

A Project Report on
**Study of Ductile to Brittle Transition Temperature of
Mild Steel**

Submitted in partial fulfillment of the requirements for the award of the degree of

**BACHELOR OF TECHNOLOGY
IN
MECHANICAL ENGINEERING**

BY

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DEPARTMENT OF MECHANICAL ENGINEERING
ADITYA COLLEGE OF ENGINEERING & TECHNOLOGY
(Permanently Affiliated to JNTUK, Approved by AICTE, Accredited by NAAC)

Recognized by UGC under section 2(f) and 12(B) of UGC Act 1956.

Surampalem, E.G. Dist., AP – 533 437.

(2019-2020)

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CERTIFICATE

This is to certify that the project report entitled “**Study of Ductile to Brittle Transition Temperature of Mild Steel**” Submitted by

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We further declare that this project work has not been submitted in full or part for the award of any degree of this or any other educational institutions.

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ABSTRACT

The Ductile-to-Brittle Transition Temperature (DBTT) is a phenomenon that can be widely observed in metals. Below critical temperature, the material suddenly loses its ductility and becomes brittle in nature. The controlling mechanism of this transition still remains unclear despite of large efforts made through experimentation. All ferrous materials (except the austenitic grades) exhibit a transition from ductile to brittle when tested above and below a certain temperature, called as Transition Temperature. The Project deals with the determination of the 'Ductile to Brittle Transition Temperature of Mild steel. Work carried out in this is purchasing the material followed by test specimen preparation. The specimens then keep in the liquid nitrogen for cooling for soaking time of 15 min and 30 min. Then the actual charpy impact testing of the specimens at variable temperature rangings are carried out in controlled atmosphere. The readings taken are the impact energy (joules) of specimen at specific temperature. The graph of energy absorbed vs temperature is plotted to get the range of transition temperature.

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Chapter 1

Introduction

1.1 Mild Steel

Mild steel is a category of carbon steel with a low carbon composition. It is also known as 'low carbon steel.' While amounts differ based on the source, the amount of carbon typically found in mild steel is between 0.05% to 0.25% by weight, whereas higher carbon steels are generally described as having a carbon content of between 0.30% and 2.0%. If more carbon is added, then it is called as Cast Iron.

Mild steel is not an alloy steel and therefore does not contain heavy mixture of other elements besides iron; in mild steel you will not find vast quantities of chromium, molybdenum, or other alloying elements. Since its content of carbon and alloy elements is relatively low, it has several properties which distinguish it from higher carbon and alloy steels.

Low carbon content makes mild steel more ductile, machinable, and weldable than high carbon and other steels but it also means that heating and quenching makes it almost impossible to harden and strengthen. The low carbon content also means that it has very little carbon and other alloying elements to block dislocations in its crystal structure. In particular, this results in less tensile strength than high carbon and steel alloys. Mild steel also has a high iron and ferrite content which makes it magnetic.

The lack of alloying elements, such as those used in stainless steels, means that if not properly treated, the iron in mild steel is susceptible to oxidation (rust). But the insignificant amount of alloying elements also helps to make mild steel relatively affordable as compared to other steels. The affordability, weldability and machinability make it such a common steel option for consumption. Increasing carbon content increases the hardness and strength and improves hardenability. But carbon also increases brittleness because of its tendency to form martensite. This

means carbon content can be both a blessing and a curse and its come to commercial steel.

1.2 Types of steels

Based on the chemical composition of Carbon and Iron, Carbon steels are classified into three types. They are namely:

1. High carbon steel
2. Medium carbon steel
3. Low carbon steel (Plain-carbon steel)

1.2.1 High Carbon Steel

High Carbon Steels are one of the largest groups of carbon steels. They cover a great diversity of shapes; from flat sheet to structural beam. Based on the properties needed, other elements are added or increased. Metallurgists described high carbon steel as being iron mixed with over 0.8 % carbon but less than 2.11 % carbon in its composition. The average level of carbon found in this metal usually falls right around the 1.5 % mark. High carbon steels has a reputation for being especially hard, but the extra carbon also makes it more brittle than other types of steel. This type of steel is the most likely to fracture under stress.

