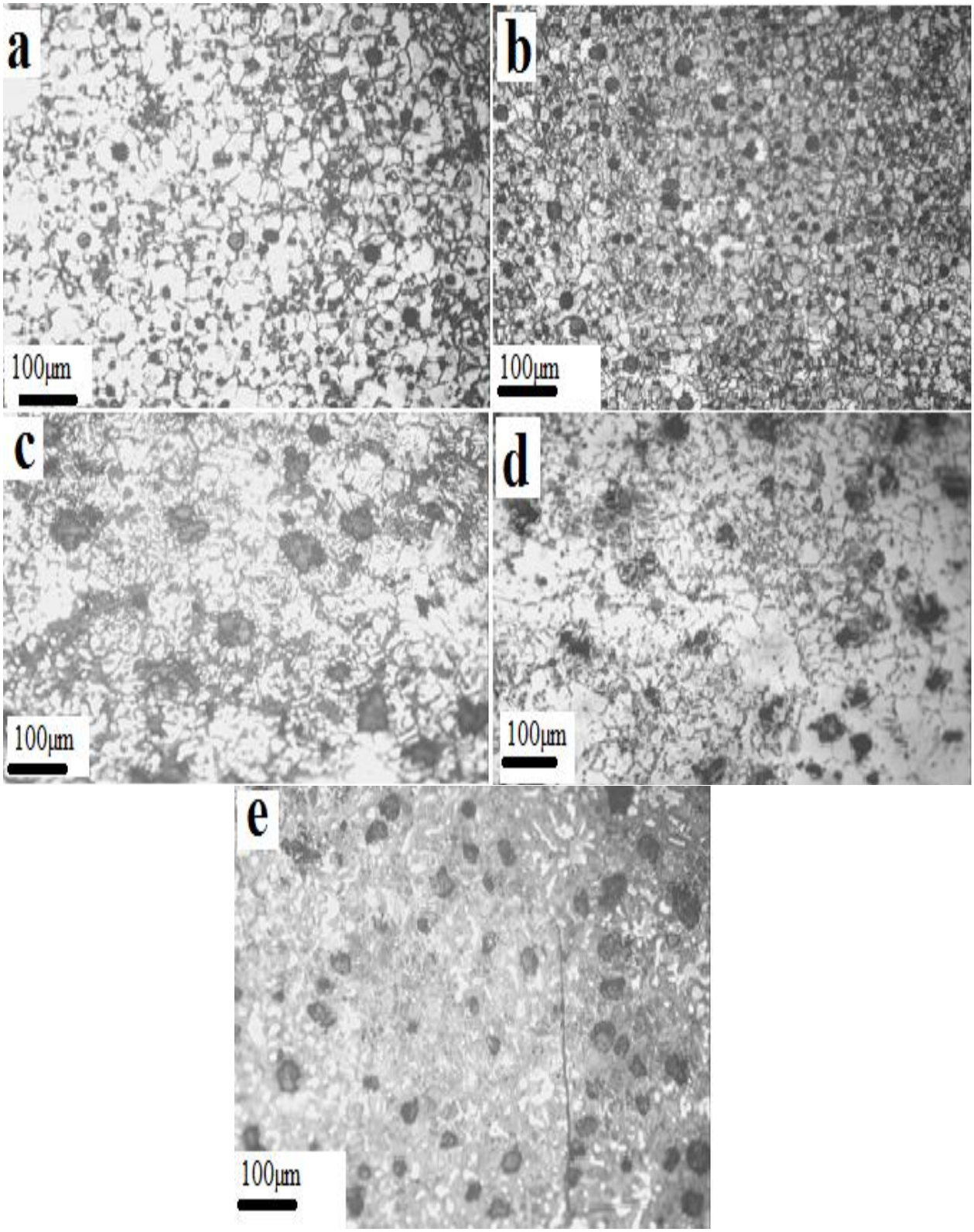


Plate 5: Optical micrograph of ductile cast iron austenitized at 850^{o}C, austempered at 300^{o}C in Sesame oil and etched in 2% Nital for (a) 1 hour, (b) 2 hours, (c) 3 hours, (d) 4 hours, (e) 5 hours. The structure consists of graphite nodules (dark balls) and retained austenite in ausferrite (x100).

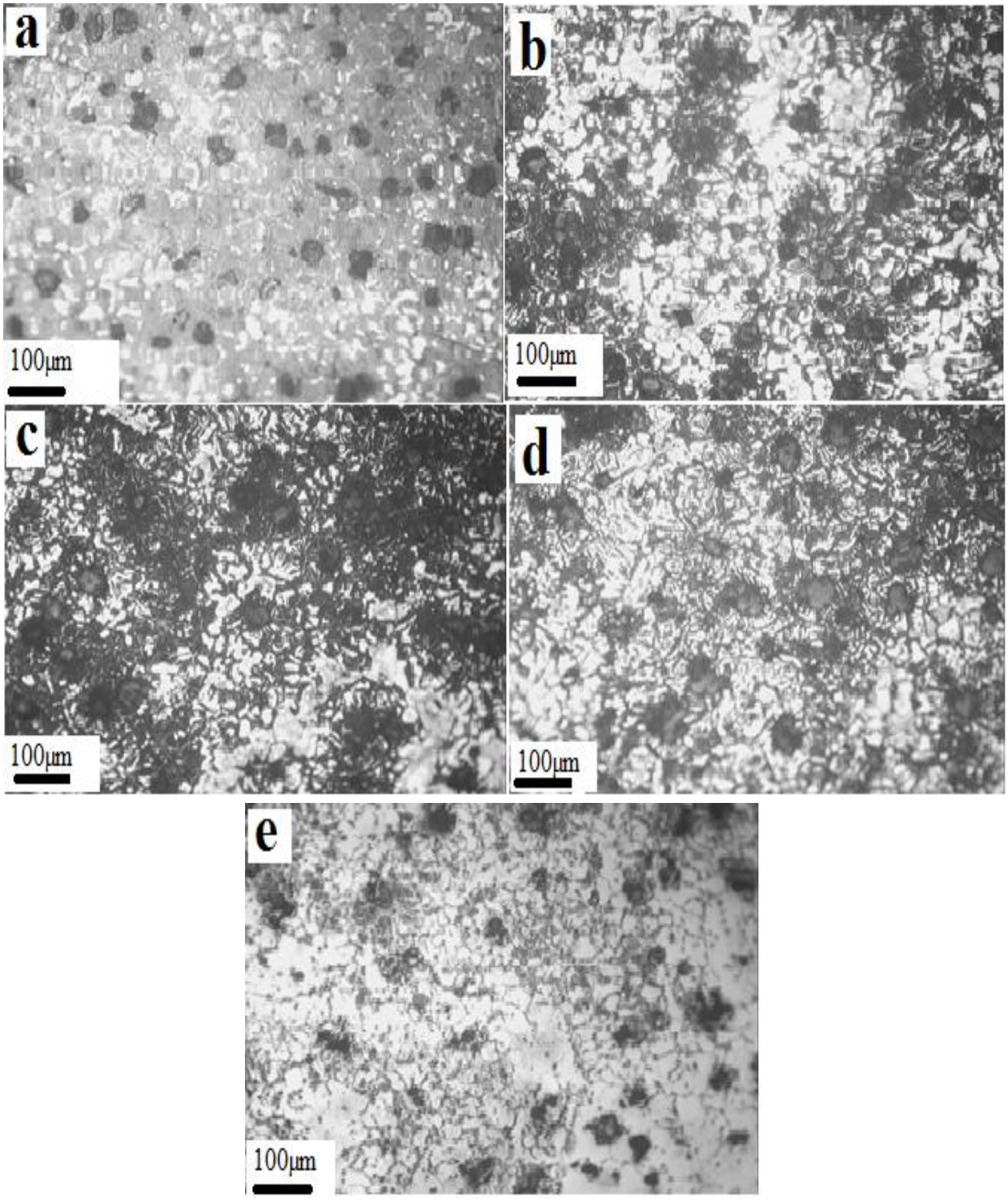


Plate 6: Optical micrograph of ductile cast iron austenitized at 900^{o}C, austempered at 250^{o}C in Sesame oil and etched in 2% Nital for (a) 1 hour, (b) 2 hours, (c) 3 hours, (d) 4 hours, (e) 5 hours. The structure consists of graphite nodules (dark balls) and retained austenite in ausferrite (x100)

