Role of Cooling Rate and Isothermal Annealing Temperature on Ductile Iron's Mechanical Characteristics

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ABSTRACT

A known composition block of ductile iron was ordered and samples were cut out of the block for multiple analyses. The ASTM E8 (Flat sub-size specimen) standard was implemented in the manufacturing of the block for tensile test specimens. Six of the nine instances in the collection experienced an isothermal heating procedure at varying cooling rates, while the remainder of three remains untreated. For ninety minutes, each one of the six specimens had been raised isothermally to 1000°C. Three of the six specimens were cooled to room temperature after having immediately refrigerated to 700°C after 90 minutes and kept there for 330 minutes. After 90 minutes, the rest of the three were subsequently cooled to room temperature. Within the furnace, all six specimens were cooled. The INSTRON 1195 UTM was used to conduct a tensile test at room temperature with a crosshead speed of 1 mm/min. Following the tensile test, the fractured specimens were sliced into tiny pieces so that the fracture surface could be examined. Vickers hardness testers had been utilized to record hardness values, as well as a 20 kg force was applied at room temperature. Phase analysis has been performed using the Philips PANalytica x-ray diffractometer and the X-ray diffraction technique. The yield strength, elongation, and hardness data for the "as-cast" and both annealed samples were plotted on graphs.

Keywords: Ductile Iron, tensile testing, X-ray diffraction, SEM.

I. INTRODUCTION :-

The term "cast iron" describes a group of materials primarily made up of iron, along with significant amounts of carbon and silicon. The carbon content in cast iron typically falls between 2 and 6.67%. Cast iron encompasses a variety of materials, each with unique properties determined by their microstructure - the phases formed during heat treatment or solidification[1]. Its high carbon content makes it brittle. Through careful alloying, heat treatment, and foundry practices, the properties of cast iron can be adjusted extensively. The main components of cast irons are the different forms of carbon, both morphological and chemical.

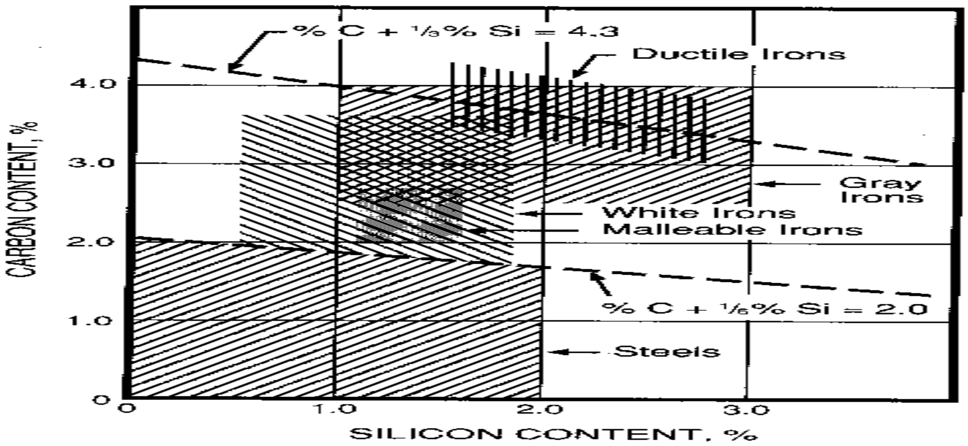


Fig 1: Variation of silicon content and type of cast iron

The Discovery of Ductile Iron The search for an ideal cast iron, one that possessed mechanical properties equal or superior to Malleable Iron, led to the groundbreaking discovery of Ductile Iron. Traditionally, cast iron was composed of graphite flakes, which limited its ductility and tensile strength. However, a significant breakthrough occurred in 1943 at the International Nickel Co. Research Lab, where metallurgist Keith Millis discovered a revolutionary process. Millis found that by adding a small amount of magnesium (typically in the form of a copper-magnesium alloy) to the molten cast iron, the solidified cast pieces would no longer contain the typical graphite flakes. Instead, the graphite would form into nearly spherical shapes, dramatically improving the material's ductility and tensile strength. This modified form of cast iron, which became known as Ductile Iron or Spheroidal Graphite (S.G.) Iron, represented a major advancement in the field of metallurgy. The discovery of Ductile Iron was a game-changer, as it allowed for the production of cast iron components with enhanced mechanical properties, making them suitable for a wide range of applications that previously required more expensive and difficult-to-manufacture materials. This breakthrough paved the way for the widespread use of Ductile Iron in various industries, including automotive, construction, and infrastructure development, where its superior strength, malleability, and cost-effectiveness proved invaluable. Today, Ductile Iron is widely recognized as a versatile and high-performance material, with applications ranging from heavy-duty machinery parts to intricate architectural elements. The discovery of this remarkable material, spearheaded by the pioneering work of Keith Millis and the researchers at the International Nickel Co., has had a lasting impact on the field of metallurgy and the manufacturing industry as a whole.

PRODUCTION OF DUCTILE IRON

Spheroidal graphite grows during solidification when cerium, magnesium, or their combination is added to molten alloy. Zr, Ba, Li, Ca, and other elements can also be applied here. Elements such as Ti, 0.009% Pb, Bi, or 0.004% Sb inhibit the formation of ductile iron; however, their influence is eliminated by adding 0.005-0.01% Ce. After adding magnesium to achieve a residual content of approximately 0.06%, ferro-silicon is added. To create ductile iron, a mixture of magnesium and cerium may also be utilized, followed by the addition of ferro silicon.

