MECHANICAL PROPERTY INVESTIGATION OF HARDENED AND TEMPERED DUCTILE IRON

Thesis submitted in partial fulfilment of the requirements for the award of the degree of

Master of Technology

in

Mechanical Engineering

Submitted by

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Under the supervision of Dr. S.Sen (supervisor)

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CERTIFICATE

This is to certify that, the work embodied in this Thesis Report entitled "MECHANICAL PROPERTY INVESTIGATION OF HARDENED AND TEMPERED DUCTILE IRON" by Mr. Litu Behera has been carried out under my supervision and guidance for partial fulfillment of the requirements for the degree of master of technology in MECHANICAL ENGINEERING during the session 2012-2014 in the department of metallurgical and materials engineering, national institute of technology, Rourkela

To the best of our knowledge, this work has not been submitted to any other University/institute for the award of any degree.

I appreciate his presentation during the project period. He completed the project successfully as per the requirements and I wish his success in all future endeavors.

Dr.S.Sen

Associate Prof. MME. Dept.

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Lastly, I take this opportunity to express my heartfelt regards and sincere thanks to my family member for their timely support and encouragement, which encouraged me a great deal in completing the thesis and put it in black and white.

LITU BEHERA

CONTENTS

Abstract

In their attempt to develop a particular type of cast iron having better mechanical properties than the malleable iron, the scientists ultimately discovered the spheroidal graphite (S.G) Iron. In this type of cast iron, graphite is present in the form of nodules so it doesn't break the continuity of structure. Moreover due to the regular shape of graphite, it provides ample amount of plasticity unlike gray cast iron where the flake of graphite acts of sharp notches and thus destroy plasticity. Due to its excellent combination of strength, ductility and toughness the S.G Iron is now being widely used in different structural applications and now-a-days it is replacing steel in various fields. The properties of S.G Iron can be improved further by means of proper heat-treatment procedure. In the present work, efforts have been made to study the effect of tempering on S.G Iron and correlate the properties with the micro-structure.

Keywords: - S.G Iron, heat treatment, Tempering, mechanical properties, micro-structure.

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Chapter-1

Introduction

and

Basic concept

1.1. Introduction to ductile iron

It was adapted in 1948, after that its evolutionary work in the 1950 predicted some improvement in engineering application and finally, its application implemented in 1960. It was also found that when solidification of molten cast by addition of cerium and magnesium or both to molten iron. [1, 2] Its name was derived as spheroidal graphite embedded in the matrix of pearlite and ferrite. The high carbon and silicon content of cast provided the advantageous of casting process, but graphite nodules has nominal change to mechanical properties of the cast. [4] The different grade was produced by spheroidal graphite controlling matrix either by heat treatment or alloying. [5] Ductile iron was employed heating process that gave desirable mechanical properties towards designing, which are used in commercial application. Ductile irons can be improved further by means of heating process to increase toughness, ductility, wear resistance and also to increase relieve internal stresses, strength, corrosion resistance and microstructure stabilization and also improve machinability. [27]

Alloy addition can be produced to matrix structure, which can be controlled by the heat treatment. It can give strange mechanical properties because of spheroidal shape of graphite in matrix. Special study of alloy ductile irons and ductile iron without alloy can provide strange mechanical properties for special commercial application. [1, 6]

1.2. Birth of ductile iron

The development of gray and malleable irons during the first half of 20^{th} century, foundry men continued to search for the ideal cast iron as known as gray iron with superior mechanical properties than malleable iron. 70 years ago, American foundry men's society made a statement, "as your indulgence is requested to permit the posing of one question will real control of graphite shape can be realized gray iron? Processing visualization a material, graphite flakes or grouping resembling those of malleable iron instead of elongated flakes". [2, 3] After some days, in INCRL, ladle addition of magnesium to melt was performed by Keith Dwight Millis. After the addition, there was no flake in cast but some graphite spheres presented there. After that the INCRL revealed his development, magnesium as graphite Spherodizer was suggested by Millis in 1943. It was granted a patent on October 25, 1949. This was the official birth of ductile iron. [1, 4]

1.3. Production of ductile iron

Spheroidal graphite irons were produced directly by the nodulising and desulphurization and also inoculation of the melt. As magnesium is carbide former, ferrosilicon can be added immediately as inoculant. Remelting causes reversion to flake graphite due to the loss of magnesium. Stirring of molten alloy after addition of nodulising elements evolves a lot of gas, which gets dissolved in liquid alloy, and forms blow –holes in solid casting. Similarly, sulphur helps to form graphite as flake. Thus the raw material for producing spheroidal graphite iron should have low sulphur or remove sulphur from iron during melting, or by mixing iron with a desulphurising agent such as calcium carbide, or soda ash. And also by the nodulising process, and in which the magnesium is added to remove sulphur and oxygen still present in the liquid alloy and provides a residual 0.04% magnesium, which causes growth of graphite to be spheroidal, probably the interface energy becomes high to have a dihedral angle of 180°, that means the graphite dose not wet the liquid alloy. Magnesium treatment desulphurises the iron to below 0.02% sulphur, before alloying with it magnesium and such elements have strong affinity for sulphur, and thus scavenge sulphur from the molten alloy as an initial step for producing spheroidal graphite iron. These additions are expensive to increase the cost of spheroidal graphite iron produced. Thus, sulphur of molten alloy, before nodulising, should kept low. Magnesium can be added when molten is neared to 1500°C, but it can vaporize at 1150° C. it floats on the top of the melt because of its lighter nature. And reactive born off also produced at the surface. In this case it is added as Ni-Mg and Mg-Si-Ni alloy or coke of magnesium to produce the violence of reaction and to have saved magnesium. Its mental is added as metal itself. Different method helps to produce this one are in-mould technique, covered ladle technique, porous plug stirring and ladle transfer etc. ferrosilicon can be produced before casting by addition of magnesium. [7]

1.4. Chemical composition

Chemical composition of this material is same with Fe-Si-C alloy and gray iron. It has led to the development of cast iron technology. The brittle nature of gray and white irons is less than developed cast iron and ductility is more than white and gray iron. However the heat treatment can be applied to this type of cast iron. [8]

Table-1.4:- chemical composition of ductile iron

%P	%Mn	%S	%Si	%C
0.01	0.3	0.01	2.5	3.7

1.5. Different micro-constituents in ductile iron

The mechanical properties of ductile iron are evaluated by the different phase in it. However the proportion of different phase can be manipulated the mechanical properties like tensile strength, ductility, damping capacity and hardness, which are used in engineering applications.

Graphite

It is a hexagonal crystal structure and also a stable form of pure carbon in ductile iron and has free carbon. It can improve damping capacity and machinability. It can reduce strength and shrinkage severely. However it depends on shape like sphere, star and flake. It is a soft material. [28] It has lubricity, low density, high thermal conductivity and low hardness. The shape of graphite plays an important role in evaluating the tensile strength, impact energy and hardness of cast irons [9]

Ferrite

It is a virgin phase and BCC crystal structure of ductile iron. Alloying elements like silicon and carbon with iron makes solid solution, which can give low temperature phase know as ferrite is a stable equilibrium. It is soft metals and has ductility but less tensile strength and hardness. [28]

Pearlite

It has produced by a eutectoid reaction. It is mixture of lamellar form of cementite and ferrite. It obtained above the bainite region by eutectoid transformation of austenite. It is a metastable, and has led strength without brittleness. And also give good machinability. [28]

Cementite

It is intermetallic phase and orthorhombic crystal structure. It is hard material can give 800-1400 HV100, and also its hardness depends upon elements which are substituting in base element. It gives very good wear resistance with less machinability. [28]

Martensite

It is a carbon in iron supersaturated solid solution. It can be formed by diffusionless transformation and also by rapid cooling. In the without tempered condition It gives very hard and brittle nature. Normally it follows the tempering heat treatment to reduce the carbon content. It provides wear resistance and high tensile strength. However it is a hard phase with metastable condition. [9, 28]

Austenite

It can be obtained by carbon dissolve in iron at high temperature region. It is a FCC crystal structure. These helps to formation of other different phase like austenite to pearlite, similarly austenite to martensite [9] it may stable equilibrium phase and also soft and ductile. [28]

Bainite

It can be produced by heat treatment or by alloying. It has the mixture of ferrite and carbide and also eutectoid product. Microstructure of bainite is different from martensite. It can be characterized by C-curve. In bainite both ferrite and cementite has own orientation. Which are follows a relation with each other for growth in grain. [9]

Carbide

It is hard and brittle in nature. It can be produced by diffusion of carbon with iron and also with molybdenum, chromium and vanadium. The wear resistance and brittle increases with increasing of carbide in cast iron and also it has poor machinability. A Spherical or Lamellar form of the carbides provides good wear resistance and tensile strength in conventional ductile iron and heat treated ductile iron. [10]

Ledeburite

It can be produced by eutectic reaction of massive austenite phase composed of cementite and austenite. And also has produced by austenite to cementite and pearlite transformation. It gives high hardness and wears resistance. [9]

1.6. Types of ductile iron

Ductile iron classified about different phases it have, it have derived as ferritic, pearlitic, martensitic, austenitic, bainitic etc.