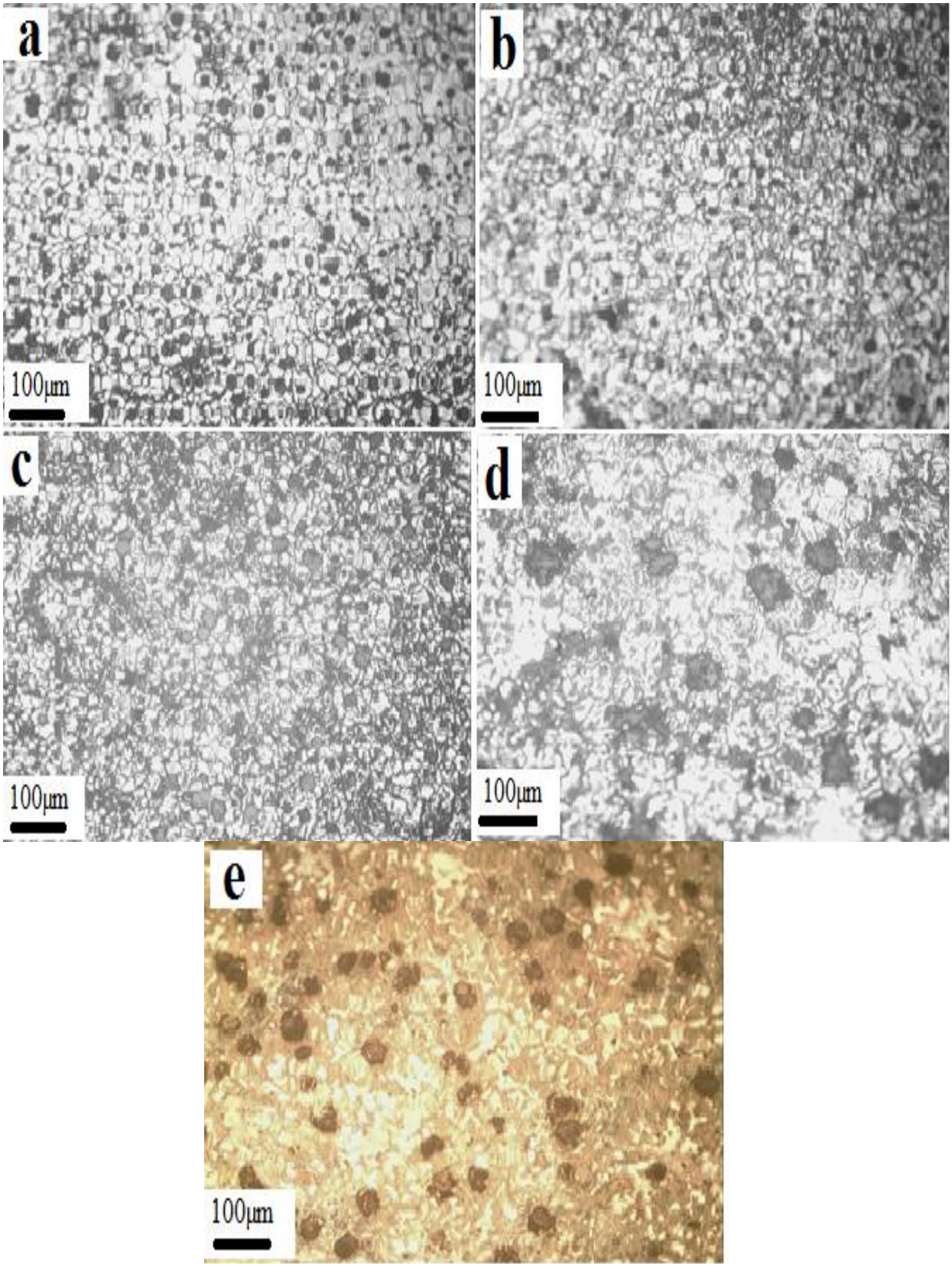


Plate 7: Optical micrograph of ductile cast iron austenitized at 900^{o}C, austempered at 300^{o}C in Sesame oil and etched in 2% Nital for (a) 1 hour, (b) 2 hours, (c) 3 hours, (d) 4 hours, (e) 5 hours. The structure consists of graphite nodules (dark balls) and retained austenite in ausferrite (x100).

The optical microstructures of the samples austempered in hot Sesame oil bath at 250^{o}C and 300^{o}C for 1-5hours from austenitizing temperatures of 850^{o}C and 900^{o}C are shown in Plates 4 to 7.As austempering time was increased progressively, ausferrite structure (Bainite) was formed. The structures are associated with retained austenite. This resulted from the early stage of austempering reaction. Complete ausferrite (bainite) structure was observed for austempered samples for 3hours. This is in agreement with what was reported by Ause (2008).

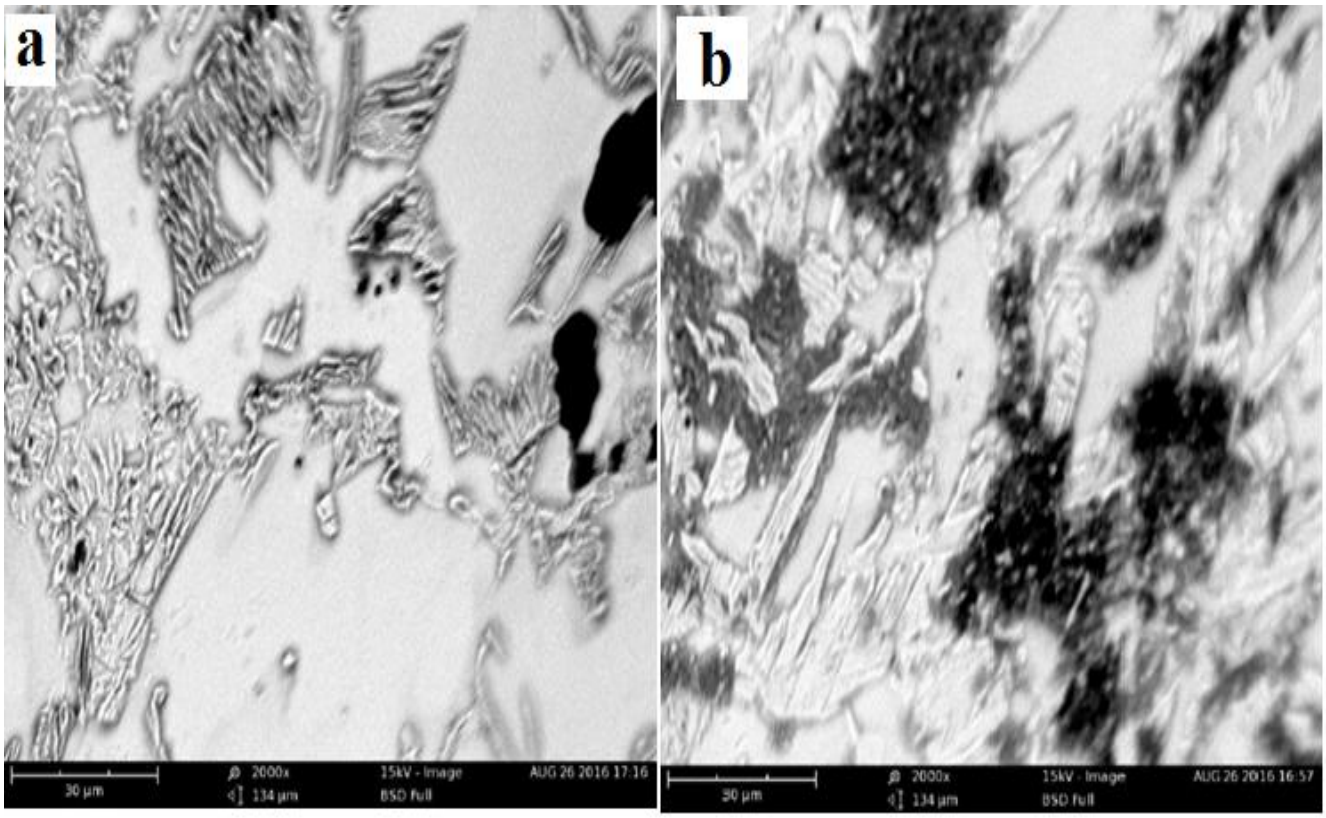


Plate 8: SEM images of ductile cast iron samples austenitized at 850^{o}C and austempered in Sesame oil at 250^{o}C for (a) 1hour, (b) 2hour. The structures show ausferrite structures (Bainite) and retained austenite.

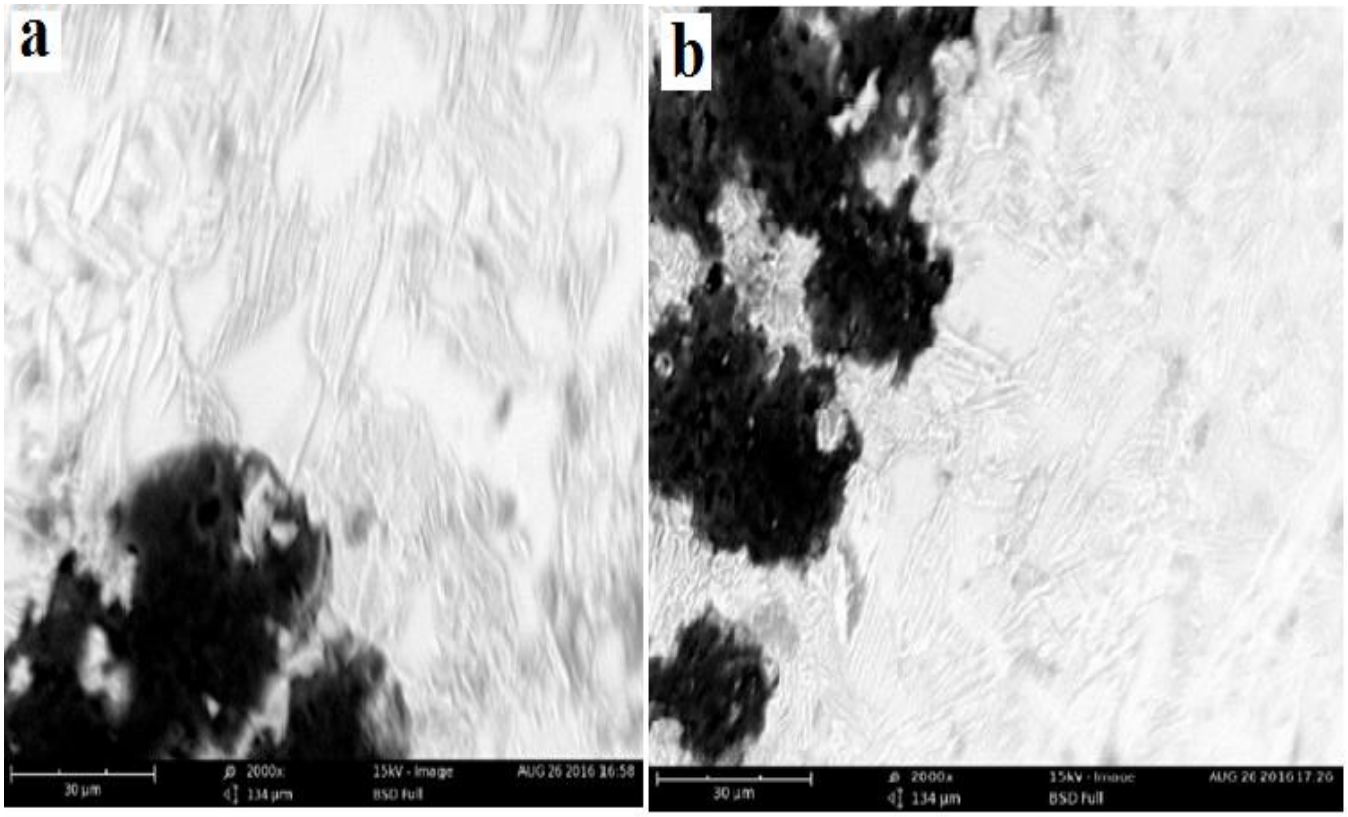


Plate 9: SEM images of ductile cast iron samples austenitized at 850^{o}C and austempered in Sesame oil at 250^{o}C for (a) 3hour, (b) 4hour. The structures show ausferrite structure (Bainite).