- **Desulfurisation:-** Sulfur is what leads to the creation of graphite flakes. Therefore, the raw materials sulfur content should be minimal (<0.10%). Sulfur can be eliminated either by melting the material or by adding a desulfurizing chemical such as calcium carbide or soda ash ($NaNO3$).
- **Nodulising:-** In order to eliminate the remaining sulfur and oxygen in the liquid alloy, magnesium is added, leaving a residual 0.05% of magnesium. This leads to the formation of spheroidal graphite, and it is likely that the high interfacial energy—which has a 180-degree angle—implies that graphite is not wetted.Less than 0.02% of iron is desulphurized after this magnesium treatment. Due to the notable affinity that magnesium and other comparable elements have for sulfur, they scavenge sulfur from the molten alloy as a first step in the production of Ductile Iron.
- **Inoculation:-** Since ferrosilicon is a carbide forming and magnesium is present, it is introduced right away as an innoculant. Remelting results in a return to graphite flake because of the loss of magnesium. Large amounts of gas are evolved during molten alloy stirring after the nodularising ingredient is applied. This gas can dissolve into the liquid alloy, forming solid casting blow-holes.

Properties of Ductile Iron:-

- **Tensile Property:-** Elongation, tensile and yield strengths, and other tensile characteristics of ductile/S.G. iron are typically referred to as mechanical behavior parameters. Numerous global specifications for Ductile/S.G. Iron use their corresponding tensile and yield strength values, as well as their elongation behavior, to show the qualities of the various grades of Ductile/S.G. Iron. The impact properties are also provided, but the hardness values are only given for a few ferritic grades. For designing reasons, other characteristics such as the modulus of elasticity and the proportional limit are also regarded as essential.

- **Modulus of Elasticity:-** Stress and strain are shown to have a proportionate or linear relationship at low tensile stress values. The value of the slope of the straight line that results is known as the Young's modulus, also known as the modulus of elasticity, and this relationship is known as Hooke's law. [8]In the early phases, Ductile/S.G. Iron's stress-strain behavior falls between that of mild steel and grey iron. Mild steels that have been normalized or annealed exhibit elasticity up until the yield point, but plastic deformation occurs abruptly and without any indication of a flow stress increase beforehand.

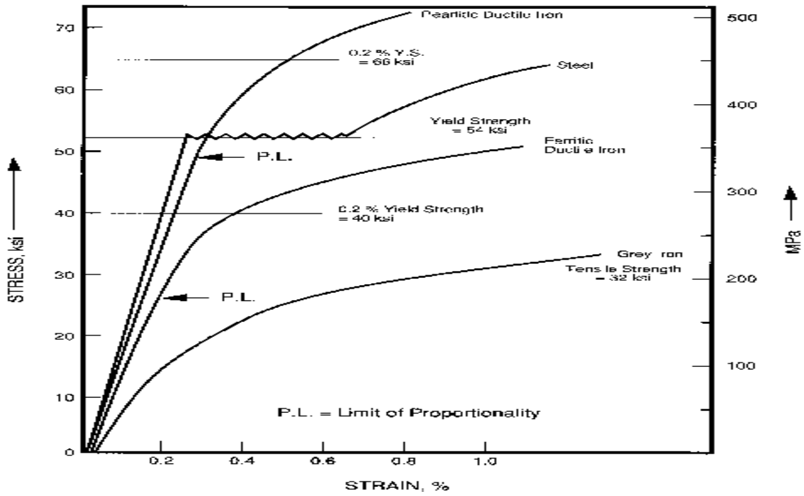


Fig 2: Elastic and yielding behavior for steel, Gray Iron and ferritic and pearlitic Ductile Irons

- **Poisson's Ratio:-** In the case of Ductile Iron, the Poisson Ratio—which is the ratio between the lateral and longitudinal elastic strains observed in a tensile test—shows relatively little variability. A widely recognized value is 0.275.

- **Proportional Limit:-** The highest stress level at which a material exhibits elastic behavior is known as the proportional limit, also referred to as the "limit of proportionality." The stress-strain curve returns to the origin when the material is stressed below the proportionality limit and the stress is subsequently released, indicating that there is no irreversible change in dimension. The plastic strain causes the stress-strain curve's slope to decrease as the applied stress exceeds the proportionality limit. When the applied tension is removed, the strain value decreases linearly and traces a line that is parallel to the elastic curve that was previously displayed. There is a permanent plastic strain, or a permanent change in the specimen dimension, when there is no applied stress because the strain does not decrease to zero.

- **Yield Strength:-** The stress value at which a material starts to exhibit noticeable plastic deformation is known as the proof stress or yield strength value. The yield strength of annealed and normalized steels may be almost precisely determined due to their quick change from elastic to plastic behavior. The offset method is applied to Ductile/S.G. Iron, wherein the yield strength is measured at a pre-established divergence from the initial linear relationship between stress and strain. The term "0.3% yield strength" is frequently used to refer to this variance, which is typically accepted to be 0.3%, and is also included when describing the yield strength in international specifications. Yield strengths for ductile iron can vary from 270 MPa for ferritic grades to more than 615 MPa for martensitic grades[8].

- **Tensile Strength: -** The highest load that a material can bear in tension before breaking is known as the tensile strength, or ultimate tensile strength (UTS). It is calculated by dividing the sample's initial cross-sectional area by the greatest load applied during the tensile test. The typical range of ductile iron tensile strengths is 410 MPa for ferritic grades and over 1370 MPa for martensitic grades[7].

- **Elongation: -** The permanent increase in length known as elongation is indicated in a tensile testing bar during failure testing and is stated as a percentage of the prescribed gage length. Elongation is a measure of tensile ductility and is a component of several ductile iron requirements. It include the localized deformation that happens before the fracture as well. Because the localized deformation only affects a small portion of the gage length, it has relatively little effect on the test bar's overall elongation value. While ferritic Ductile/S.G.

Irons can exhibit elongation of approximately 26%, brittle materials like gray iron may fail in stress without exhibiting any discernible elongation at all[9].