Bainitic ductile iron

It has obtained through alloying or by heat treatment. It has given high hardness and wears resistance in material. [10]

Austempered ductile iron

It has obtained by special austempering heat treatment. It has given superior fatigue strength and wear resistance and also twice stronger than pearlitic ductile iron. [9]

Austenitic ductile iron

It has obtained by austenising heat treatment. It has provided good strength and dimensional stability at elevated temperature and also offered good corrosion and oxidation resistance. [10]

Martensitic ductile iron

It has produced by the quenching and hardening. It has given martensite structure, which is very hard, develops wear resistance and very high strength but less in ductility and impact capacity. [28]

Pearlite ductile iron

It has produced by the alloying or by heat treatment. Its machinability is superior to steels because of graphite spheroids in matrix of pearlite. And also it has given high strength, good wear resistance and damping capacity. [10]

Ferritic pearlitic ductile iron

It has produced in as cast condition. It gives good machinability because of graphite spheroids are in matrix of both pearlite and ferrite phase in ductile iron. The properties of this type ductile iron are intermediate between pearlite and ferrite phase. [9]

1.7. Effect of alloying on matrix structure

Nickel

It is the mildest perlite promoter increases proof stress but little effect on tensile strength. Their large additions in excess of about 2% are danger of embrittlement. It does not give rise to carbides unlike manganese. Carbide free pearlitic structure can be produced by the addition of 1 to 1.5% nickels with manganese. Hardenability in normalizing and quenching treatments is promoted by nickel addition. [10]

Molybdenum

It is a pearlite promoter in molten Iron and in the heavy section it operates the annealing cycle in slow rate. It can increase hardness and proof stress and also Danger of embrittlement that's why it giving low elongation and tensile strength, which may improves at elevated temperature. In heavier section castings, molybdenum can be employed to increase hardness and tensile strength. The acicular structure may be obtained by the combine addition of nickel and molybdenum, which can be characterized by creep strength toughness, fatigue strength high tensile strength. [28]

Copper

It is strong pearlite promoter and the tensile strength, proof stress, and hardness with no embrittlement of matrix can be obtained by the addition of copper because of it is pearlite promoter. However it was found the spheroidal graphite by addition of copper. It can raise transition temperature and decrease impact resistance when lower silicon content. And also decrease ferrite amount. [10]

Chromium

It is very strong carbide former. It should not be employed to the molten iron when the carbide free structure is required because of it is carbide former. The carbide and pearlite

stabilizer can be occurred by the addition of chromium. Particularly, it is used for developing as cast ferritic structure. [9]

Carbon

Hardness can be decreased by addition of 0.15% carbon at that moment the ductility increase. However the carbon can be affected the modulus of elasticity of ductile iron by its volume present in the matrix. [10]

Silicon

The tensile strength, proof stress, hardness and danger of embrittlement can be increased by the addition of silicon and increase spheroidal amount however it can reduce the carbide and pearlite but increase in ferrite. Impact value will fall below the normal when silicon content and temperature are changed. [10]

Manganese

Pearlite can be promoted by addition of manganese. It can increases hardness and proof stress. And also it is danger of embrittlement. The intercellular carbides in heavy section can be formed by manganese addition. Manganese act as an alloy when the sulphur is absent and also it dose refining and stabilizing the pearlite and stabilizer of carbide. Similarly, reduce in ferrite in molten iron. The pearlite to ferrite transformation can be performed by the manganese addition. [28]

Phosphorus

It can promote temper embrittlement when added to molten metal. The undesirable brittle phosphide can be formed at high phosphorus levels as network manner. However the damping capacity and ductility are adversely affected and also impact resistance may be seriously changed. The iron is to be used as cast there should be kept 0.04% of phosphorus. [10]

Magnesium

It promotes nucleate graphite nodules in ductile iron when added to molten metal. [10]

Cerium

It is added to liquid cast iron to inclusion of cerium sulfide for controlling the sulfide shape. It is a strong deoxidizer. [9]

1.8. Mechanical properties of ductile iron

Mechanical properties of spheroidal graphite cast iron are influenced by different matrix proportion. The proportion of matrix is depending on composition of the metal. And also depends upon the graphite spheroidal nature. However ferrite, pearlite and cementite phase proportion are changed the mechanical properties of ductile iron. Similarly alloying elements and its quantity also affected the mechanical properties. The hardness with ductility, tensile strength and impact energy can be changed by the area fraction of martensite, bainite and austenite phase. The damping capacity decreased by the spheroidal graphite than flak graphite situated in ductile iron. It can be observed the machinability decrease by change of phase from ferrite to pearlite. Dimensionally the ductile iron is stable at high temperatures because of there is no internal oxidation. [24]

Tensile strength

Progressively higher strengths could be obtained in cast iron by controlling the shape, size and quantity of the graphite. Changing the shape to aggregates of temper carbon as in the case of the malleable irons or to spheroidal graphite which causes least interruption to the continuity of the matrix as in the case of spheroidal graphite iron, has not only helped to increase strength but also introduce appreciable ductility. Strength is also influenced significantly by the nature of the matrix. In all cases a fine fully-pearlitic matrix increases strength and hardness together with satisfactory machinability. Cementite increase hardness and wear resistance at the expense of toughness and machinability while ferrite increase toughness and ductility at the expense of strength, hardness and wear resistance. [23]

Modulus of elasticity

The variation in strength is brought about by the nature of the matrix; the elastic modulus remains virtually constant and dose not vary with strength. However, in the case of flake graphite cast iron, the quantity, shape and size of the graphite has been found to affect elastic modulus in the same way as strength. [23]

Hardness

While hardness bears some direct relationship to strength in other types, correlation is poorer in the case of the flake graphite types, as the quantity, shape and size of the graphite and the presence of cementite and the phosphide eutectic cause a much wider scatter in the hardness values for the same strength. It will be seen that the range widens with increasing strength but the maximum hardness attained can be less with properly alloyed irons. A lower maximum harness for the same strength can mean better machinability and less risk of hard spots which is even more important to prevent non-uniform wear. [23]

Impact energy

The excellent damping capacity of grey cast iron is due to its graphite content. Damping capacity may be taken to very roughly inversely as the elastic modulus. A good damping capacity readily absorbs vibrations and minimizes stress resulting therefrom. [23]

1.9. Factors affecting Properties of Ductile Iron

Spheroidal graphite iron is a special kind of material. Now a day, the application of ductile iron in heavy engineering industries is continuing due to its good combination of strength with ductility. It can be shown by microstructure also.

Effects of Graphite Shape

It can be described by the morphology of irregular shape and nodularity of the graphite. The yield and tensile strengths of Ductile Iron changes with changing of nodularity, For ferritic irons, the relationships between nodularity and strength has been changed by two methods, one is controlling magnesium and another is lead control. Nodularity is decreased by reducing the amount of residual magnesium. It shows nodules become elongated, but do not become sharp or "spiky". [10]

Effects of Nodule Count

The number of graphite nodules per millimeter square shows the nodule count and it expresses in number. The mechanical properties of Ductile Iron can be influenced by the nodule count. Good metallurgical quality can be indicated by high nodule count is present

there. The microstructure is affected by the nodule count but it has not affected tensile properties. [9]

Effects of Graphite Volume

The carbon content in ductile iron can be influenced the volume fraction of graphite in Ductile Iron. The volume fraction and size of graphite nodules can be influenced by the casting size. It also influences mechanical properties. The cooling rate of the casting can be reduced by increasing section size. The stable graphite phase is causing more carbon to precipitate in it, instead of higher cooling rates. Mechanical properties can be influenced by the graphite flotation which provides variations in graphite volume within larger castings. Graphite flotation occurs when at high carbon equivalent and low cooling rates are produced graphite flotation which combines to produce large nodules that may rise during solidification. [10]

Effects of Carbide content

The mechanical property of ductile iron castings are influenced because of Carbides in ductile irons can occur in three forms: Eutectic carbide (or chill) results mainly from the rapid solidification and is most prevalent in corners and thin sections. Inadequate inoculation, low carbon and in particular low silicon and the presence of carbide promoting elements increases the likelihood of carbides being present in the structure. Inverse chill, which has fine acicular form, occurs at or near the heat center of a casting section. The geometry of the casting and method of running the casting are important variables and the problem is often only solved by re-positioning or altering the size of in gates to change the pattern of solidification of casting. [10]

Effects of Matrix

Mechanical properties of ductile iron are determined by the different type of matrix, and also by their hardness, nodule count, low porosity modularity and carbide content. The matrix consists of ferrite and pearlite and both in common grades of ductile iron. The purest iron phase in Ductile Iron is ferrite. It shows good machinability, tensile strength and hardness and ductility and toughness. [10]

1.10. Heat Treatment of Ductile Iron

Annealing

In this process, heating the castings in electrical furnace up to 900°C-1000°C and is kept there for 1 hour and the holding time increases in 1hr with increasing the 25mm thickness of specimens. The holding time can be increased up to 8hrs for heavy casting specimen. After the austenization, the specimens are cooled in same furnace to 650°C and are kept there for 5 hours and also the holding time increases in 1hr with increasing the 25mm section thickness of specimens. The cooling should not exceed 20°c per hour. Here it forms ferrite matrix. Annealing sometimes referred to as full annealing if castings are carbidic as – cast. The samples are hold at a temperature of 900°c for 2hours and one additional hour per inch section thickness. Then, cool to 700°c and hold there for 5 hrs. Finally, the cooling rate is 110°C per hour to 480°C, and then air cool. [11]

Normalizing

In this process, heating the castings in electrical furnace up to 870°C-1000°C and is soaked there for 1 hour. Normalizing temperature and soaking time are varied with shape and composition of the specimens. The result of normalizing is a fine pearlite matrix. Heat the casting to 900°c, if massive carbides are present in the structure. Then, the holding time increases in 1hr with increasing the 1 inch thickness of specimens. Remove the casting from the furnace and cool in atmospheric air. The heavier the section the more alloying is needed. To increase hardness and strength Cu is mixed. When Si content is more than 2.5%, the casting should be fast cooled to get a fully pearlitic matrix. [11]

Quench hardening and tempering

In this process, Casting are austenitized at 850°c-1000°c in electrical furnace, this is followed by oil or water quenching. It is reduced to tresses in quenched sample, oil or water is preferred as quenchant. If Castings are complex shapes then it can be quenched in at 80°C-100°C maintained oil to avoid quenched cracks. After that, quenched samples were placed in another electric furnace at different tempering temperatures the range is 300°c-600°c and tempering time. After that, the samples were cooled by atmospheric air. The microstructure of quenched specimens is revealed the martensite phase. [11]

Austempering

In this process, Casting are austenitized at 850°c-1000°c in electrical furnace, this is followed by salt bath quenching. It is reduced to tresses in quenched specimen; salt bath is preferred as quenchant. If Castings are complex shapes then it can be quenched in at 230°C-430°C maintained salt bath to avoid quenched cracks. After that, quenched samples were placed in another electric furnace at different tempering temperatures the range is 200°c-500°c and tempering time. After that, the samples were cooled by atmospheric air. The microstructure of quenched specimens is revealed the bainite phase. [11]

1.11. Applications of ductile iron

The applications of the S.G. iron have increased tremendously in recent times. Spheroid iron has used in India railway and also automobile sectors. Examples are **Engine crank shaft, Brake caliper, disc brake anchor, brake anchor plate, Rack and pinion of steering assembly, Brake shoe for heavy duty brakes, Piston rings etc**. and also in production sectors, Examples are **Machine tool bed, Electric insulator post and cap, Steering knuckle, Piston for impact drills, Rolling mill rolls, Moulding boxes and mould box clamp, Glass moulds, Spacer cage for rolling bearing, Wind mill items etc**. Because of its good wear resistance and fluidity properties. And corrosion is also superior to steel those having low carbon. However, at elevated temperature it prevents the scaling and growth. [22]

Chapter-2

Literature review

2.1. A Review of Earlier Research work

Literature review

After the studied of all available literatures that was made to understand and given some idea about how the quenching and hardening process followed by tempering of spheroidal graphite cast iron was conducted. It was also given some idea about study of nodular and ferrite matrix structure which was changed to quenched martensite, tempered martensite and acicular ferrite matrix, after the quenching and hardening followed by tempering heat treatment and also studied the mechanical properties of heat treated sample with different processing variables and its possible applications.