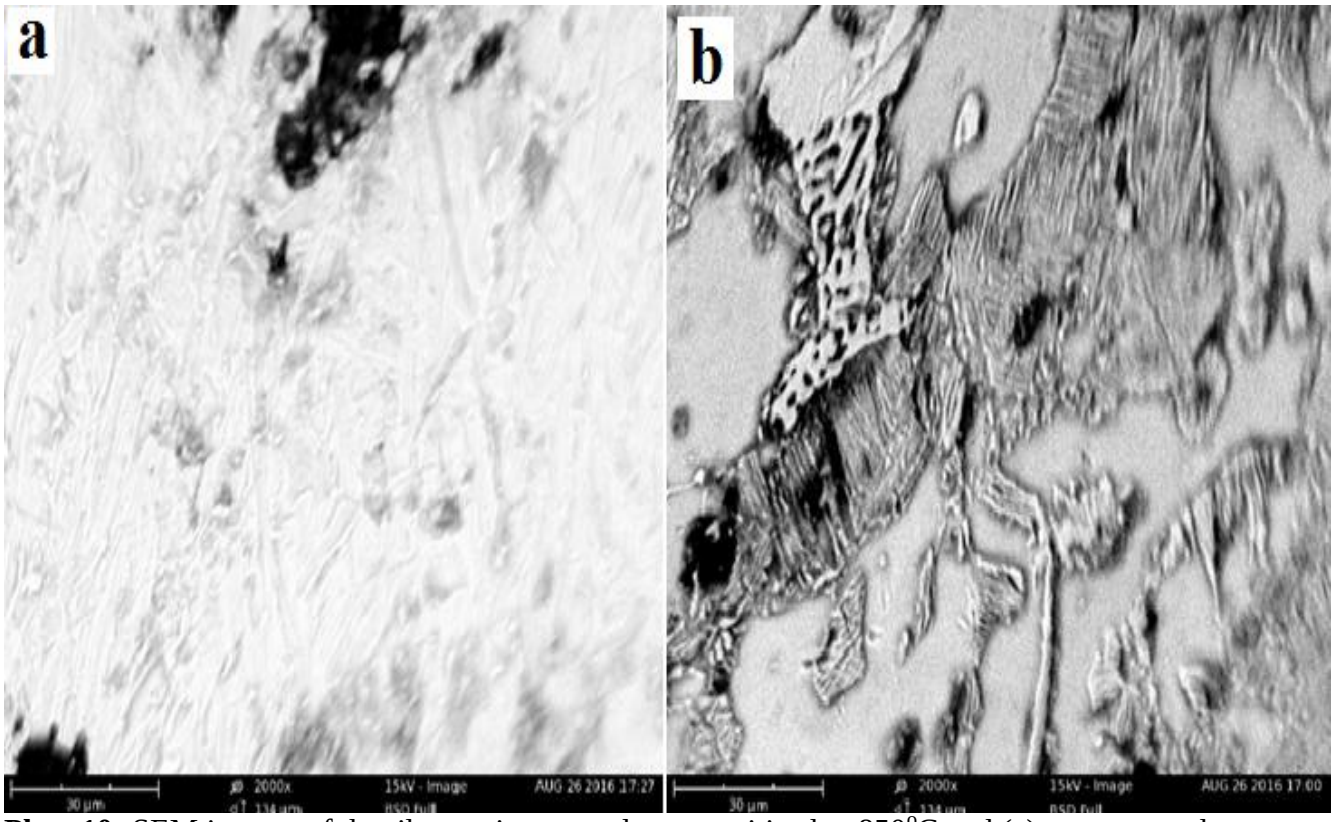


Plate 10: SEM images of ductile cast iron samples austenitized at 850^{o}C and (a) austempered in Sesame oil at 250^{o}C for 5hour, this structure shows bainite (b) austempered in Sesame oil at 300^{o}C for 1hour. The structures show ausferrite structures (Bainite) and retained austenite.

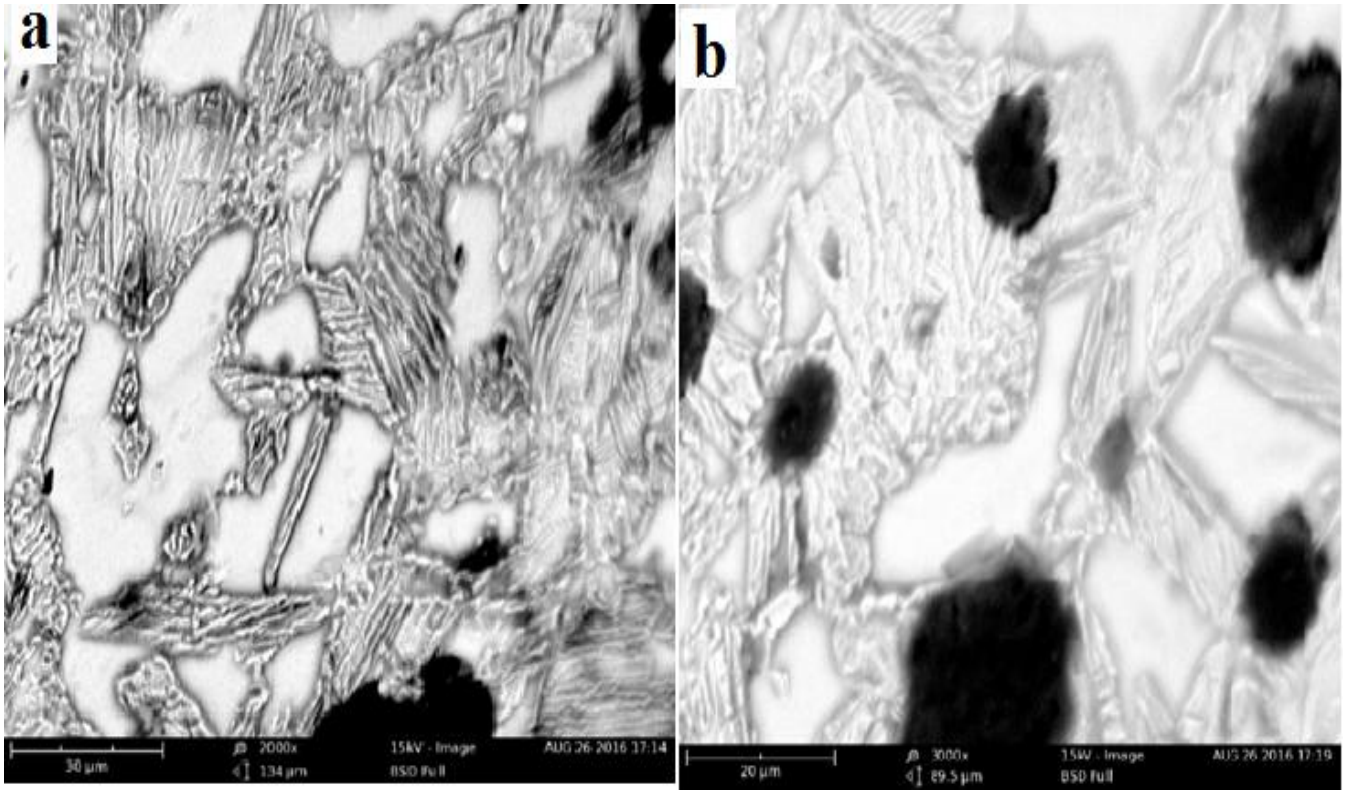


Plate 11:SEM images of ductile cast iron samples austenitized at 850^{o}C and austempered in Sesame oil at 300^{o}C for (a) 2hour, (b) 3hour. The structures show ausferrite structures (Bainite).

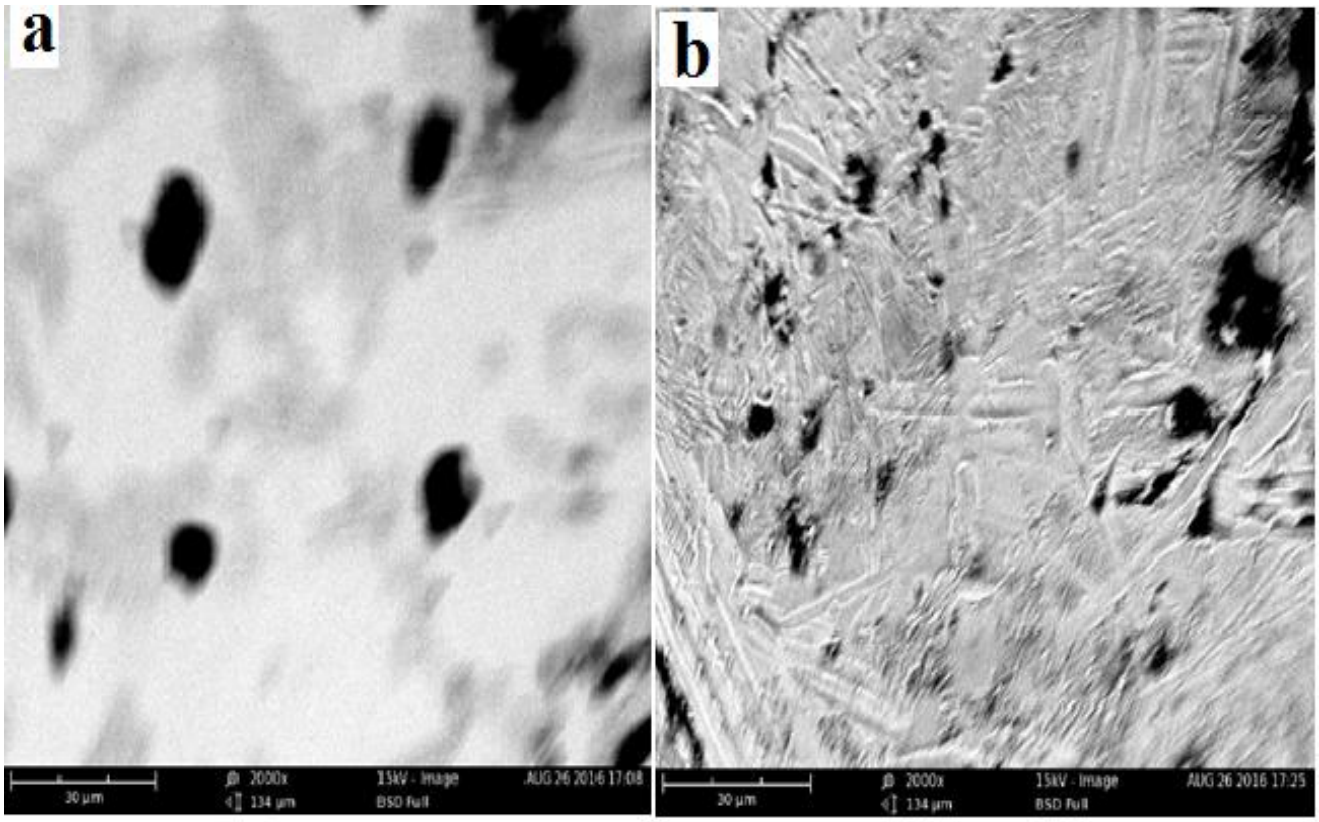


Plate 12: SEM image of ductile cast iron samples austenitized at 850^{o}C and austempered in Sesame oil at 300^{o}C for (a) 4hour, this structures show ausferrite structures (Bainite) (b) 5hour, this structure shows bainite.