EFFECT OF VARIOUS PARAMETERS ON PROPERTIES OF DUCTILE IRON

- **Effect of Graphite Shape: -** That modularity is a major factor in defining qualities within the Ductile Iron family, as would be expected given the stark variations in mechanical properties between Gray and Ductile Irons. Dynamic Elastic Modulus and modularity are correlated.[6] This relationship shows that modularity has a significant impact on DEM and that modularity may be measured using DEM measurements from sonic testing (graphite volume and nodule count should be generally consistent). The yield and tensile strengths of Ductile Iron modularity are significantly influenced by nodularity and the shape of the non-spherical particles generated when modularity declines. Lead control or magnesium control are the two ways in which modularity can be modified. The nodules extend and do not become sharp or "spiky" when the nodularity is reduced by the chemical used in commercial ductile iron[2]. This results in a 15% reduction in tensile strength and a 10% drop in yield strength, with a 30% reduction in modularity. Tiny amounts of lead reduce modularity by forming "spiky" or plate-like intergranular networks in graphite, which drastically reduces the material's tensile qualities[14].
- **Effect of Nodule Count:-**
  Nodule count, which is defined mathematically as the number of graphite nodules/MM2, influences the mechanical properties of Ductile Iron, albeit not as strongly and directly as graphite shape. Generally speaking, a high nodule count indicates good metallurgical quality, though there is an optimal range for every casting and a large proportion of it may result in a degradation of properties[13]. Nodule count in and of itself has a mild effect on tensile properties, although it affects the microstructure, which can greatly influence properties[3]. Nodule count has an inverse relationship with the pearlite content of as-cast Ductile Iron; increasing the nodule count decreases the pearlite content, decreasing strength and enlarging elongation. Nodule count also affects carbide content. Decreasing the volume percentages of chill carbides, separated carbides, and carbides associated with "inverse chill" is how increasing the nodule count improves tensile strength, ductility, and machinability[4].

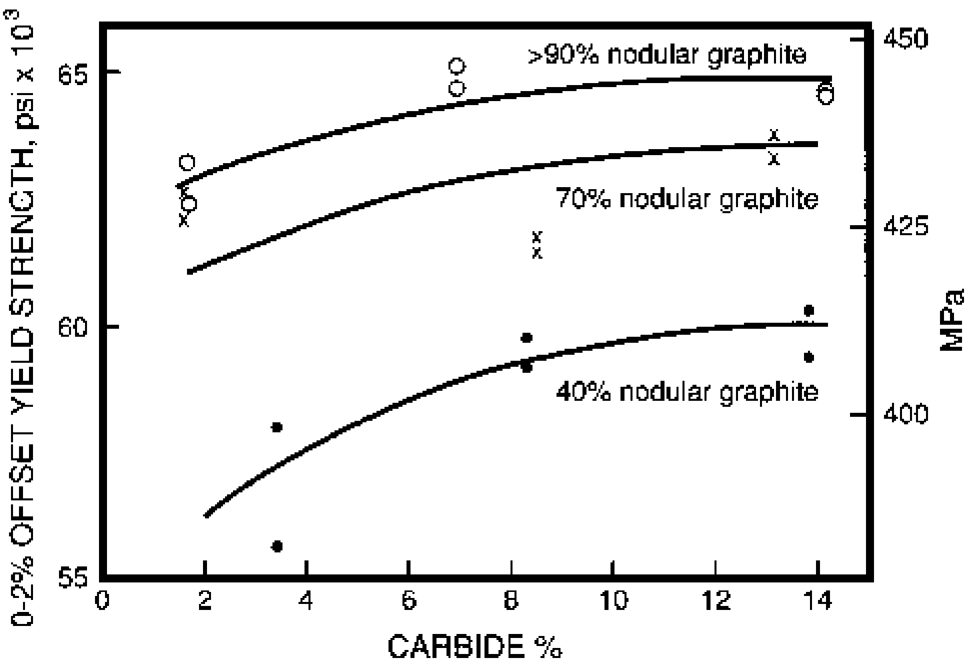


Fig 3: Relationship between tensile strength and hardness of Ductile Iron.

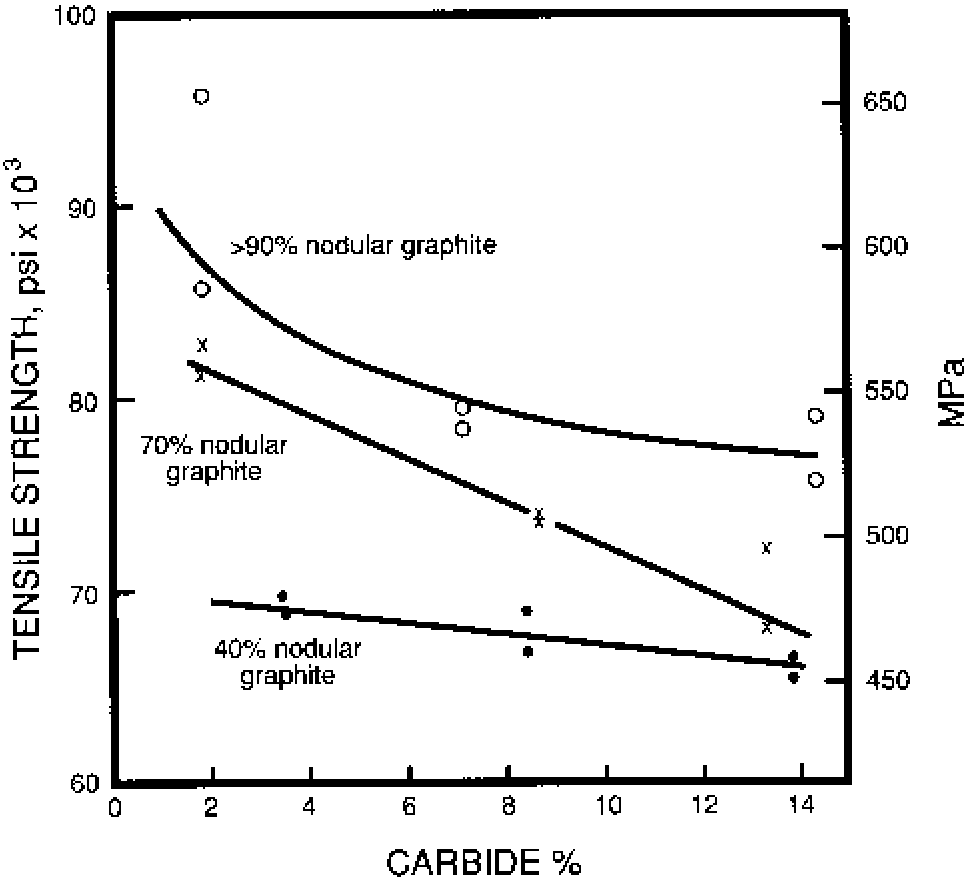


Fig 4: Effect of nodularity and carbide content on tensile strength of pearlitic Ductile Iron.