2.1. A Review of Earlier Research work

Addressing the published research works.

M.Moshrefi-Torbati and Ali M.Rashidi, investigated the effect of the tempering conditions on the mechanical properties of dual matrix structured ductile iron. The quenching and tempering heat treatment process were applied to the cast iron. The composition of material was 3.56%C, 1.94%Si, 1.33%Ni, 0.28%Mn, 0.29%Mo, 0.017%P and 0.012%S for analyze. First of all the specimens were austenized at 950°C and kept there for 2hrs then quenched. After that the specimens were tempered at 300°C, 400°C, 500°C and 600°C for 1hrs respectively and also tempered for 30, 90, 120, 150 and 180 minutes at 500°C. After that the specimens were machined down to standard dimension. The tensile test was carried out after the dimensioning. It was seen that the ductility value increased with increasing tempering temperature, but in the range of 400°C to 500°C, there was increased by slowly and gradually. However the good ductility and high toughness was obtained by tempering the specimen at higher than 500°C. it was produced dual matrix which have ferrite and martensite matrix structure and it was also seen that UTS, YS was decreased by increasing tempering temperature, but within the range of 400°C to 500°C, it was roughly constant. However the ductility value increased with increasing the tempering time up to 120 minutes. Finally, it was dropped because of due to tempered embrittlement in the range of400°C to 500°C. [14]

I.C.Hsui and L.C.Chang, studied the erosion behavior of ductile iron. The quenching and tempering heat treatment process were applied to the cast iron. The composition of material was 2.88%C, 0.11%Si, 0.039%Mn, 0.29%Mo, 0.011%P and 0.046%S for analyze. The

specimens were austenized at 950°C and kept there for 2hrs then quenched in water to produce martensite structure. Lower and upper bainite structure was obtained by austempering at 420°C and 280° C for 1hrs respectively. After the austenization of ductile iron, the specimens were polished to remove oxide on the surface. After that the specimens were subjected to erosion wear test, and then the rate of erosion was manipulated an average of three test results which were taken as the total weight loss in 8 minutes that was 500gm of the impacted SiO_2 particles. The short term erosion wear test was also conducted to characterize the wear mechanism. The specimen were polished and etched in 3% nitric acid after that erosion wear test was done with 1gm of SiO_2 eroded the particles for short term. The results of erosion wear test of ductile iron was shown as the erosion rate increased first then decreased with impact angle. Erosion test for short term was revealed the both of soft ferrite and hard martensite were affected by cooling of ductile iron. However the hardness was increased with increasing impact angle while the ductility value or erosion rate was shown as inverse trend. [15]

Kadir Kocapete et.al, studied the tensile fracture behavior of oil quenched from inter critical annealing temperature range and the tempered ferritic ductile iron having dual matrix structure with different martensite volume fraction and its morphologies was studied for a ductile ferritic cast iron. The chemical composition of specimen was 3.5%C, 0.005%Cu, 2.63%Si and 0.318%Mn for this experiment. The specimens were austenized at 900C ads then quenched in oil and tempering was also done. After that comparison was done between as cast material and quenched material, and revealed that the ferrite plus graphite structure in as cast condition and martensite structure in quenched condition and then the specimens were annealed at 795C and 815C for 30 second respectively, then quenched specimens were tampered at 550C for 1hrs and 3hrs. The tensile test, microstructure and fracture surface examination was done after these treatments. It is found that the ductility of dual matrix structured ductile iron was compared with ferritic ductile iron, but it was also superior to fully martensite structured ductile iron. Ductile fracture pattern changed to moderate ductile pattern in the case of increasing continuity of martensite structure. Dual matrix structured ductile iron was shown by fractographic examination. However the 0.2% proof stress and tensile strength compared in-betweens conventionally heat treated and quenched specimens. [16]

Gulcan Toktas et.al, studied the influence of the matrix structure on the mechanical properties and impact toughness. The Chemical composition of specimen was 3.6C%, 2.29Si%, 0.053P%, 0.011S%, 0.08Mn% and Fe is balanced. The cast ingots were divided into five groups in this work. The material in the first group was used directly or without any heat treatment i.e. as-cast. For the second group the materials were subjected to ferritic heat treatment. The ferritization procedure followed the usual two stage isothermal holding, in which spheroidal graphite cast iron was held at 920°C for 5hrs, furnace cooled to 720°C for the second isothermal holding for 7hrs, and then furnace was cooled to room temperature. The materials of the remaining groups were heated to 920°C then holed there for 5hrs at this temperature, then cooled to room temperature following different cooling rates, as in still air cooled, cooled in an isolated and block forced air cooled. Microstructure of ferritic heat treatment was shown the graphite nodules embedded in the fully ferritic matrix. The microstructure of ferrite-pearlite matrix with graphite nodules in the as-cast condition was shown typical "bull's eye" in which many of the graphite nodules are surrounded by an envelope of ferrite. Both the graphite nodules and their ferrite envelopes were embedded in a pearlitic matrix. While the microstructure was same in forced air and in still air but pearlite to ferrite ratio was increased in latter case and there ferrite imbedded in pearlite. This ferrite formed without any particular relation to the graphite nodules. The tensile properties vary mainly through the influence of the pearlite content of the matrix. The 0.2% YS ranges from 240 MPa for ferritic to 457 MPa for pearlitic matrix of spheroidal graphite cast iron. The UTS of the present material was also increased due to the increase of pearlite level. Compared to the fully ferritic matrix material, the percentage increase of the UTS was found to be about 25% in a ferritic /pearlitic matrix. The ferritic matrix was shown the higher ductility then the pearlitic matrix. The spheroidal graphite cast iron with ferrite matrix exhibits the highest fracture energy, while with increasing percentage of pearlite, have lower fracture energy. The hardness value increased sharply as the matrix structure approaches a fully pearlitic condition. The fractrography was shown that in a ferritic pearlitic matrix structure material the fracture was traveling along a path that connects as many as possible graphite spheroids. It avoided the pearlite structure as much as possible. The fracture surface of ferritic spheroidal graphite cast iron was shown the dimple pattern of fracture. Two different fracture patterns were observed in a ferritic-pearlitic matrix structure, in the vicinity of the graphite nodules, the wider areas of the ferrite phase are deformed considerably. Thus the fracture occurs in a ductile manner, while the brittle fracture with river pattern in pearlitic areas can be observed. [17]

J.O. Choi, J.Y. Kim, C.O. Choi, J.K. Kim and P.K. Rohatgi, investigated the effect of rare earth element on microstructure formation and mechanical properties of thin wall ductile iron castings. Ductile iron castings with 2, 3, 4, 6, 8, and 25mm thickness and various amount of rare earth elements (RE) (from 0 to 0.04%), were casted in sand molds to identify the effects of specimen thickness and the content of RE% on microstructural formation and selected mechanical properties. The effects of RE content and specimen thickness on microstructural formation, including on graphite nodule count, graphite nodule shape, spherodization, and ferrite amount were observed. The yield strength of the specimens with RE within the range investigated were lower than those of the specimens without RE. The elongation was improved with the addition of RE up to 0.03% in ductile iron castings. The additions of 0.02% RE caused a smaller graphite nodule size and a higher number of graphite nodules than those in the specimens without RE at all levels of RE addition; the nodule count decreased with increase in section size. The chill zones were observed in the 2mm thick samples, but were absent in the specimens from castings which were thicker than 2 mm, irrespective of the addition of RE. The nodularity of graphite nodules improved due to the addition of 0.02–0.04% RE. The specimens with RE content up to 0.03% had a lower tensile strength and hardness, higher elongation than those of the specimens without RE. The ferrite content in all castings increased with additions of 0.02% RE. The tensile strengths of the 2 and 3mm thick specimens were also estimated using the relationship between strength and hardness, obtained from the data on the tensile strength and hardness of the 25mm thick specimens. Microstructural features in thin wall ductile iron castings, including the thickness of ferrite layer around graphite ferrite, graphite nodule size, and graphite nodule count, were observed to depend on the amount of rare earth elements and specimens thickness. In the 2mm thick specimens, the addition of RE leaded to a decrease in the amount of chill formation, a higher graphite nodule count and size as compared to those in the specimens without RE. However, in the specimen whose thickness is in the range of 3–6 mm, the addition of RE led to a smaller graphite nodule size and a higher number of graphite nodules than those in the specimen without RE. These results suggest that the role of RE varies with specimens thickness. In addition, chill formation was not observed in the specimens thicker than 2 mm, irrespective of the addition of RE, suggesting that the effect of rare earth in reducing chill formation is important in very thin sections. The nodularity of graphite nodules improved due to the addition of RE. In the samples without RE, the nodularity increases with decreasing sample thickness; the RE addition significantly reduced the variation in nodularity

with sample thickness. The amount of ferrite was observed to depend on RE content and sample thickness. The addition of RE led to a higher amount of ferrite than that of the specimens without RE. The ferrite content was the lowest for the 2mm thick specimen with 0.02% RE. The specimens with RE had a lower tensile and yield strength as compared to same thickness specimens without RE. The lower strength appears to be related to a lower amount of pearlite in the specimens with RE. The ductility of the specimens with RE were lower than those of the specimens without RE. [19]

A.Kutsov, Y.Taran, K.Uzlov, A.Krimmel and M.Evsyukov, studied the kinetics of kinetics of bainite transformation under isothermal conditions in Ni-Mn-Cu-Mo alloyed ductile iron. They studied the formation of lower and upper bainite in the ductile iron were described by different C-curved shaped. They concluded from their work was that morphology of the bainite changes accordingly: the upper bainite had a feathery-like morphology and the lower bainite had a plate like one. These facts were probably, a result of different crystallographic shears during the formation of the upper and lower bainite. A comparison of the dilatometrical data with the X-ray results shows that the bainite transformation ceases once the carbon concentration in low carbon austenite reaches a certain value. It was suggested that this concentration corresponds to curve and the composition of high carbon austenite was increasing. It seemed to be that the increase of the bainitic a-phase volume fraction results in an increase of the volume fraction of high carbon austenite. [22]

Chapter-3

Experimental procedures

3.1. Preparation of test specimen

The tensile and impact samples of ductile iron is produced in commercial foundry known as L&T kansbahl, sundargarh, odisha, India, has been used for this experiments.