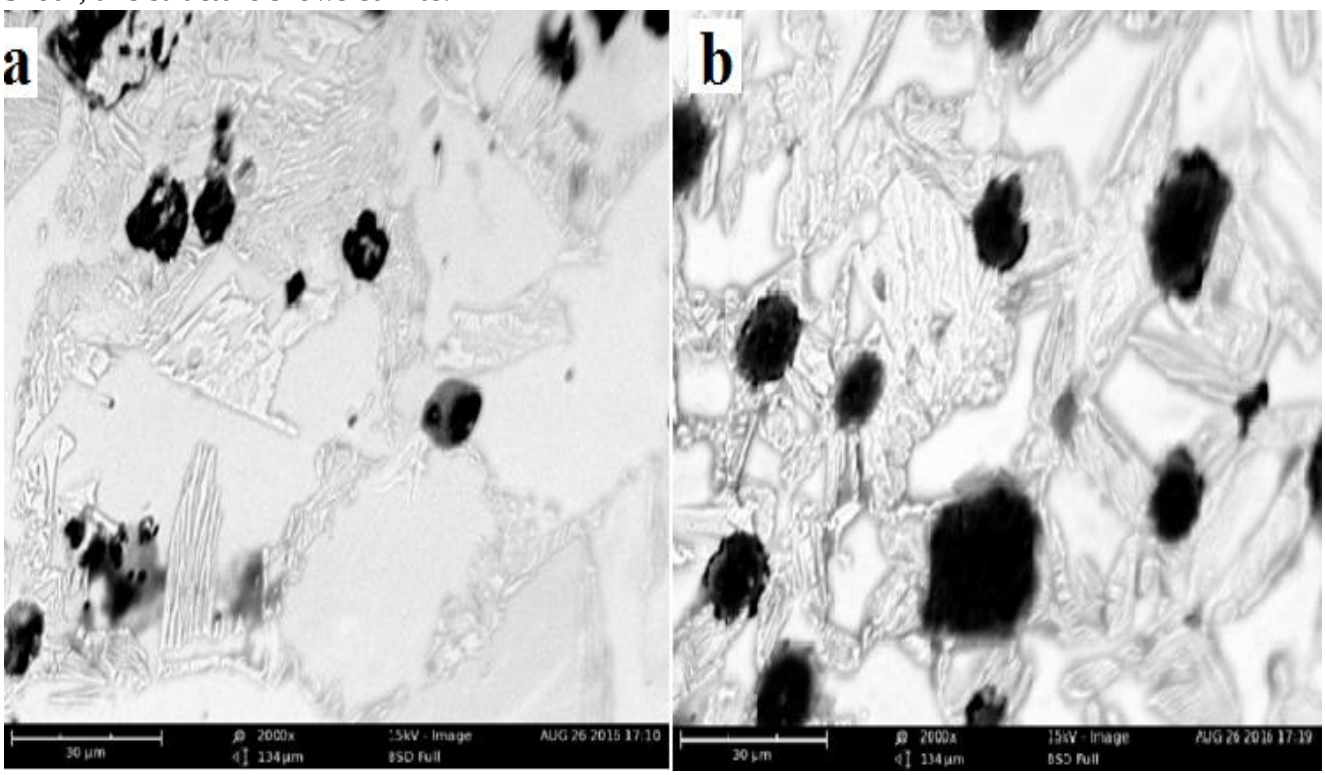


Plate 13: SEM image of ductile cast iron samples austenitized at 900^{o}C and austempered in Sesame oil at 250^{o}C for (a) 1hour; the structure shows ausferrite structure (Bainite) and retained austenite (b) 2hour; the structures show ausferrite structures (Bainite).

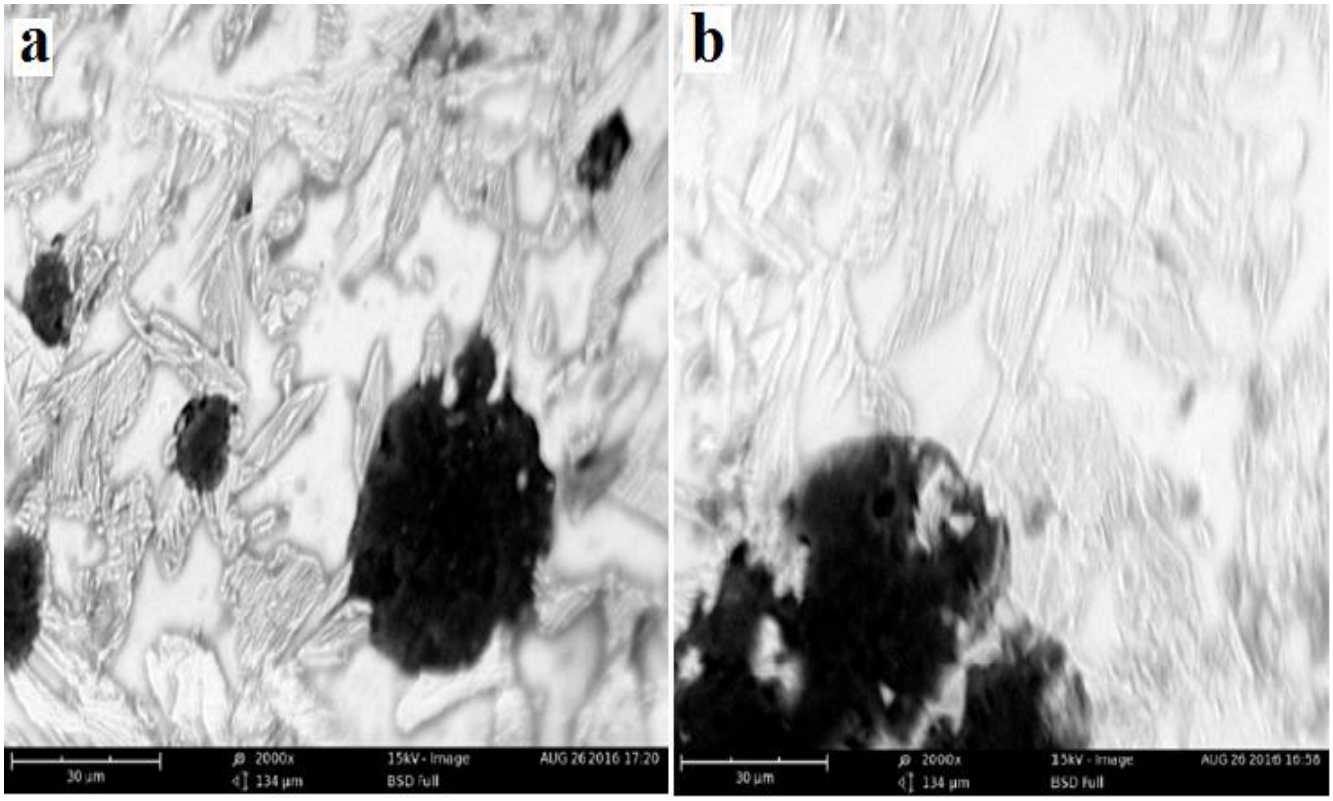


Plate 14:: SEM images of ductile cast iron samples austenitized at 900^{o}C and austempered in Sesame oil at 250^{o}C for (a) 3hour, (b) 4hour. The structures show ausferrite structures (Bainite).

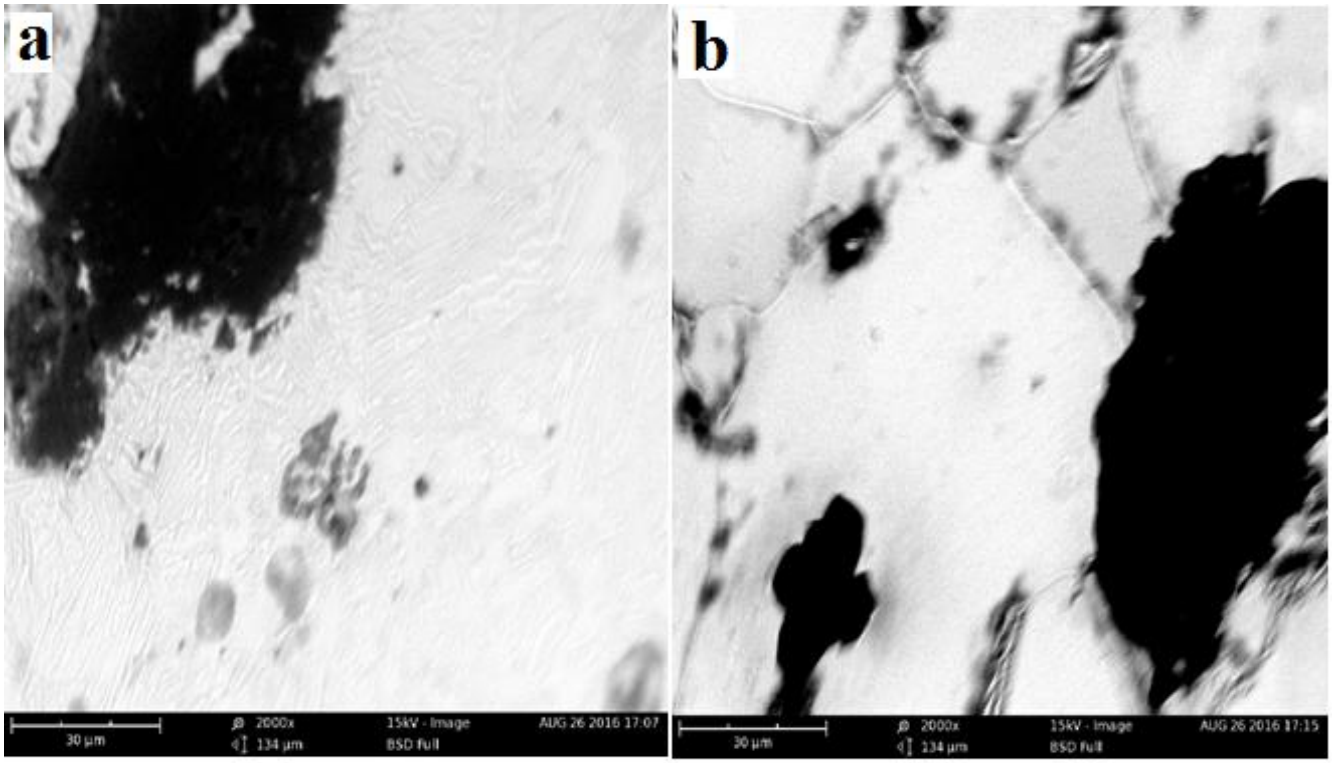


Plate 15: SEM images of ductile cast iron samples austenitized at 900^{o}C and (a) austempered in Sesame oil at 250^{o}C for 5hour, this structure shows bainite (b) austempered in Sesame oil at 300^{o}C for 1hour the structure shows ausferrite structures (Bainite) and retained austenite.