- **Effect of Graphite Volume:-**
  A portion of the tensile characteristics of S.G. Iron are influenced by the graphite volume fraction. For a given section size, the DEM decreases as the volume fraction of graphite increases due to an increase in carbon content. The graphite nodule size and volume fraction are both changed by the casting section size[12]. A larger section size reduces the cast's cooling rate, which leads to the precipitation of carbon in the stable graphite phase rather than the carbide phase that is produced by faster cooling rates. Reduced nodule count but increased nodule size is a result of the higher diameter bars' decreased cooling rates, which also have an impact on the graphite nucleating environment[15]. The primary cause of the decreased DEM is an increase in nodule size that is comparable to section size, but an increase in graphitic carbon production during solidification would also contribute to it[5].

- **Effect of Carbide Content:-**
  Properties are either directly or indirectly impacted by carbide content. Gray iron castings' yield strength increases when the volume fraction of hard, brittle carbide is increased, but their tensile strength decreases. Carbides that are present in a matrix of ductile iron also improve the dynamic elastic modulus and significantly reduce machinability. As carbide and graphite compete for the carbon in the liquid iron, the accumulation of eutectic carbide during solidification affects the volume proportion of graphite produced[9].

- **Effect of Temperature:-**
  The temperature range in which the component will operate as well as the effect of temperature on tensile behavior must be known by the designer when choosing design stresses for a Ductile Iron section. For both ferritic and pearlitic materials, Ductile Irons suggests that complicated design stresses be applied at low temperatures in order to increase yield strength with decreasing temperature[10]. Since room temperature

performance is a part of maximum low temperature applications, design stresses should be determined using the room temperature yield strength. However, low temperature applications should only use a yield strength-related design stress if a quasi-static test with a low strain rate can imitate the applied stress state[11].

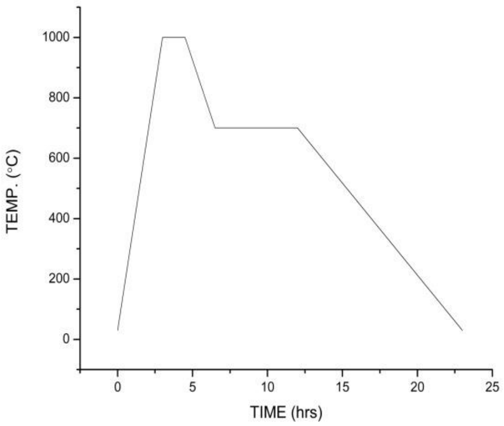
II. EXPERIMENTAL WORK

- **Experimental procedure:-**
  The ASTM E8 (Flat sub-size specimen) standard was followed in the machining of tensile test specimens from a test block. C-3.44%, Si-2.06%, Mn-0.14%, S-0.007%, P-0.023%, Cr-0.02%, Ni-0.14%, and Mg-0.042% make up the alloy used in this investigation. Six of the nine specimens in the collection underwent isothermal annealing at varying cooling speeds, while the remaining three were left untreated.

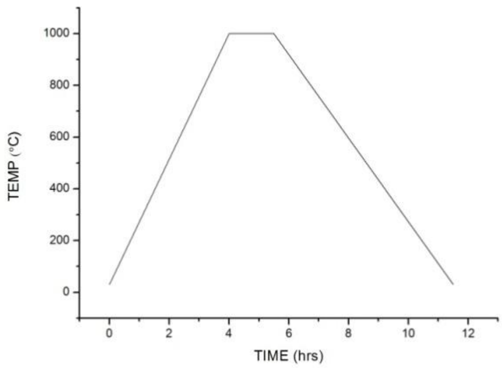
Element	Percentage(wt%)
C	3.44%
Si	2.06%
Mn	0.14%
S	0.007%
P	0.023%
Cr	0.02%
Ni	0.14%
Mg	0.042%

Table2.1: Chemical Composition used for the study

- **Heat Treatment:-** Isothermally heated to 1000°C, all six specimens were held there for ninety minutes. Three of the six specimens were promptly cooled to 700°C and kept there for 330 minutes. & then allowing it to cool to room temperature. After 90 minutes, the remaining three were promptly cooled to room temperature. Within the furnace, all six specimens were cooled. Figures 1 and 2 depict the two heat treatment procedures, respectively.



(a)



(b)

Fig:2.1 (a) – heating the specimen to 1000°C and holding there for 90 mins, cooling to 700°C, holding there for 330 mins followed by furnace cooling.
(b) – heating the specimen to 1000°C and holding there for 90 mins followed by furnace cooling.

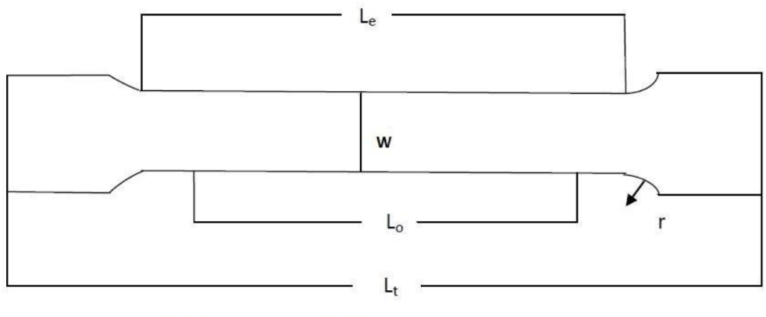
➢ **Tensile test :-**

Tensile test was performed at room temperature using INSTRON 1195 UTM at a crosshead speed of 1mm/min. Using an electronic slide caliper the thickness and the total length of the specimen was measured. The diameter of the specimen and the gauge length which was fixed at 50mm was fed to the testing machine. The distance between the jaws was fixed according to the gauge length of the specimen. The specimen was gripped by the jaws and axial load was applied to it. Ultimate tensile strength, 0.2% yield strength was obtained directly from the computer integrated to the m/c, after completion of test. Percentage elongation was calculated by dividing the displacement at break to the gauge length. hardness values.