3.2. Material composition and dimension

The chemical composition of the samples was determined as given in table-3.2. Samples were subjected to hardening and tempering in accordance to ASM international standards.

Table-3.2

Elements	C	Si	Mg	Mn	S	P	Cr	Ni	Cu	Mo	Ce	Fe
Weight%	3.48	2.14	0.034	0.14	0.008	0.021	0.03	0.46	0.01	0.001	0.007	93.669

Dimension of tensile test specimen

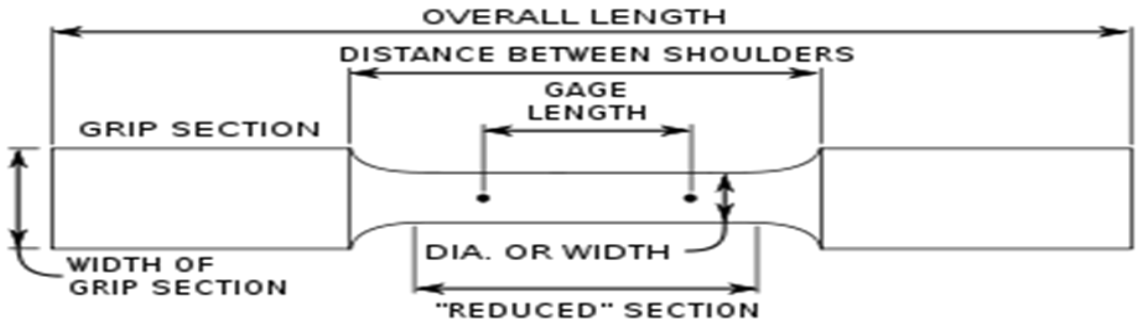


Figure 3.2.1:- dimension of test specimen

FLAT SUBSIZE SPECIMEN, ASTM E8

Grip section=1.25 inch, width of grip section=3/8 inch, Gauge length =1±0.003 inch

Width=0.25±0.005inch, reduced section=1.25 inch, overall length=4 inch

Thickness=0.005≤T≤0.25 inch

Dimension of Impact test specimen

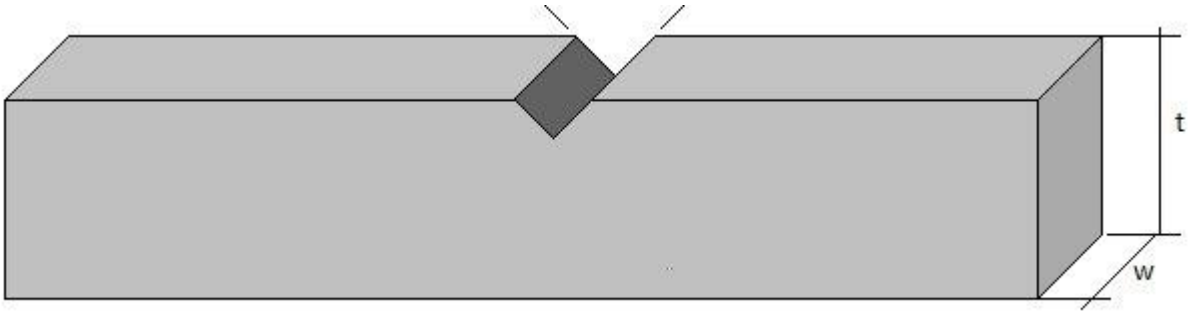


Figure 3.2.2:- dimension of impact test specimen

SUBSIZE SPECIMEN, ASTM D250

Length=2.5 inch, Width=0.5 inch, Thickness=0.25 inch

3.3. Hardening and Tempering

The ductile irons (castings) for the test specimens were austenitized at 1000°C in electrical furnace and then were kept there for 90 minutes. After that, the sample was dropped into mineral oil for 30 minutes. Quenched samples were placed in another electric furnace at 200°C, 300°C, and 400°C for 1hrs and 2hrs respectively. After that, the samples were cooled in atmospheric air.

Figure 3.3.1:- Electrical furnace

The heat treatment condition is listed in table-3.3.

Table-3.3

Sample No.	Austenizing temp.(°C)	Holding time In hours	Tempering temp. (°C)	Tempering time In hours
T2001	1000	1.5	200	1
T2002	1000	1.5	200	2
T3001	1000	1.5	300	1
T3002	1000	1.5	300	2
T4001	1000	1.5	400	1
T4002	1000	1.5	400	2

3.4. Mechanical properties determination

Tensile test

For tensile testing, oxide layers of heat treated samples were removed by stage-grinding and then polished. Mechanical properties of the treated samples were determined using standard methods. For tensile properties, tensile specimens were loaded into a 50kN capacity instron-1195UTM. After that, the stress-strain graphs were obtained from recorded load-elongation data. By the stress-strain graph, ultimate tensile strength, yield strength, young's modulus, percentage elongation and percentage reduction were determined, in accordance with ASTM (Automated materials testing system) standard test procedures. The tensile test has been performed at given parameter: gross head speed=2mm/min. and max. Load=50kN, ASTM E8.

Figure 3.4.1:- UTM machine

Hardness test

For hardness testing, oxide layers of heat treated samples were removed by stage-grinding and then polished. By the micro hardness testing, the average Vickers hardness numbers were determined from taking thirty hardness readings at different positions on the specimens. Micro-hardness test has been performed at given parameter: load=100gf and dwell time=10 sec.

Figure 3.4.2:- Micro-hardness machine

Impact test

For impact testing, oxide layers of heat treated samples were removed by stage-grinding and then polished. By Izod testing, Impact energy readings were recorded thirty digital display of Izod test machine. Impact test has been performed at given parameter: hammer (input energy) =21.7J and setting angle=150°.

Figure 3.4.3:- Izod test machine

3.5. Microstructure examination

For Microstructure examinations, the treated samples were carried out. Each sample was carefully grounded progressively on emery paper in decreasing coarseness (1/0, 2/0, 3/0 and 4/0). The grinding surface of the specimens was polished on Al_2O_3 contained micro cloth. The crystalline structure of the specimens were made visible by etching using solution containing 2% Nitric acids and 98% methylated spirit on the polished surfaces. Microscopic examination of the etched surface of specimens was successfully completed using a metallurgical digital microscope through which the resulting microstructure of the samples was all photographically recorded.

Figure 3.5.1:- digital microscope

3.6. Surface morphology examination

After the microstructure examination, the surface morphology of the treated samples was successfully completed. Surface morphology examination of the etched surface of treated specimens were accomplished using a metallurgical image analyzer with magnification of 200x, by which the resulting nodule size values, nodule count values and area fraction of graphite of the samples were all numerically measured.

Figure 3.6.1:- image analyzer

3.7. Fracture surface examination

After the determination of mechanical properties (tensile test), the fracture surface examination of the treated samples was successfully completed. Fracture surface examinations of the treated samples were undertaken using a metallurgical SEM (scanning electron machine) with magnification of 300x, by which the resulting percentage of dimples and river marking of the samples were all photographically recorded.

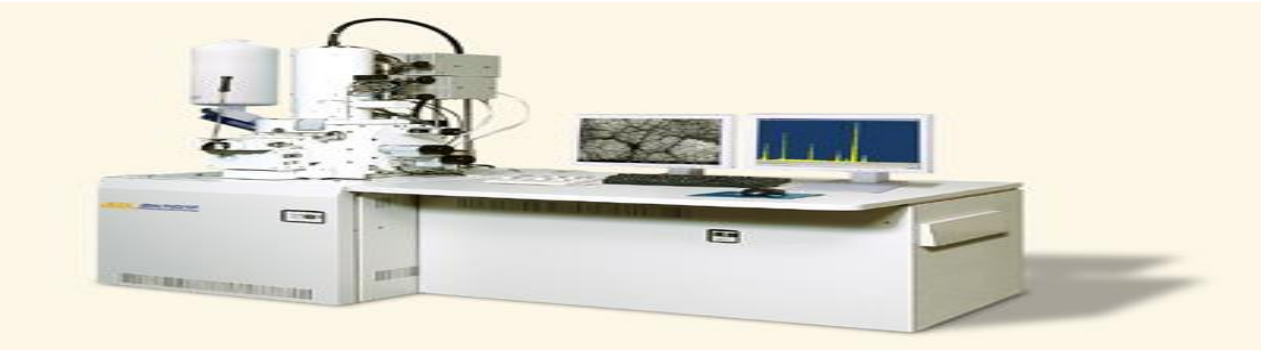


Figure 3.7.1:- scanning electron machine

3.8. Phase analysis

In this technique, after the tensile test, all the specimens were analyzed to estimate the volume fractions of retained austenite, ferrite, graphite nodules, martensite and tempered martensite in treated samples. The XRD has been performed at given parameter: voltage=30kV, current=20Ma and scanning was done in 2θ range from 30° to 90° at the scanning speed of 3° per minute. After that, the profile were analyzed in JCPDS and X-pert high score software.

Figure 3.8.1:- XRD machine

Chapter-4 Results & discussion

4.1. Introduction

In this present work, the properties of hardened and tempered ductile iron were investigated. The specimens were tempered at temperatures 200°C, 300°C, and 400°C for tempering time 1hr and 2hr respectively. These different variables effect on mechanical properties (U.T.S, Y.S and % Elongation, Hardness and Impact energy) and optical investigation (microstructure and facture surface) and XRD analysis of ductile iron are discussed below. The specimens were designated based on the tempering temperature and time, e.g. T2001 means specimen is tempered at 200C for 1 hour after quenching.

4.2. Effect of heat treatment on surface Morphology

Figure 4.2.1: Microstructure of Specimen, Tempered at 200°c, held 1hr (T2001).