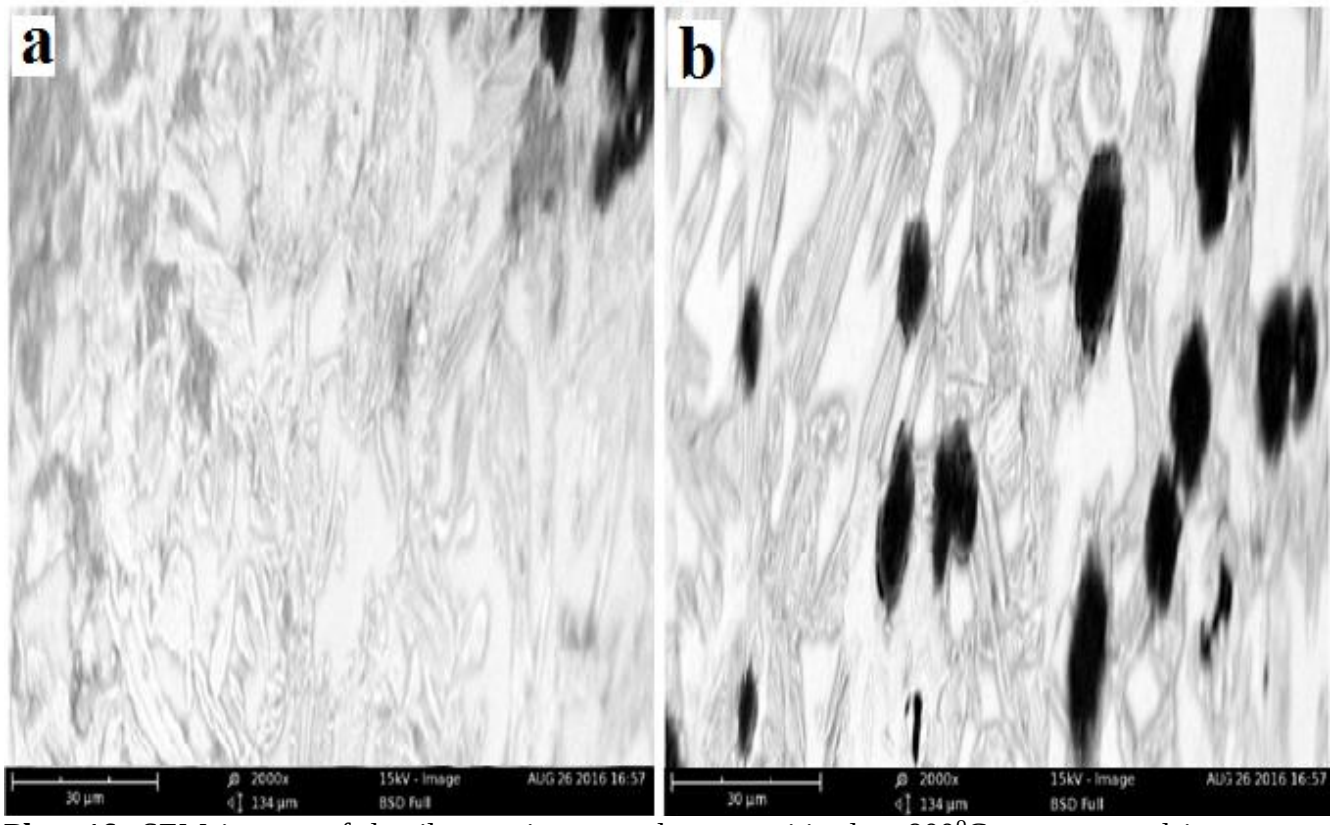


Plate 16: SEM images of ductile cast iron samples austenitized at 900^{o}C, austempered in Sesame oil at 300^{o}C (a) for 2hours; the structure shows ausferrite structures (Bainite) plus retained austenite (b) for 3hours; the structure shows ausferrite structures (Bainite).

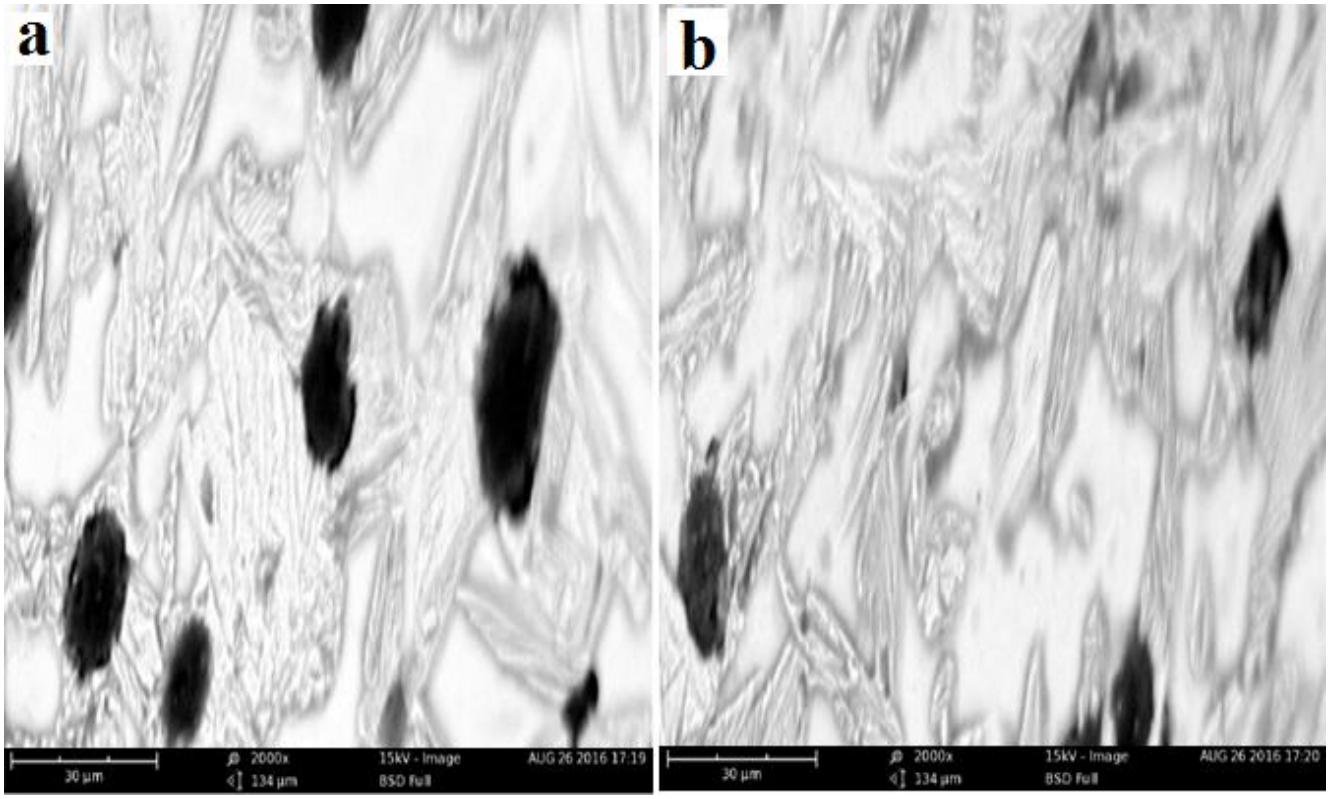


Plate 17: SEM images of ductile cast iron samples austenitized at 900^{o}C, austempered in Sesame oil at 300^{o}C (a) for 4hours (b) for 5hours; the structures show ausferrite structures (Bainite).

The SEM images (Plates 8 to 17) show that the proportion of ausferrite structures (Bainite) increase with increase in austempering time. This could be attributed to the progressive setting in of stage I austempering reaction. This trend clearly explained the high strength and toughness obtained for samples austempered up to 3 hours. However, samples austempered at 4 and 5 hours proved to show relatively coarser ausferrite lath with fine precipitates of brittle carbides. The brittle carbide formed as a result of second stage austempering reaction. This caused the inferior mechanical properties in samples austempered at 4 and 5 hours. These SEM images are similar to those obtained from samples austempered in molten salt bath by Tun and Lwin (2009).