- **Specification of tensile testing specimen**:

  The tensile testing specimen as per the Indian Standard is collected.

  The specification is as per the ISO 1608: 1995 ANNEX C.



Lt = overall length =101 mm
w = width of the parallel section =6.30 mm
r = fillet radius =6.30 mm
Length of grip = 31.70 mm
Width of grip = 9.520 mm
Lo = gauge length = 25.3 mm

- **Fractography:-**

  After completion of tensile test the broken specimens were cut down to small pieces to study the fracture surface. The fractured area for all the three types of specimen was put under scanning electron microscope (SEM) and was analyzed at 50, 100, 250 & 500 magnification levels. The SEM micrographs are shown.

- **Hardness test:-** Hardness values were recorded from Vickers hardness tester by application of 20Kg load at room temperature. The average of five readings for each type of specimen is reported.
- **XRD analysis:** X-ray diffraction technique was employed for phase analysis with Philips PANalytica x-ray diffractometer. Test was conducted at 30KV, 20mA, at a scanning rate of 2° per minute for scanning range of 40°-90°. The diffraction patterns were analyzed using Xpert Highscore and JCPDS and the results are shown.

III Result and discussion

- **Mechanical properties:**

  Table 3.1 displays UTS, 0 point 2 percent YS, percentage elongation, and hardness for the test specimens. In comparison to the as cast and the specimen hold for 90 minutes at 1000°C, it is evident that for the specimen hold at 700°C (AN 1), the tensile strength and hardness are decreased, while ductility is slightly increased (185%). This phenomenon was seen because specimen AN 1 was held at 1000°C for a longer period of time than specimen AN 2, which was only held for 90 minutes. for the specimen that had been brought to room temperature. UTS and hardness are discovered to be greater than those of the as-cast specimen but lower than those of the specimen that was held at 700°C for 330 minutes after 90 minutes (AN 2). Although AN 2's percentage elongation has somewhat increased, it is still far lower than AN 1's. When AN 1 and AN 2 are compared to the as cast sample, the yield strength of the former has increased, but more so in the latter case.

Table:3.1 Mechanical Properties of as cast & annealed specimen

SPECIMEN	UTS (MPa)	0.2% YS (MPa)	% El	HARDNESS
AS CAST	**520**	**192.67**	**18.80**	217 HV20
AN 1	**349.9**	**198.4**	**35.12**	124 HV20
AN 2	**429.4**	**227.9**	**19.11**	153.2 HV20