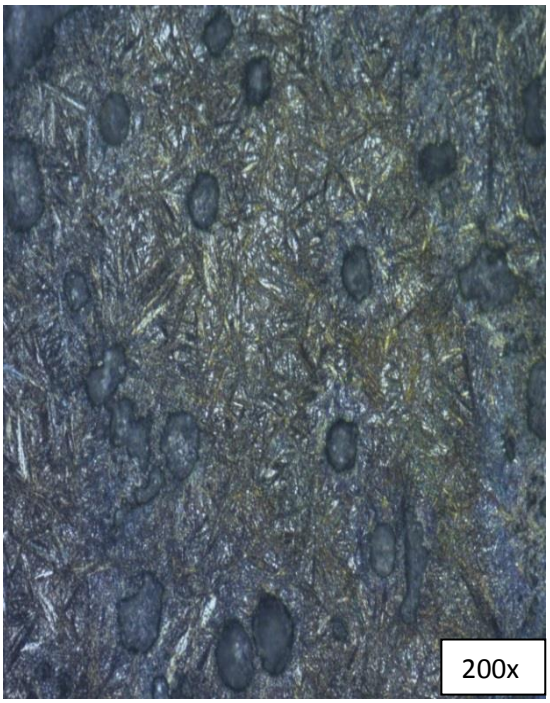


Figure 4.2.2: Microstructure of Specimen, Tempered at 200°c, held 2hr (T2002).

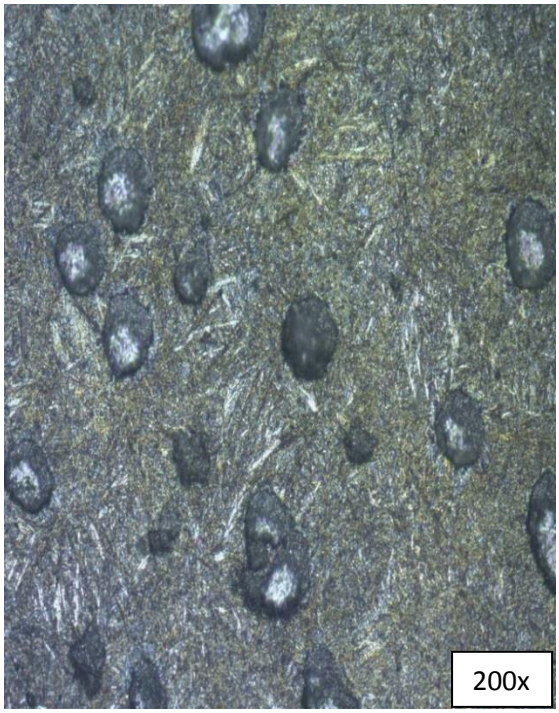


Figure 4.2.3: Microstructure of Specimen, Tempered at 300°c, held 1hr (T3001).

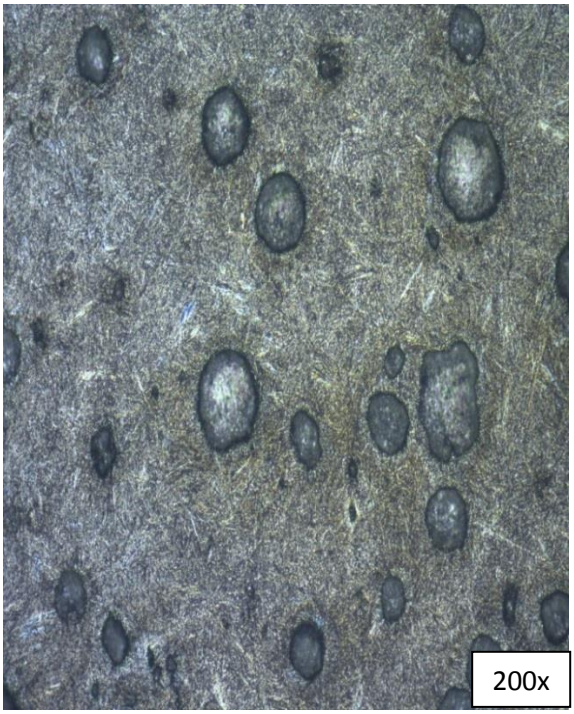


Figure 4.2.4: Microstructure of Specimen, Tempered at 300°c, held 2hr (T3002).

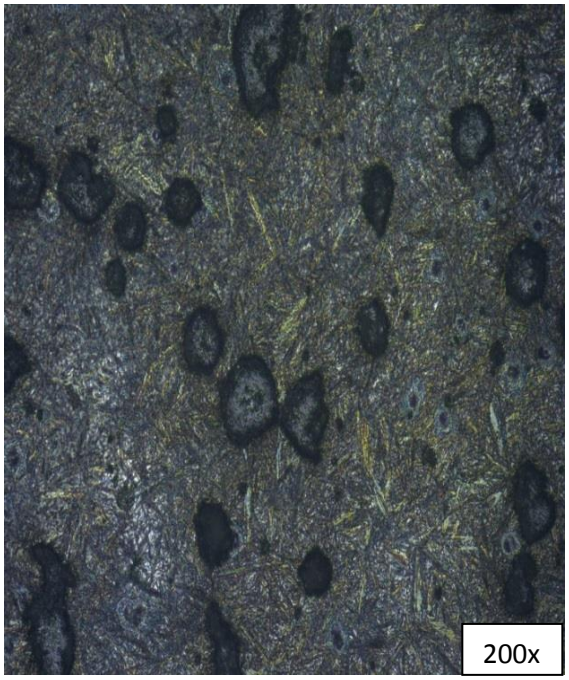


Figure 4.2.5: Microstructure of Specimen, Tempered at 400°c, held 1hr (T4001).

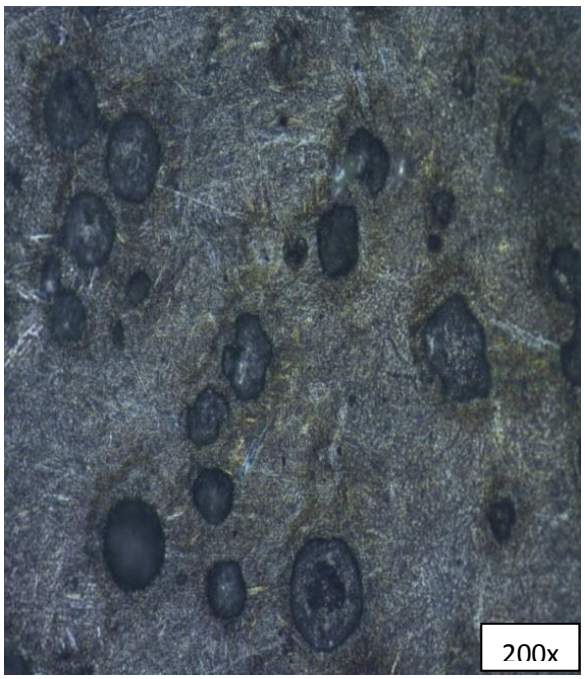


Figure 4.2.6: Microstructure of Specimen, Tempered at 400°c, held 2hr (T4002).

Table-4.2:-Nodularity, nodule count and area fraction of heat treated specimens

Samples	Nodularity	Nodule count	Area fraction of graphite	Area fraction of martensite	Area fraction of tempered martensite
T2001	96.182	435	49.44	50.56	Nil
T2002	96.994	395	48.97	46.56	Nil
T3001	96.878	904	38.04	26.28	35.68
T3002	98.363	479	27.45	21.77	50.78
T4001	97.590	243	19.65	10.01	70.34
T4002	98.582	238	16.94	1.91	81.15

From the figure 4.2.1, it is clearly observed that martensitic structure was obtained after quenching and tempered at 200°C for 1 hour. When the specimens are tempered at 200°C for 2 hours no significant change was found. When tempered at 300°C for 1 hour and 2 hour the martensite structure transformed to tempered martensite. However, the percentage of tempered martensite was found to be more in T3002 than T3001. Further when tempered at 400°C the martensite structure was observed to be transformed into tempered martensite more than that of 300°C for 1 hour. Further when tempered for 2 hour at 400°C martensite was appeared to be transformed into tempered martensite more than 97.7%. From quantitative analysis it was found that martensitic area fraction for the specimen T2001 and T2002 are 50.56 and 46.56 percentage respectively.

For the specimens T3001 and T3002 it was observed that 57.58% and 70% of martensite was transformed to tempered martensite with remaining 42.42% and 30% martensite respectively. Similarly for specimen T4001 and T4002 the tempered martensiteic area fraction was found to be 87.55% and 97.7%. However, very miner percentage of retained austenite was observed after quenching.

4.3. Effect of heat treatment on phase analysis

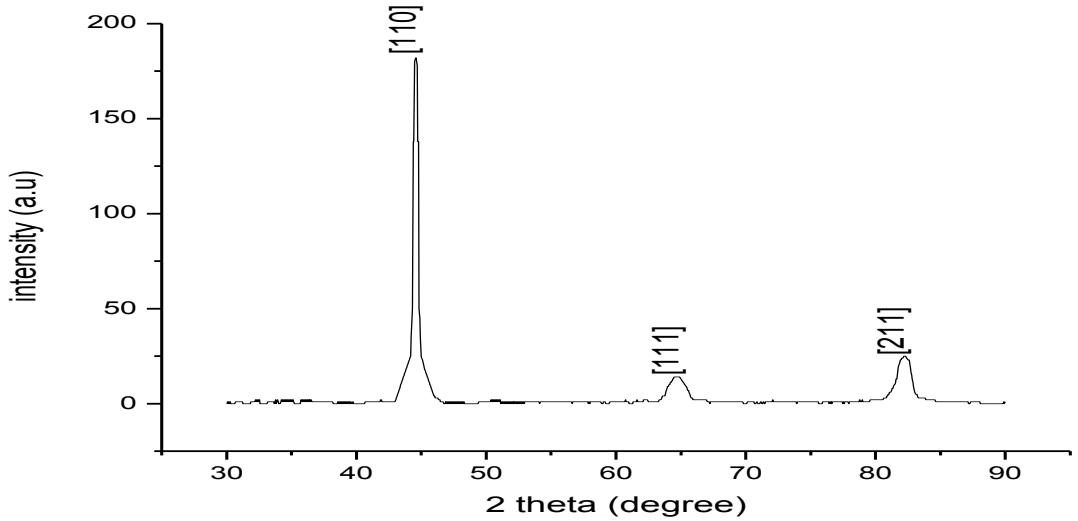


Figure 4.3.1: XRD of Specimen Tempered at 200°c, held 1hr (T2001).