4.5 Fourier Transform Infrared Spectroscopy (FTIR)

The Fourier transform infrared spectroscopy (FTIR) of the Sesame oil before and after heat treatment are shown in Figure 4.13 and 4.14 below:

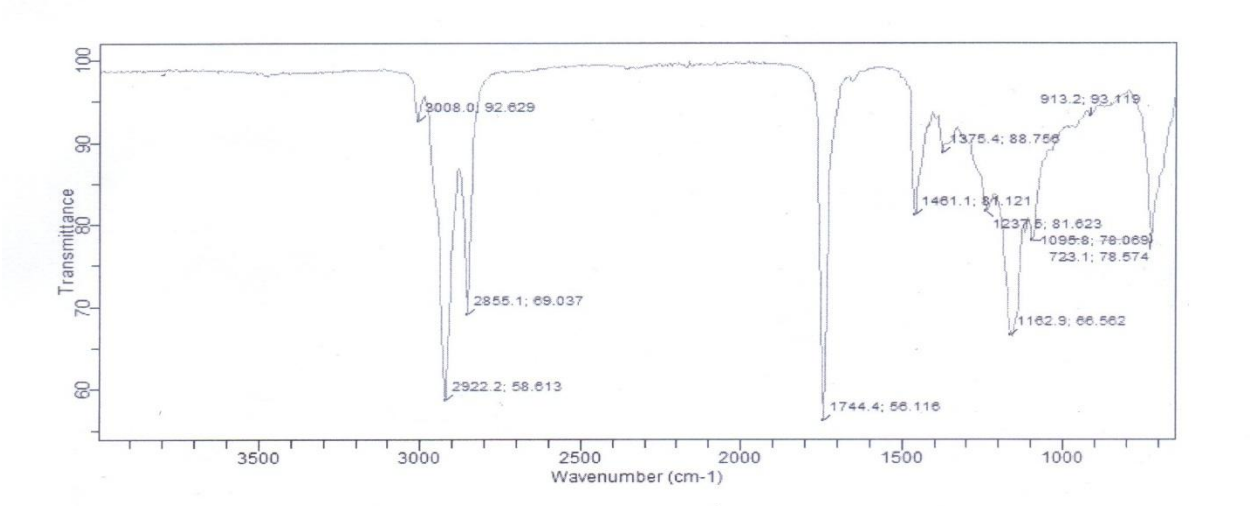


Figure 4.13: FTIR of the Sesame oil before heat treatment.

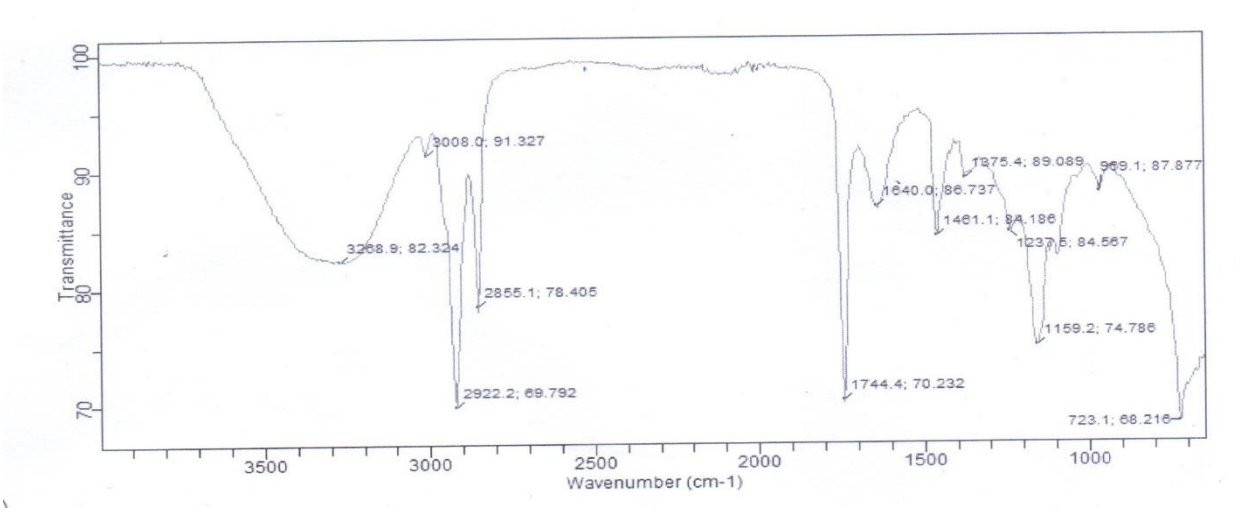


Figure 4.14: FTIR of the Sesame oil after austempering heat treatment

Figures 4.13 and 4.14 showFourier transform infrared spectroscopy (FTIR) of the Sesame oil before and after austempering heat treatment respectively. It was observed that some peaks (functional groups/radicals) werere-configured after the austempering heat treatment .This is attributed to the fact that some peaks 1095.8, 1162.9, 1237.5 and 1461.1cm^{-1} representing functional groups and indicating the class of compounds (Ethers, Esters and Alkyls) before heat treatment changed to 1159.2, 1237.5, 1375.4, 1461.1 and 3268.9 cm^{-1} indicating theclass

of compounds (Alcohol, Alkanes, Amides and phenol)may be responsible for the result obtained.

Table 4.2: Characteristic infrared absorption bands of functional groups for Sesame oil beforeaustempering heat treatment

S/N	Peaks (Absorption) (cm^{-1})	Intensity	Functional group and type of vibration/Class of compound
1	723.1	W	$-(CH)_n$ bend
2	913.2	m+s	=C-H bend
3	1095.8	Vs s	C-F stretch Alkyl halides C-O-C stretch Ethers
4	1162.9	Vs, s-vs	C-F stretchAlkyl halides O=C-O-C stretch Esters
5	1237.5	m-s	=C-O-C sym. & asym. Stretch Ethers C-O stretch
6	1375.4	m	CH_3-CH bend
7	1461.1	S m-s	C-H bend Alkanes Ring C=C stretch Alkyls
8	1744.4	S Ss	C=O stretch C=O sym. & asym. stretch
9	2855.1	S s, broad	C-H stretch O-H stretch
10	2922.2	S S, broad	C-H stretch O-H stretch
11	3008.0	S, broad	O-H stretch

Table 4.3: Characteristic infrared absorption bands of functional groups for Sesame oil after austempering heat treatment

S/N	Peaks (Absorption) (cm^{-1})	Intensity	Functional group (Assignment)
1	723.1	W	$-(CH)_n$ bend
2	969.1	S m+s	=C-H bend =C-H bend
3	1159.2	Vs m-s	C-F stretch Alkyl halides C-O stretch Alcohols
4	1237.5	m-s m-s	C-O stretch Alcohol =C-O-C sym. asym. Stretch Ethers
5	1375.4	m	CH_3C-CH bend Alkanes
6	1461.1	S m-s	C-H bend Alkanes ring C=C stretch Alkyls
7	1640.0	m-s w-m s-broad vw-m s, broad	N-H bend N-H bend C=O stretch Amides C=O stretch ΞC-H bend
8	1744.4	S s Ss	C=O stretch C=O stretch C=O sym. & asym. stretch
9	2855.1	S s, broad	C-H stretch O-H stretch
10	2922.2	S S, broad	C-H stretch O-H stretch
11	3008.0	S, broad	O-H stretch
12	3268.9	S, sharp w-m	ΞC-H stretch Alkynes N-H sym. asym. Stretch Amides OH stretch Phenol

CHAPTER FIVE

CONCLUSIONS AND RECOMMNDATIONS

5.1 Conclusions

Based on the results of this study, the following conclusions can be drawn:

- The smoke, boiling and flash points of the oil were found to be 223^{o}C, 304^{o}C and 391^{o}C respectively.
- Austempering in hot sesame oil bath gave microstructures that consist of ausferrite structure (Bainite) and retained austenite.
- As the holding time for austempering increase, the tensile strength, impact strength and percentage elongation initially increaseprogressively to a maximum at 3hoursand then slightly decreased at 4 and 5hours.
- At 3 hours of austempering time, a full austempered microstructure (a mixture of fine acicular ferrite and high carbon stabilized austenite)were found adequate to develop the necessary enhance mechanical properties in ductile cast iron samples in hot Sesame oil bath.
- The hardness of ductile cast iron samples increase progressively as the austenitizing temperature increase from 850^{o}C to 900^{o}C.

5.2 Recommendations

Based on the study conducted, the following recommendation can be offered:

- The suitability of Sesame oil for austempering of Gray cast iron, medium carbon steel, high carbon steel and low alloy steel should be investigated.
- Researchers should be encouraged to use local raw materials as quenching media in heat treatment processes to conserve the limited foreign exchange due to importation of salt bath and its associated consumables.

- Since the sesame oil investigated has been able to produce bainite structure, and considering the fact that it is cheap and environmentally friendly compared to molten salt and lead bath, therefore should be added to the list of existing austempering quenching media

5.3 Contribution to Knowledge

From the study carried out the, following can be established:

- Austempering in Sesame oil was able to cause the formation of ausferrite (Bainite) in the component in the same way as molten salt and lead bath. Hence, Sesame oil could be a good alternative to molten salt and lead bath for use as an austempering quenching media.
- Considering the fact that sesame oil is cheap and environmentally friendly compared to molten salt and lead bath, therefore should be added to the list of existing austempering quenching media.

REFERENCES

Ademoh, N.A. (2015). Effect of Nigerian Neem Seed oil as an austempering quenchant for Locally recycled steel. *Middle-East Journal of scientific research, 23(1), 110-118.*

Adewuyi, B.O., and Afonja, A.A. (2000). Austempering ductile Iron – a viable alternative to steel. *Nigerian journal of engineering management,* 1(1): 6-13.

Aigbodion, V.S., Yaro, S.A., and Abdulrahman, A.S. (2005). Evaluated the use of engine oil as austempering medium for the austempering of ductile iron for the production of agricultural machines and farm implements. *Journal of science and industrial studies,*
3(4): 51-54.

Akor, T., and Tuleum, L.T. (2014). Effect of austempering time on the mechanical properties of ductile iron, austempered in Rubber seed oil. *International journal of engineering Research and development, 10(8), 31-34.*

Alan, v. (2012). Microstructure and mechanical properties of austempered ductile iron. *International journal of engineering, Annals of faculty engineering Hunedoara. (1):* 1584-2665.

Alexey, V. S., and Arnord, R.N. (2006).*Fundamental Concepts in steel Heat Treatment, Boca Raton,* Florida: (2nd Ed), Taylor and Francis Group LLC.

Almaida, G.G., and Hasan, A. (2009). Heat treatment of ductile cast iron. *13th international research /expert conference. Trends in the development of machinery and associated technology, Hummamet, Tunisia.*

Alp, T., Wazzan, A. A., and Yilmaz, F. (2005). Microstructure property relationship in cast iron. *The Arabian journal of science and engineering.* 2(B): 163-175.