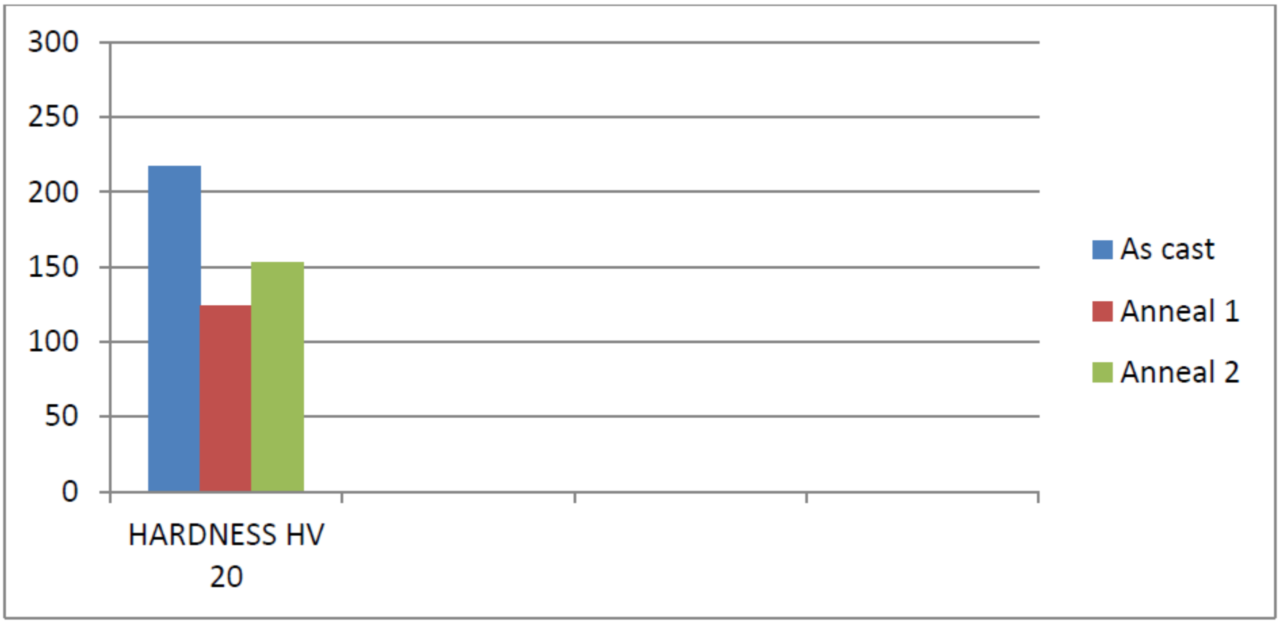


Fig3.1 : Hardness of as cast and annealed specimen

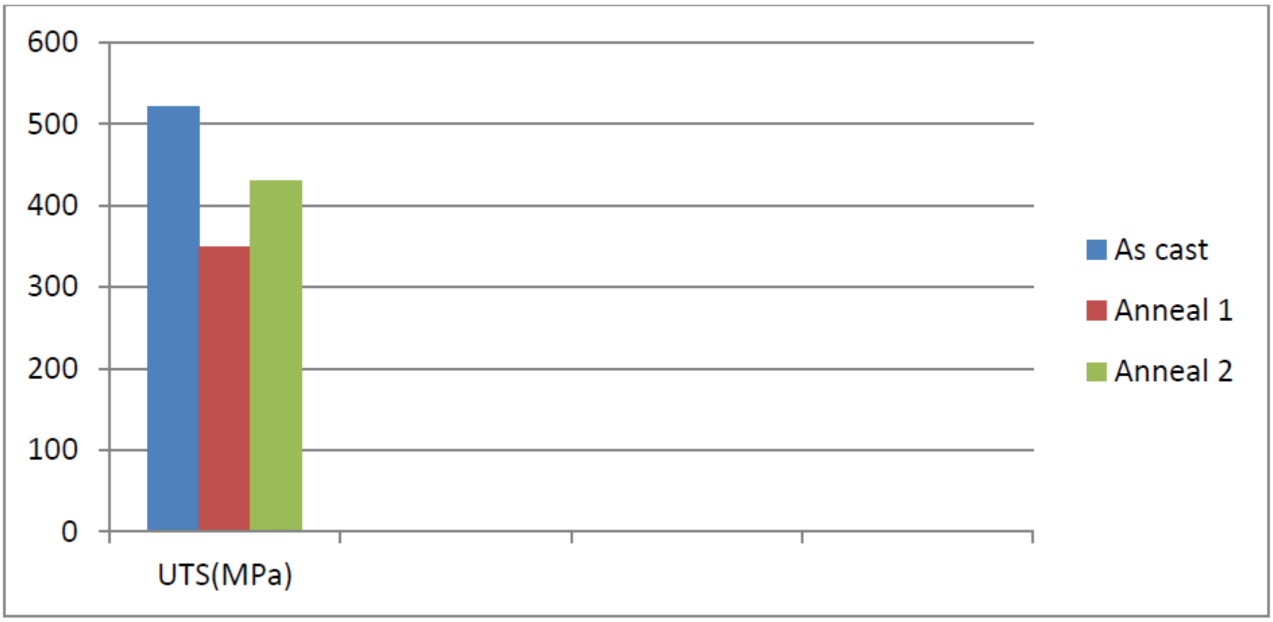


Fig3.2 : UTS of as cast and annealed specimen

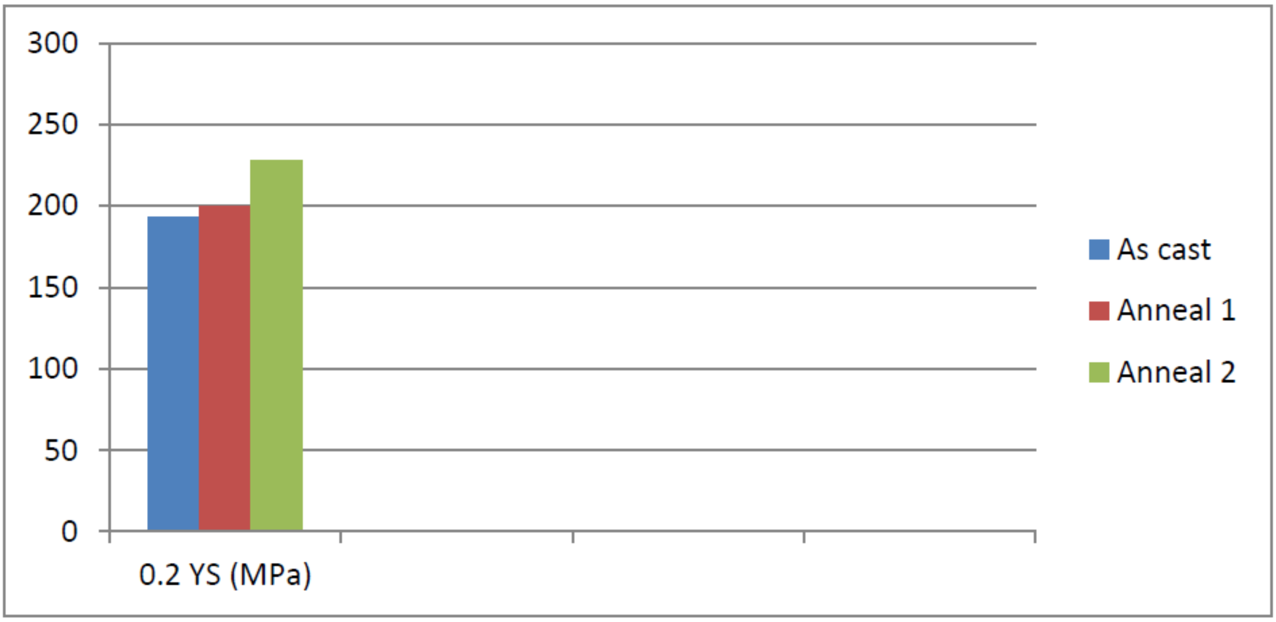


Fig3.3 : Yield strength of as cast and annealed specimen

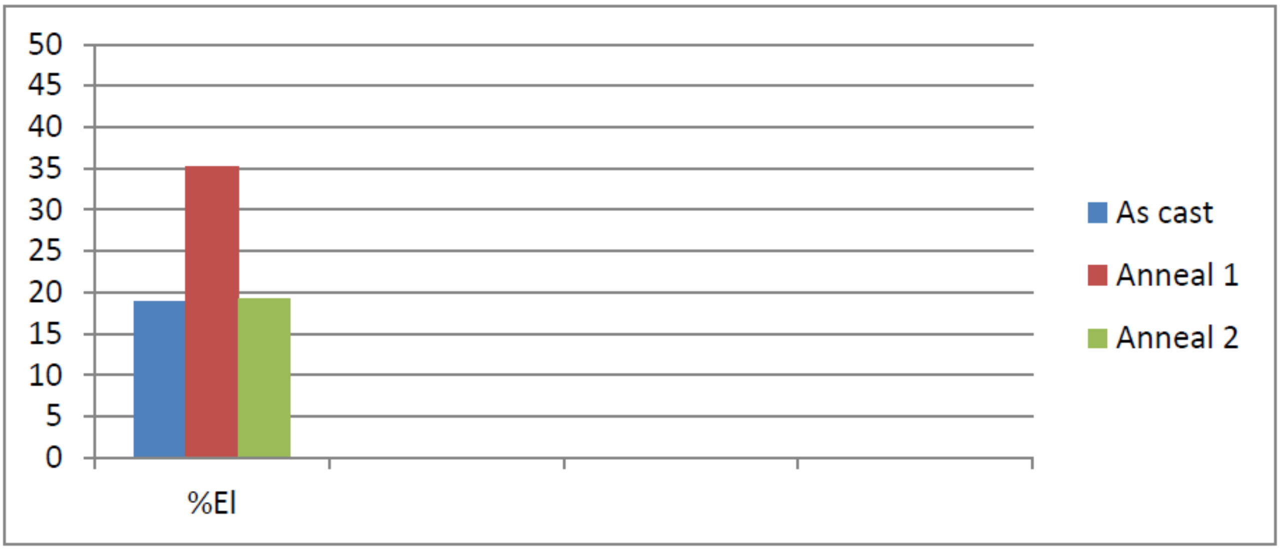


Fig 3.4 : Elongation of as cast and annealed specimen

Fractography: Figure displays the SEM images of the treated and cast specimens. Every specimen has uniformly dispersed graphite nodules throughout the ferrite matrix when examined under a 49.9X magnification level. Each treated sample had a dimple-type fracture mechanism visible when examined closely at 249.9X magnification. On the other hand, it was discovered that the fracture surface of the cast sample was fragile. This could be caused by uneven cooling during casting in relation to the geometry of the specimen, which would leave residual stresses in the matrix. However, both of the annealed specimens exhibit improved matrix homogenization, which leads to ductile fracture.