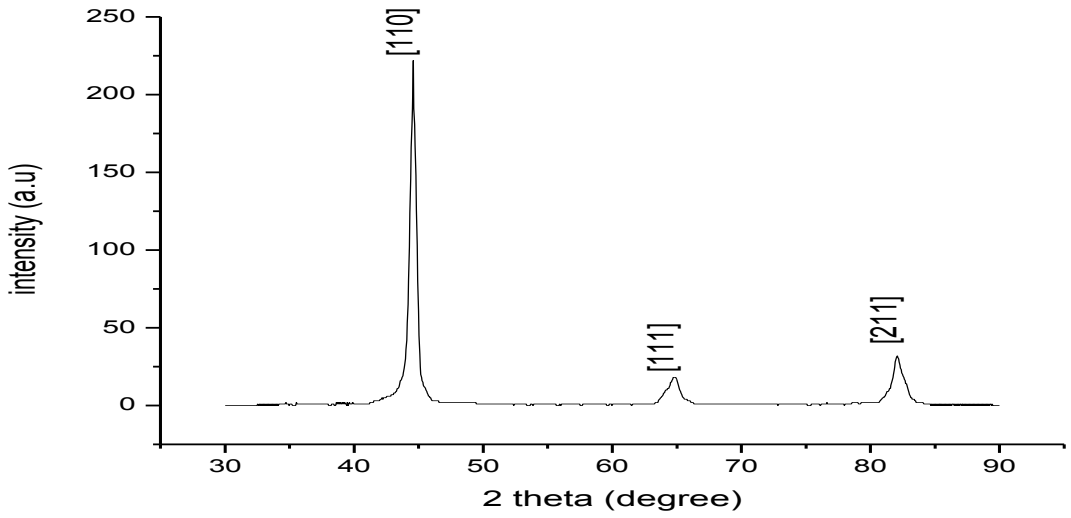


Figure 4.3.2: XRD of Specimen Tempered at 200°c, held 2hr (T2002).

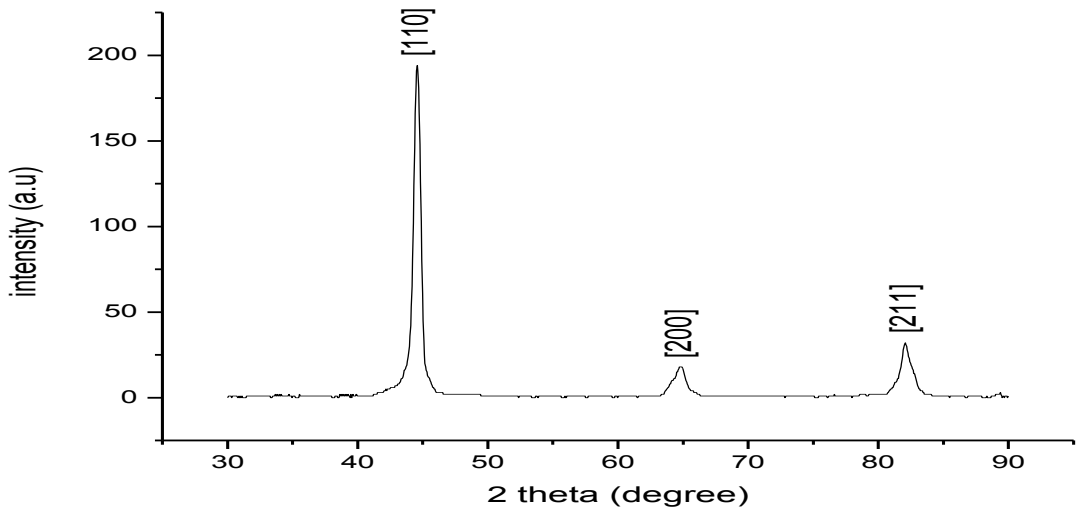


Figure 4.3.3: XRD of Specimen Tempered at 300°C, held 1hr.

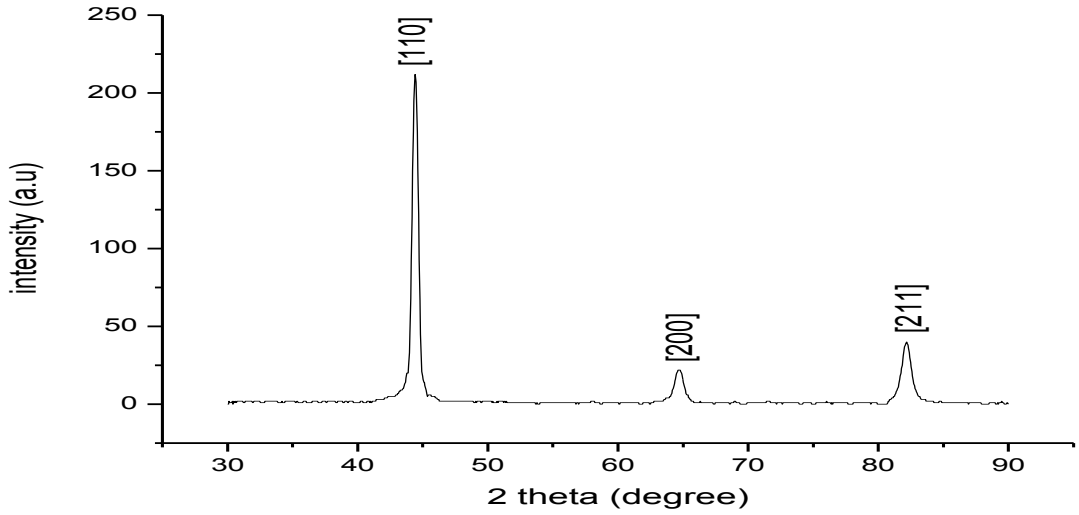


Figure 4.3.4: XRD of Specimen, Tempered at 300°c, held 2hr.

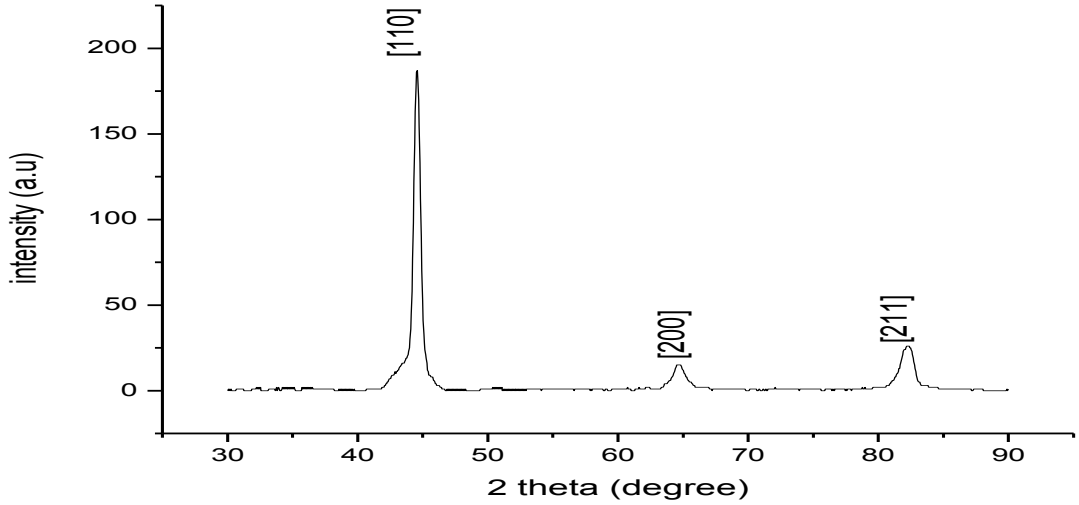


Figure 4.3.5: XRD of Specimen, Tempered at 400°c, held 1hr.

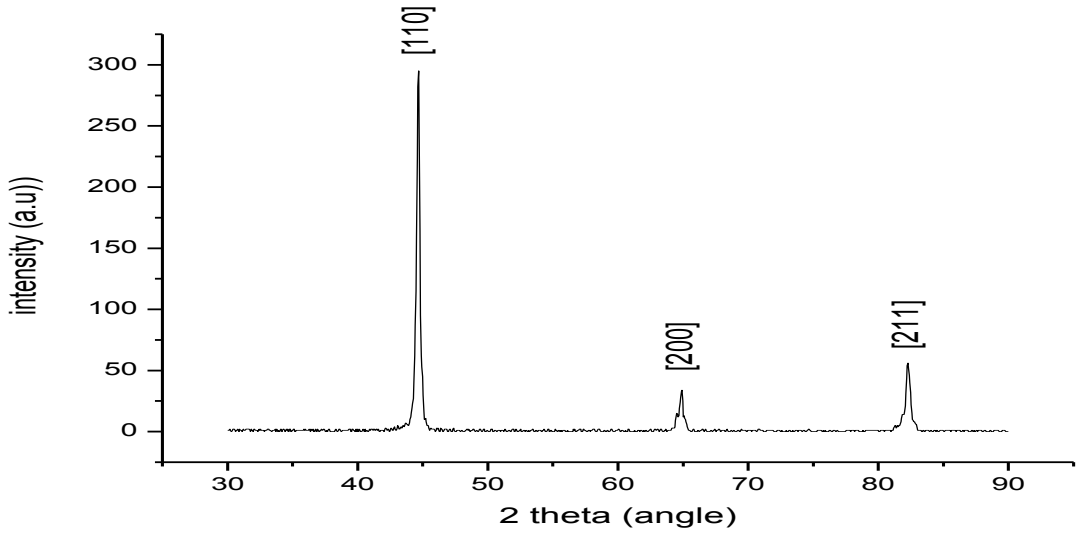


Figure 4.3.6: XRD of Specimen, Tempered at 400°c, held 2hr.

Each of the specimens was under gone XRD study and upon analysis BCC crystal structure was found with one single austenitic peck for specimen T2001 and T2002.

4.4. Effect of heat treatment on Fracture Surface

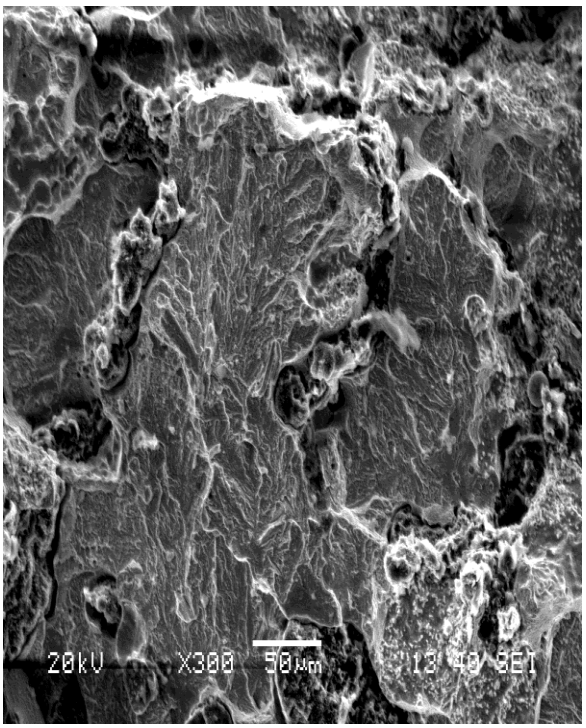


Figure 4.4.1: Fracture surface of Specimen, Tempered at 200°C, held 1hr.