Amar, K.D., Jtoti, P.D., Ranjit, K.P., Mishra, S.C., and Sen, S. (2013). Effect of alloy in Elements and processing parameters on mechanical properties of austempered ductile iron. *Journal of materials and metallurgical engineering, 3(1), 2231-3818.*

Ambooken, M.T. (2006). *The influence of magnetic austempering on microstructure and mech-*
anical properties of austempered ductile cast iron (ADI). Thesis, department of mate-
rials science and engineering, Wayne state University Detrioy, Michigam.

American Society of Metal (ASM), (2000*). Handbook,* 5th Edition Pp 26-56.

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

American Society of Metal (ASM), (1978*). Handbook,* pp27-56

ASTM (2007). "*Standard Test Method and definition for mechanical testing*".

Ause, T. (2008*). Evaluation of Hot Bitumen Bath as a Quenching Medium forAustempering of Steel and Ductile Cast Iron,*unpublished (PhD Thesis, Department of Metallurgical and Material Engineering, Zaria: Ahmadu Bello University).

Balogun, S.O. (2008). *Potential of Cassava Liquid Extract as Quenching Medium for medium Carbon and Low alloy Steels* (M.Sc Thesis, Department of Metallurgical and Materials Engineering, Zaria: Ahmadu Bello University).

Bisht, A. (2009*). Effect of heat treatment procedures on microstructure and mechanical properties of nodular iron.* (MSc. Thesis, Department of Metallurgical and Materials Engineering, Rourkela, University India.

Borchani, C., Besbes, S., Blecker, Ch., and Attia, H. (2010*). Chemical Characteristics and Oxidative Stability of Sesame Seed, Sesame Paste and Olive oil: J. Agr. Sci. Tec 12, 585-596.*

Branka, B., and Branko, R. (2004). Effect of Alloying Elements and Austempering temperature on Austempering Kinetics of Ni-Mo alloy Ductile iron. *Material in technology*, 38(6): 307-312.

Dauda, B.Y. (2005). *Effect of Austempering in Boiling Bitumen on Microstructure of Ductile Iron* A.B.U. press Ltd Pp 3-30

Dodo, R.M. (2015). *Potential of Cane Molasses as Quenching Medium for (0.61% C) High Carbon Steel.* (MSc Dissertation, Department of Metallurgical and Material Engineering, Zaria: Ahmadu Bello University).

Dommarco, R.C., Jaureguiberry, A.J., and Sikora, J.A. (2008). Rolling contact fatigue resistance of ductile iron with different nodule counts and matrix microstructure, 261(2): 172-179.

Elliott,R., and Yazdani, S. (1999). Influence of molybdenum on austempering behavior of ductile iron. Part 1-Austempering kinetics of ductile iron containing 0.13%Mo. *Journal of Materials Science and Technology, 15: 233 - 240.*

Eric, U., Javanovic, M., Sidjanin, L., and Rajnovic, D. (2004). Microstructure and mechanical properties of Cu, Ni, Mo Austempered ductile iron. *Journal of mining and metallurgy,* 40B (1): 11-19.

Fariku, S., Ndonya, A.E., and Bitrus, P.Y. (2007). Biofuel characteristics of beniseed (Sesanum Indicum) oil*. African Journal of Biotechnology 6(21), 2442-2443.*

Fracasso, F. (2010). *Influence of Quench Rate on the Hardness Obtained after Artificial Ageingof Al-Si-Mg Alloy,* A thesis work perform at Jonkoping institute of Technology

Tekniska Hogskolan.

Hamza, Z.U., and Mohammed, M.I. (2008). Physico-Chemical Properties of oil Extract from Sesanum indicum L. seed grown in Jigawa state- Nigeria. *Journal of apply Science Environ. Manage 12(2), 99-101.*

Hassan, S. B., Agboola, J.B., Agbodium, V.S., and Williams, E.J (2010).Hardening Characteris
tics of plain Carbon Steels and Ductile Cast Iron using Neem oil as Quenchant. *Journal* of *Metallurgical and Materials Engineering, 5(1), 31-36.*

.

Hassan, S.B. (2009). "*Advance Physical Metallurgy*: (M.Sc lecture note, Department of Metallurgical and Materials Engineering, Zaria: Ahmadu Bello University).

Hassan, S.B., Dauda, E.T., Nyior, G.B., and Mohammed, R.A. (2007). Evaluation of local Vegetable oils as quenchants for hardening process in grey cast iron. *Journal of applied*
Science, Engineering and technology, 7(2): 13-21.

Hasirci, H., and Erdogan, M. (2000). Effect of alloying elements and austempering time on microstructure and mechanical properties in austempered spheroid graphite cast iron. *Journal of science, Ankara* 13(4):32-34.

Henkel, and Pense (2002*). Structure and properties of engineering Materials*. 5th edition Mc Graw- Hill publishers, America. Pp 150-358.

Higgins R.A. (1995): Properties of Engineering Materials.2nd Ed. Edward Arnold Ltd. London UK. pp. 186 - 284.

Isah, L.A. and S.B. Hassan. 2008. "Suitability of Khaya Senegalensis Seed Oil as a Quenchant
in Austempering Heat Treatment of Ductile Iron". *Journal of Metallurgy and Material Engineering*. 4(2):38-43.

Kathy, L.H. (2002). *Production of Austempering Ductile Iron.* World conference on ADI, Applied process technologies division, Livonia.

Kalpakjan, s. (1989). Manufacturing Engineering and Technology. *International Journal of production research*, 28(2): 32-37.

Kazerooni, R., Nazarboland, N., and Elliott, R. (1997). Use of Austenitizing Temperature in Control of Austempering of an Mn – Mo – Cu Alloyed Ductile iron. *Journal of Materials Science and Technology,* Vol. 13; p1007 – 1015.

Kempster, M. H. A. (1979). *Material for Engineers*. Biddles ltd, Guildford and King Lynn Publisher. Britain. Pp 48 -186.

Khanna, O.P. (2002*). Material Science and Metallurgy*. Dhan Pat Rai Publication Ltd Pp 60-70.

Khanna, O.P. (2010*). Material Science and Metallurgy*. Dhan Pat Rai Publication Ltd.

Lauralice, C.F.C., Maro, R.F., Sylvana, C.M.A., George, E.T., and Alessandro, F.F. (2005). Oxidation of vegetable oils and its impact on quenching performance. *International journal of materials and product technology,* 12(1-4): 101-125.

Lawrence, I.A. (2011). *Evaluation of Khaya Senegalensis Seed Oil as a Quenching medium in Austempering process of Ductile Iron.*(PhD Thesis, Department of Metallurgical and Material Engineering, Zaria: Ahmadu Bello University).

Lee, S.M., Park, S.S., and Moon, B.M. (2003). High Si and Mn Containing Austempered Ductile Iron for Wear Application. *Journal of Korea institute of industrial technology, Incheon, Korea.* 3(1): 407-419.

Nelson, J.A. (1985). Cast irons, Metallography and Microstructures, American Society for Metals ASM *Handbook,* Vol.9.

Rajan, T.V., Sharma, C.P., and Sharma, A. (1988). *Heat Treatment: Principles and Techniques* .Prentice Hall of India, Private Limited, New Delhi.pp51-384.

Raymond A. Higgins. (1970*). Engineering metallurgy*. 2nd Edition, Edward Arnold Publisher. Pp 354-365.

Rundman, K.B. (1990) .*Metal casting, quickest least expensive route to a near shape product*.
Michigan tech university.

Sandeep, K.S. (2012*). Effect of austempering temperature, time and copper addition on Mechanical Properties of austempered ductile iron.* (Thesis, Department of Metallurgical and Materials Engineering, National Institute of Technology, Rourkela). Roll NO.28MM 102.

Sani, S.A. (2008*). Potentials of using some vegetable oils as quenchants for austempering of steel and cast irons* (PhD Thesis, Department of Metallurgical and Materials Engineering, Zaria: Ahmadu Bello University.

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

Smith, W.F. (1993). *Structure and properties of engineering alloy.* 2nd New York NY: Mc Graw-Hill.

Smallman, R. E., and Bishop, R. J. (1999*). Modern physical Metallurgy and Materials Engineering.* Oxford, Butterworth-Heinemann.

Totten, G.E.(2006). *Steel Heat Treating Metallurgy and Technologies,* Oregon, USA: Taylor and Francis Group LLC.

Tun, T., and Lwin, K. (2009). Optimizing the Microstructure and Mechanical properties of Austempered Ductile iron. *Journal of metals, Materials and Minerals, 18(2) 34-39*

Vivek,T., Adarsh P., Zuber M., and Chandrashekhar I.B. (2014) Prediction of quench severity of various quench media based on hardness and microstructure studies.*International Journal of Innovative Research in Advanced Engineering (IJIRAE) (1)* (3).

Wilson, T. (1975). *Metallurgy and heat treatment of tool steels.* 1st edition McGraw Hill Book Company, London. p34 – 94. *199- 205.*

Yang, J., and Susil, K. P. (2004*). Influence of a novel two-step austempering process on the strain-Hardening behavior of austempered ductile cast iron (ADI)*. Material Science and Engineering, A 382.

Yoon, J.K., Hocheol, s., Hyounson, P. and Jong, D.L. (2007). Investication into mechsnical properties of austempered ductile cast iron in accordance with austempering temperature. *Journal of materials letters, 62(2008): 357-360.*

APPENDIX A

Microstructures of austempered ductile cast irons obtained by other researchers.

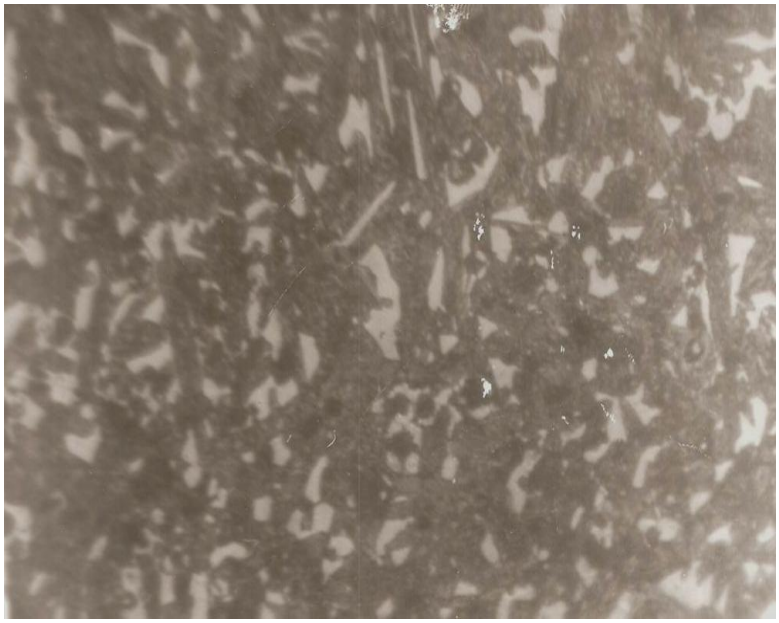


Plate B1: Microstructure of austempered nodular cast iron in hot groundnut oil at 250^{0}C for 4hrs, austenitized at 900^{o}C. The structure shows graphite nodules (black balls) in ausferrite and martensite (dark) matrix etched in 2% nital (x200).
(*Source:* Sani, 2008).