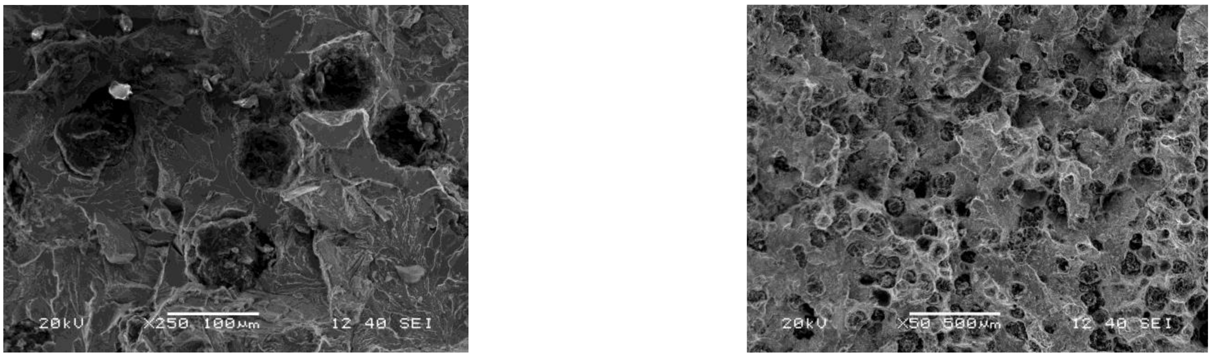


Fig 3.5. (a): AS-CAST Specimen at 49.9X & 249.9X

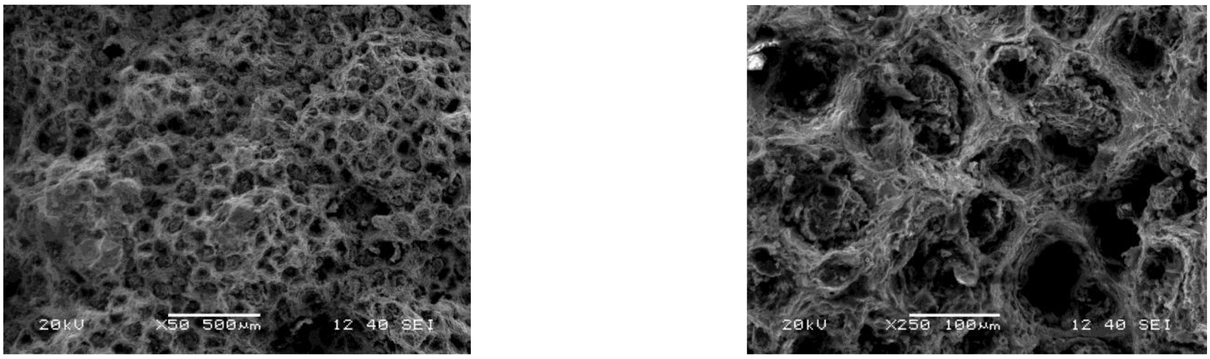


Fig 3.5. (b): AN 1 Specimen at 49.9X & 249.9X

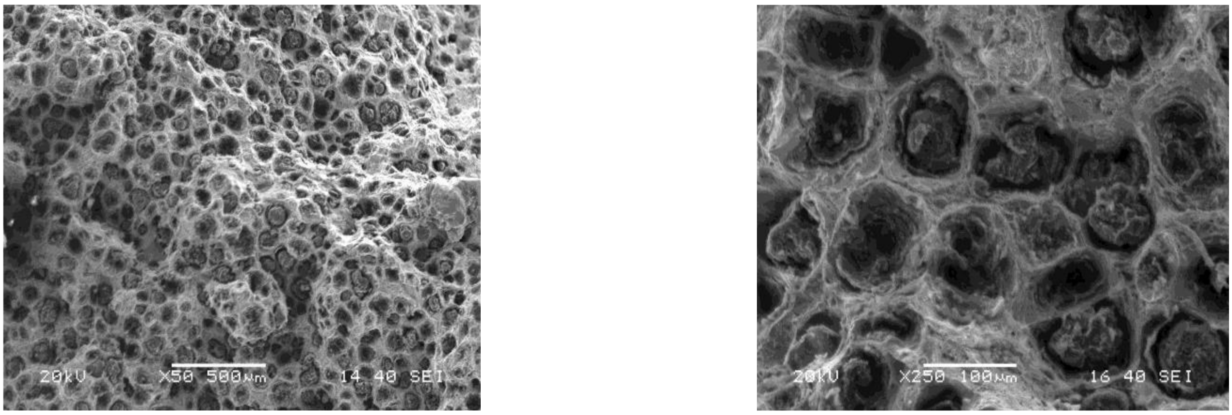


Fig 3.5 (c): AN 2 Specimen at 49.9X & 249.9X

Figure 4 below displays the pattern created by XRD for each type of samples. It is evident that the cast specimen's (1 1 0) plane has a greater peak intensity than the AN 1 & AN 2 specimens. This could be the outcome of modifications to the atomic packing factor, planar density, or atom orientation. However, in the instance of the (2 0 0) plane, the cast specimen's intensity is lower than the AN 1 fragment and does not change as much as the specimen descended from 1000°C. The shifting of atoms from the (1 1 0) plane owing to the change in orientation may be the cause of the increase in peak intensity for the (2 0 0) plane. In the process of analyzing the XRD pattern using JCPDS software, ferrite phase was discovered for every category of material.

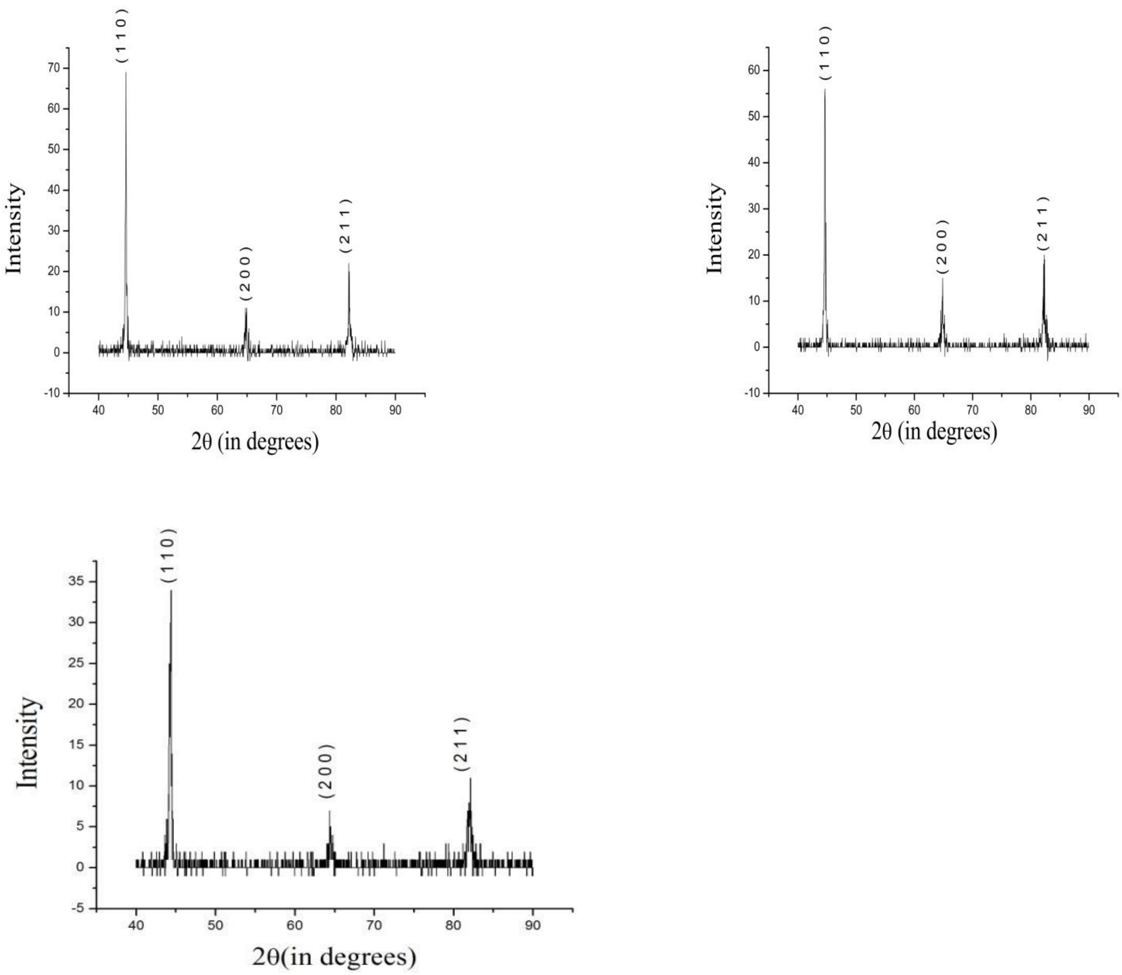


Fig 3.6. XRD Pattern; (a) AS-CAST Specimen, (b) AN 1 Specimen, (c) AN 2 Specimen

IV. CONCLUSION

1. Each treated sample had a dimple-type fracture mechanism visible when examined closely at 249.9X magnification. On the other hand, it was discovered that the fracture surface of the cast sample was fragile. This could be due to non-uniform cooling during casting in relation to the geometry of the specimen, which caused residual stresses to remain in the matrix. However, both of the annealed specimens exhibit improved matrix homogenization, which leads to ductile fracture.
2. It is discovered that the cast specimen's (1 1 0) plane has a greater peak intensity than the AN 1 & AN 2 specimens. This could be the outcome of modifications to the atomic packing factor, planar density, or atom orientation.
3. The intensity of the cast specimen in the case of the (2 0 0) plane is lower than that of the AN 1 specimen and doesn't differ as much as that of the item that was cooled from 1000°C. The shifting of atoms towards the (1 1 0) plane owing to the change in alignment may be the cause of the increase increased peak intensity for the (2 0 0) plane.
4. Anneal 1 has the highest ductility but the lowest yield strength and hardness value, according to observations of the hardness value, yield strength, and ductility.This is because homogeneity from isothermal holding and slower cooling rates in the anneal 1 sample resulted in the production of more ferrite phase.

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