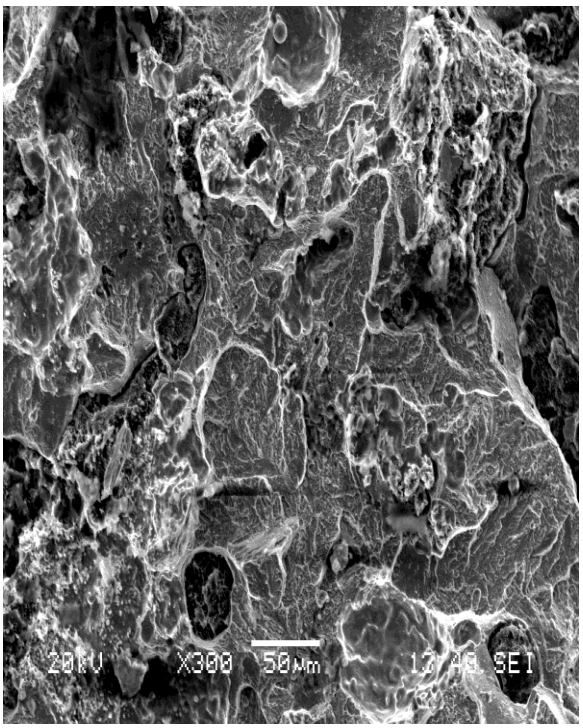


Figure 4.4.2: Fracture surface of Specimen, Tempered at 200°C, held 2hr.

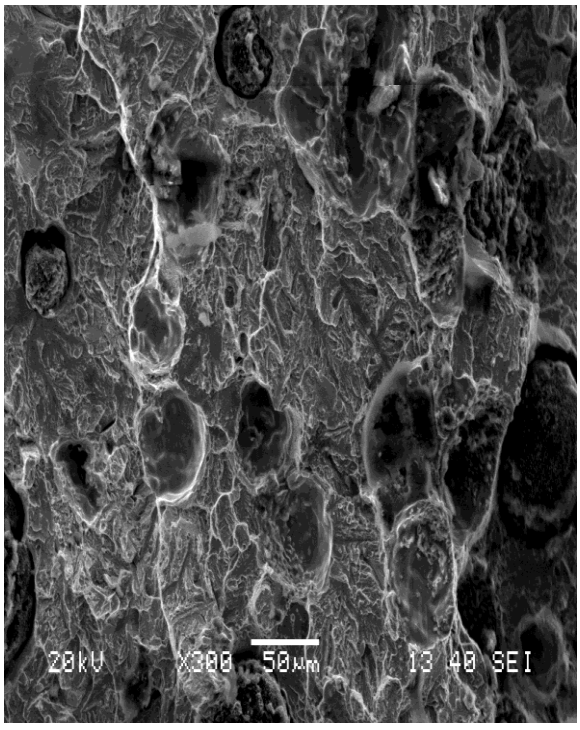


Figure 4.4.3: Fracture surface of Specimen, Tempered at 300°C, held 1hr.

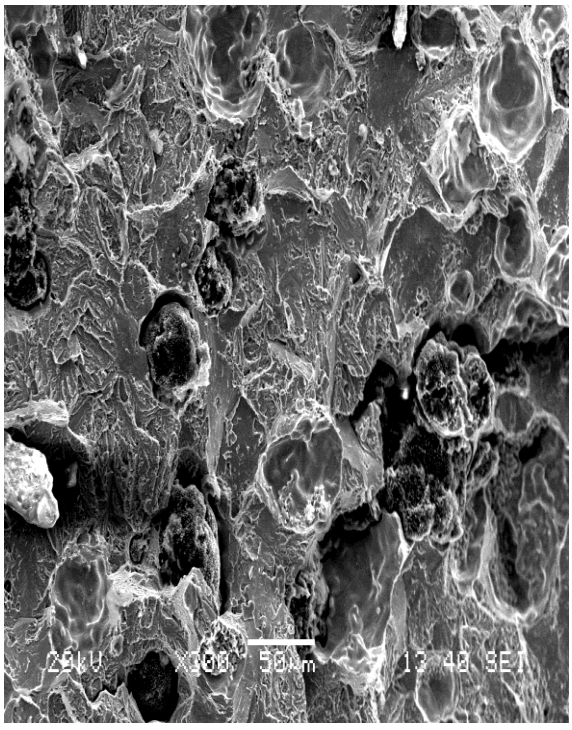


Figure 4.4.4: Fracture surface of Specimen, Tempered at 300°C, held 2hr.

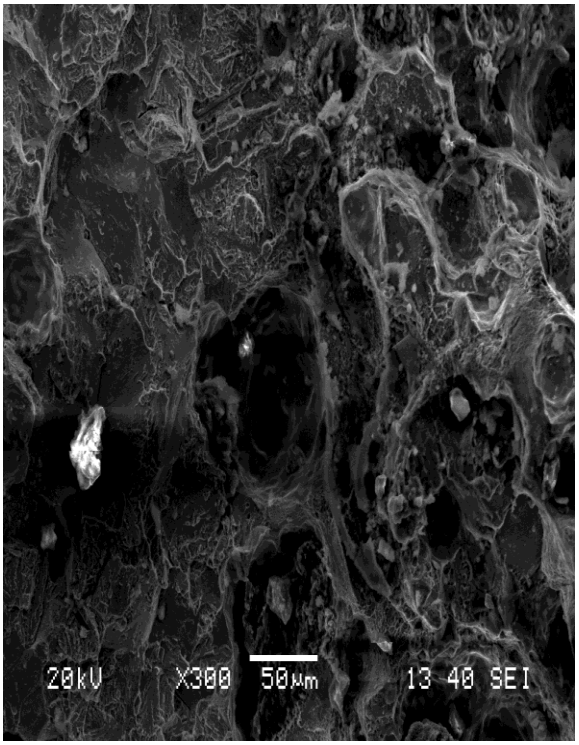


Figure 4.4.5: Fracture surface of Specimen, Tempered at 400°C and held 1hr.

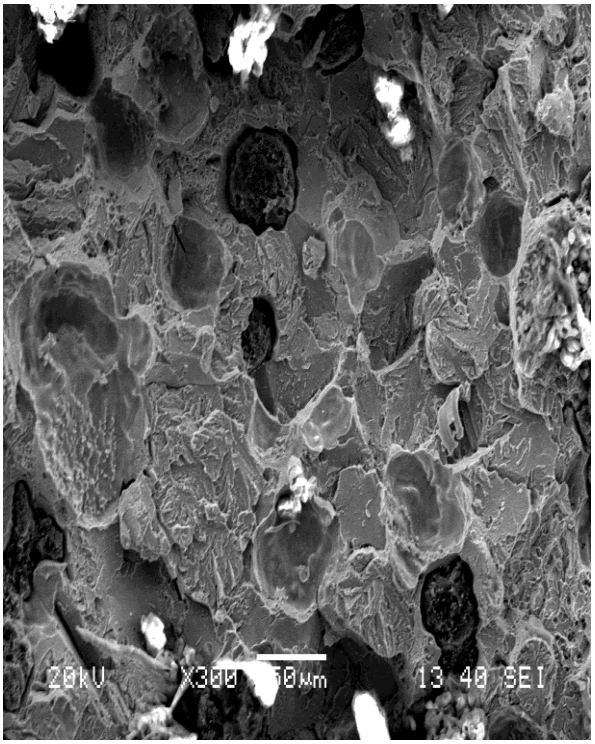


Figure 4.4.6: Fracture surface of Specimen, Tempered at 400°C and held

Each Fracture surface of the specimens was under gone ESM study and river marking were visualized in every specimen.

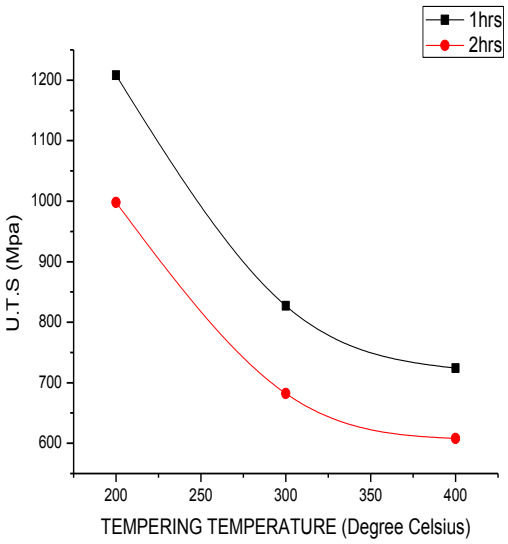
4.5. Effect of heat treatment on mechanical properties

The effect of hardening and tempering heat treatment on the mechanical properties such as ultimate tensile strength, yield strength, percentage elongation, hardness and impact energy of the treated samples is shown in table 4.5.

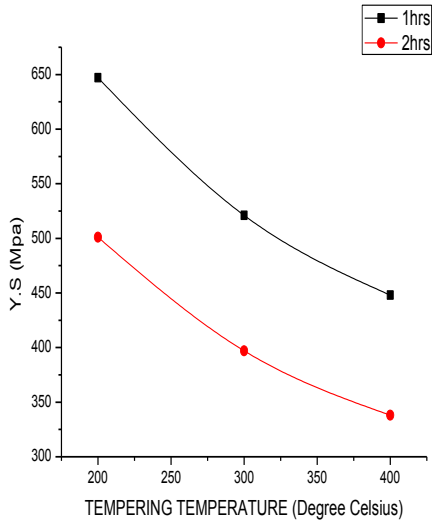
Table-4.5:- mechanical properties of treated ductile iron.