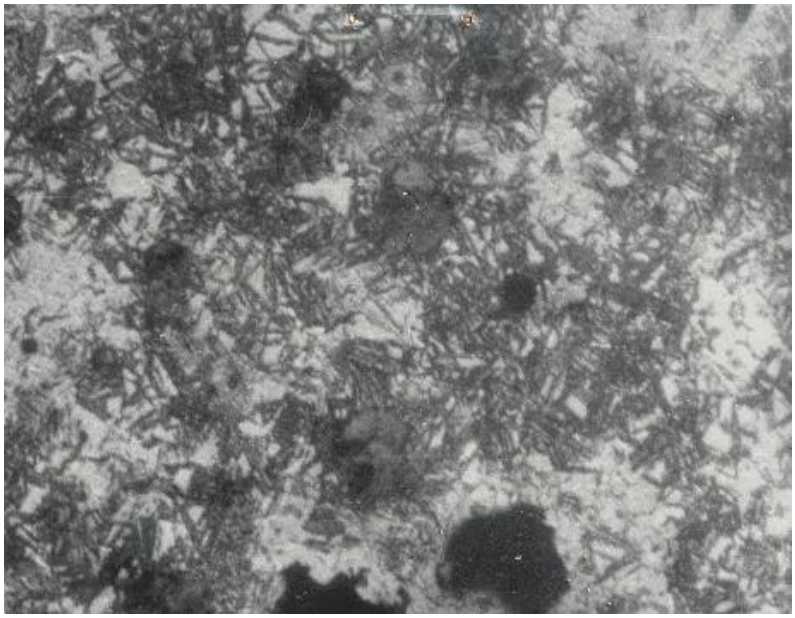


Plate B2: Microstructure of austempered nodular cast iron in hot bitumen at 350^{o}C for 3hrs, austenitized at 900^{o}C. The structure shows ausferrite (bainite); etched in 2% nital (x100), (*Source:* Ause, 2008).

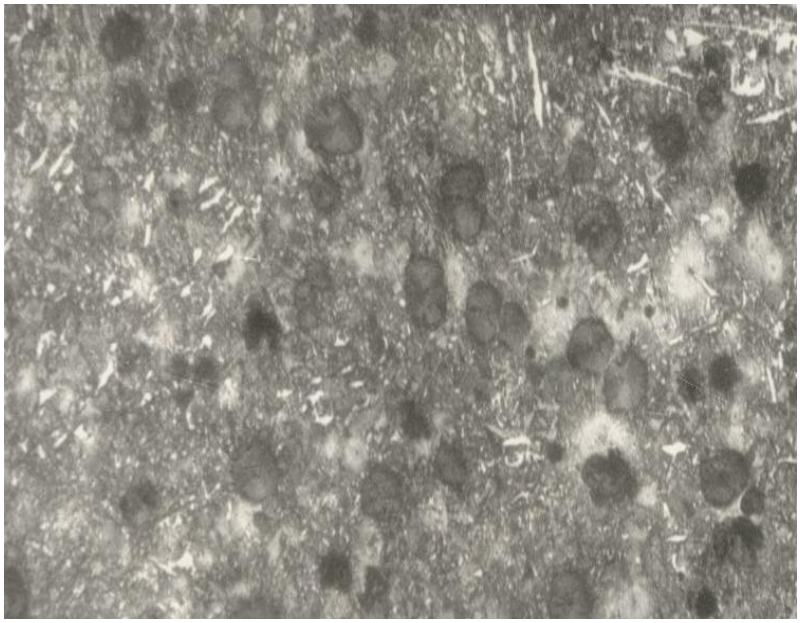


Plate B3: Microstructure of austempered nodular cast iron in hot bitumen at 350^{0}C for 5hrs, austenitized at 900^{o}C. The structure shows ausferrite (bainite) plus cementite; etched in 2% nital (x100), (*Source:* Ause, 2008).

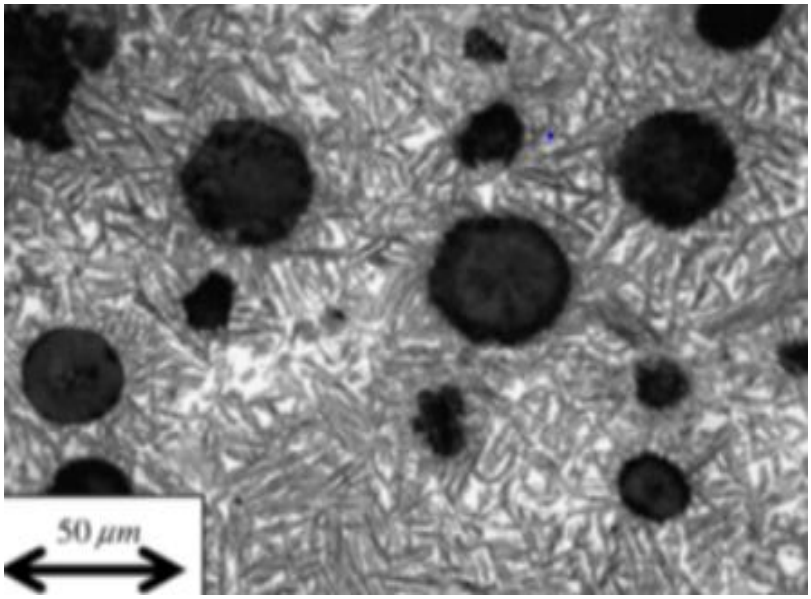


Plate B4: Microstructure of austempered ductile cast iron in molten salt bath at 350^{O}C for 90mints, austenitized at 910^{o}C. The structure shows ausferrite (bainite);
(*Source:*Yoon *et al.*, 2007)

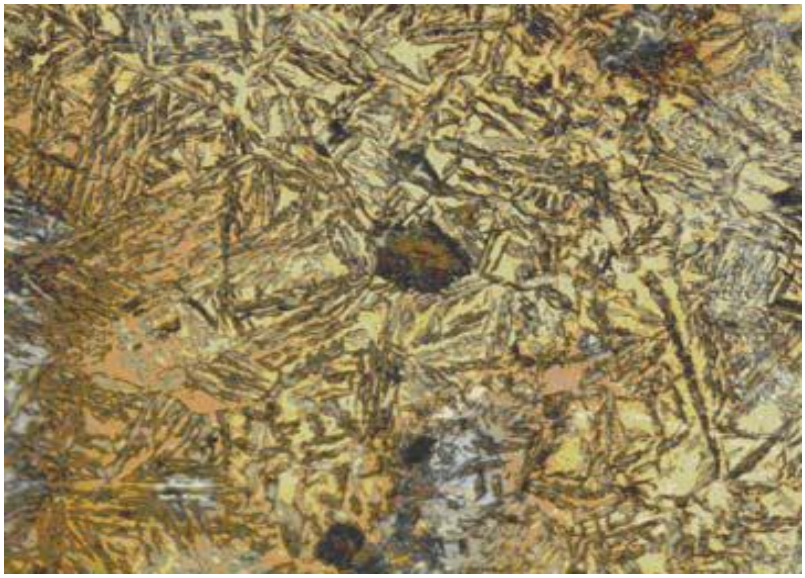


Plate B5: Microstructure of austempered ductile cast iron in molten salt bath at 350^{O}C for 90minuts, austenitized at 900^{o}C. The structure shows ausferrite (bainite).
(*Source:* Almaida and Hassan2009)

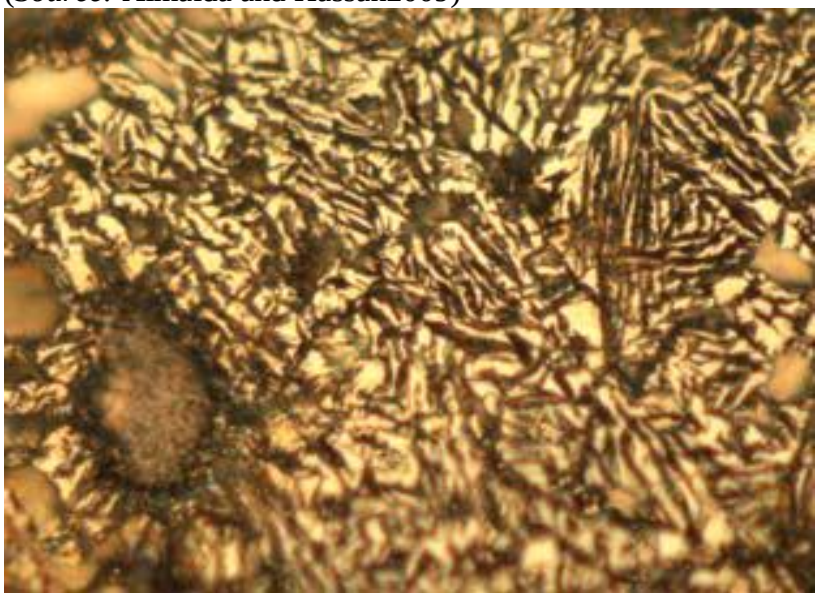


Plate B6: Microstructure of austempered ductile cast iron in molten salt bath at 350^{O}C for 90minuts, austenitized at 850^{o}C. The structure shows ausferrite (bainite).
(*Source:* Tun and Lwin 2009)



Plate B7S: Microstructure of austempered ductile cast iron in molten salt bath at 250^{O}C for 90mints, austenitized at 900^{o}C. The structure shows ausferrite (bainite).
(*Source:* Alan, 2012).