Sample No.	U.T.S (Mpa)	Y.S (Mpa)	Elongation (%)	Hardness (HV)	Impact Energy (Joule)
T2001	1208	647	8.35	429.1	6.167
T2002	998	501	9.19	363.4	9.903
T3001	827	521	9.45	403.3	10.152
T3002	682	397	10.55	351.4	16.152
T4001	724	448	11.59	400.4	17.875
T4002	608	384	13.41	291.2	20.137

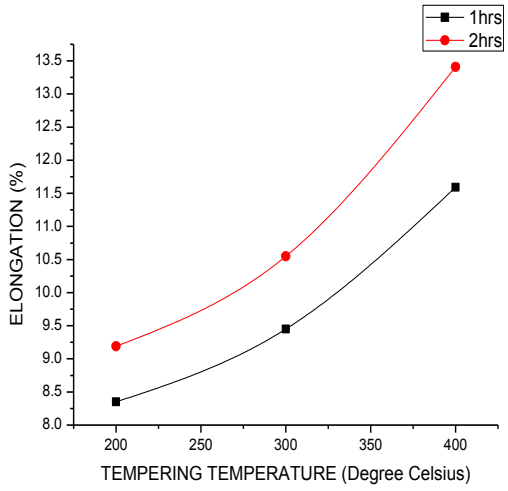
PLOTS OF MECHANICAL PROPERTIES



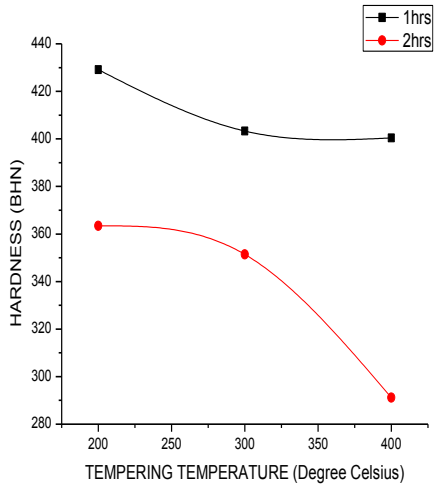
Graph 4.5.1: U.T.S v/s Tempering Temperature.



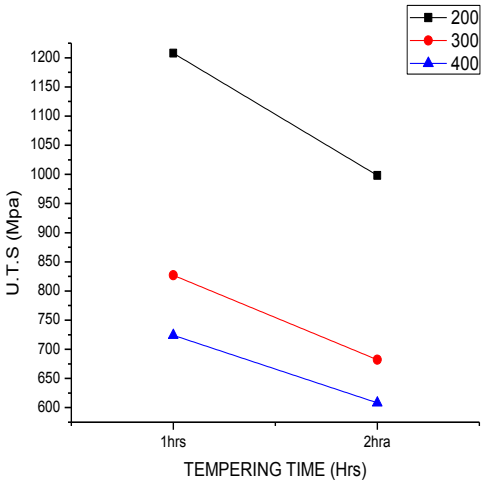
Graph 4.5.2: Y.S v/s Tempering Temperature.



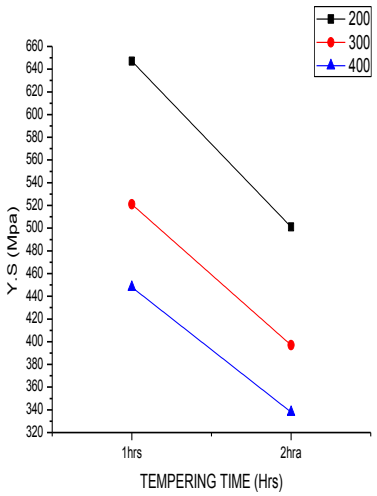
Graph 4.5.3: Elongation v/s Tempering Temperature.



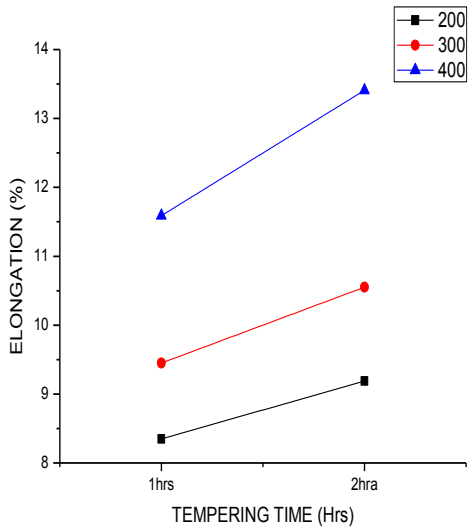
Graph 4.5.4: Hardness v/s Tempering Temperature.



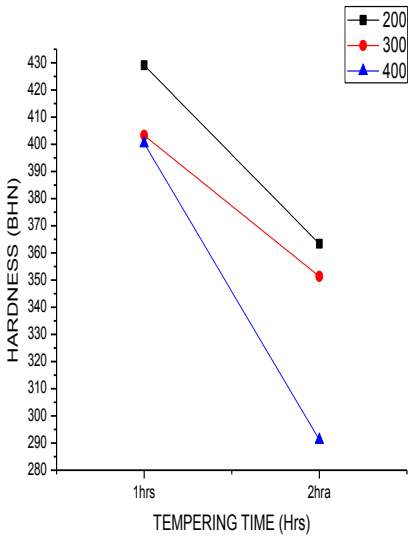
Graph 4.5.5: U.T.S v/s Tempering Time.



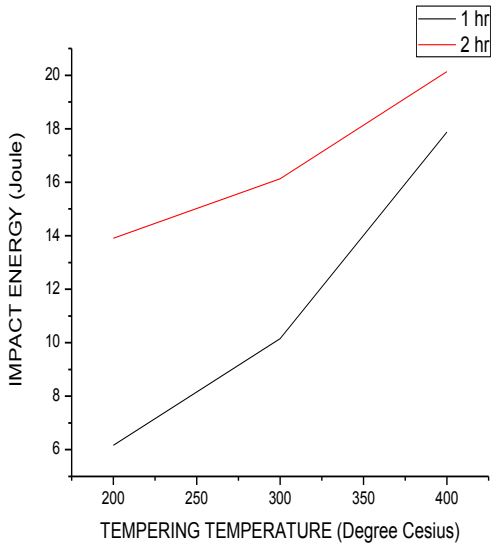
Graph 4.5.6: Y.S v/s Tempering Time.



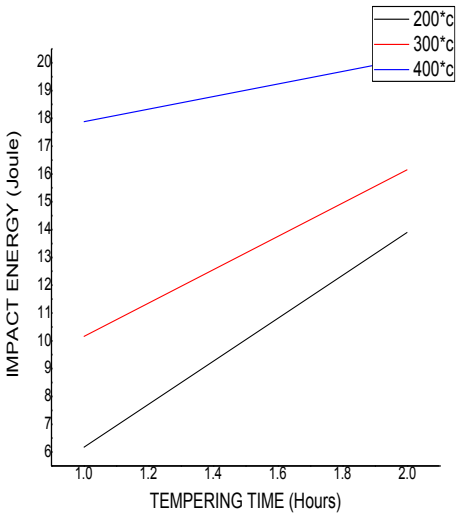
Graph 4.5.7: Elongation v/s Tempering Time.



Graph 4.5.8: Hardness v/s Tempering Time.



Graph 4.5.9: Impact energy v/s Tempering Temperature.



Graph 4.5.10: Impact energy v/s Tempering Time.

It was observed from graph 4.5.1, that U.T.S value decreased with increasing tempering temperature. It was found that 40% and 12.5% U.T.S value decreased at tempering temperature 400°C as compared to 200°C and 300°C. Similar trend was observed for 0.2% Y.S, 30.75% and 14%, from graph 4.5.2 and hardness, 19.86% and 17.13%, from graph 4.5.3. However, elongation value, 46% and 27%, from graph 4.5.4 and Izod impact energy, 44.87% and 28.66%, from graph 4.5.9 increased with increasing tempering temperature.

It was also observed from graph 4.5.5 that U.T.S value decreased with increasing tempering time.it was also found that 17.38%, 17.53% and 16% U.T.S value decreased for 2hrs as compared to 1hrs at 200°C, 300°C and 400°C respectively. Similar course was observed for 0.2%YS, 22.56%, 23.8% and 14.28%, from graph 4.5.6 and hardness, 15.3%, 12.86% and 27.21%, from graph 4.5.8. However, elongation value and impact energy, 10%, 10.42% and 15.7%, from graph 4.5.7 and 40.39%, 59.10% and 17.6%, from graph 4.5.10 increased with increase tempering time respectively.

4.6. Discussion

Maximum UTS value of 1208Mpa was obtained for T2001 with ductility 8.35% is due to the presence of hard martensitic phase, although for specimen T2002 there was no significant change in microstructure. There is decreased in UTS and increased in ductility. The reason behind this is due to the effect of longer tempering time. This leads to reduction of martensite area fraction. The decrease in hardness as well as increase in impact energy for the specimen T2002 is due to the fact mentioned above. As mentioned earlier, when tempered at 300°C and 400°C the martensite was transformed to tempered martensite, which is softer than martensite and residual stress developed during quenching is removed due to tempering. The UTS and hardness value for the specimen tempered at 300°C and 400°C decreases and consequently increase in elongation and impact energy was obtained. Further when tempered for 2 hour the area fraction of tempered martensite as well as nodularity increases, leading to increases in ductility and impact energy. The fracture surface of respective specimen after tensile test observed under SEM. Brittle characteristic of fracture i.e. presence of river marking were found in every specimen.

Chapter-5
Conclusions

CONCLUSIONS

- Elongation and impact energy increased with increasing tempering temperature, but decreased in hardness and tensile strength.
- Elongation and impact energy increased with increasing tempering time, but compromised with hardness and tensile strength.
- The brittle fracture confirmed by river marking or cleavage obtains at lower tempering temperature and the ductile fracture confirmed by dimples or dimple rupture obtains at higher tempering temperature. This suggests that fractographic analysis can help in correlating structure and property.
- For higher tempering time (particularly at high temperature) can gets ample time to diffuse out the structure i.e. martensite. This releases the residual strain and makes the structure more ductility.

Chapter-6

Future scope

FUTURE SCOPE

Tempered ductile iron have desirable mechanical properties like hardness, tensile strength, elongation and impact energy and damping capacity, which are most used in different structural applications. The need of ductile iron in various such as agriculture, auto-motive parts, structural and many more applications is increasing continuously. Every application has specific mechanical property and morphology aspect, which can be achieved by opting different heat treatment processes, hence in order to meet the market demand more work to be carried out to improve the properties of ductile iron.

Chapter-7

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