The steels successfully undergo the heat-treatment and have a carbon content ranging between 0.30 and 1.70% on average. Trace impurities from various other elements could have a big impact on the created steel's quality. Trace amounts particularly of sulphur will make the steel red. Low carbon steel alloy contains has 0.05% sulphur and a melting temperature of around 1538-1426°C. Manganese is usually added to better the of low carbon steel hardenability. The additions make the material a low alloy one by some definitions, and AISI's understanding of carbon

steel includes up to 1.65% on average. The higher ones have approximately 0.6 to 0.99% carbon content and very strong, utilized in high-strength wires and springs.

High carbon steel has important advantages over other materials. This type of steel is excellent for making cutting tools or masonry nails. The carbon gives the steel hardness and strength while being relatively inexpensive compared to other hard substances. Manufacturer's value high carbon steel for metal cutting tools or press machinery that bends and forms metal parts. Some disadvantages also come with the use of high carbon steel. It is difficult to weld, posing challenge for manufacturers and fabricators. The same quality of hardness that makes it preferred for cutting tools also means it is brittle, making it prone to fracture or break.

1.2.2 Medium Carbon Steel

Usually Medium Carbon Steels have a carbon range of 0.31% to 0.60%, and a manganese content ranging from .060% to 1.65%. These products are stronger than low carbon steels, and it is more difficult to form, weld and cut. Medium carbon steels are quite often hardened and tempered using heat treatment. Balancing ductility and strength, medium carbon steel and has fantastic wear resistance; it is used in forging and for large parts like automotive components.

1.2.3 Low Carbon Steel (Mild Steel)

Low Carbon Steels typically contain 0.04% to 0.30% carbon content. Undoubtedly, it is the commonest form of steel because its price is usually low, and it provides the material properties which are acceptable under many circumstances. The carbon level is kept low and Aluminum is added, and for Structural Steel the carbon level is higher and the manganese content is increased.

Low carbon steels contain less carbon than other steels and are easier to coldform, making them easier to handle. Mild steel is also known as plain-carbon steel and low

carbon steel, is one the most common form of steel because its price is relatively low while it provides material properties that are acceptable for many applications. It contains a small percentage of carbon, strong and tough but not readily tempered. Mild steel contains approximately 0.05-0.25% carbon making it malleable and ductile. The low carbon steel has approximately 0.05 to 0.15% carbon, so it is neither ductile nor brittle. It is normally used when huge quantities of steel are required, for instance in the form of structural steel. Mild steel density is around 7.85 g/cm³ (0.284 lb/in³ or 7850 kg/m³) and Young's modulus is 30,000,000 psi or 210,000 MPa. Mild steel is characterized by a low tensile strength practically speaking, but it is malleable and cheap; surface hardness may be increased with carburization.

1.3 Material Properties

To finalize the material for an engineering product or application, it is important to understand the mechanical properties of the material. The mechanical properties of a material are those which affect the mechanical strength and ability of a material to be molded in suitable shape. Some of the typical mechanical properties of a material include:

- Strength
- Toughness
- Hardness
- Hardenability
- Brittleness
- Malleability
- Ductility
- Creep and Slip
- Resilience
- Fatigue

1.3.1 Strength

It is the property of a material which opposes the deformation or breakdown of material in presence of external forces or load. Materials which we finalize for our engineering products, must have suitable mechanical strength to be capable to work under different mechanical forces or loads.

1.3.2 Toughness

It is the ability of a material to absorb the energy and gets plastically deformed without fracturing. Its numerical value is determined by the amount of energy per unit volume. Its unit is Joule/ m³. Value of toughness of a material can be determined by stress-strain characteristics of a material. For good toughness, materials should have good strength as well as ductility.

For example: brittle materials, having good strength but limited ductility are not tough enough. Conversely, materials having good ductility but low strength are also not tough enough. Therefore, to be tough, a material should be capable to withstand both high stress and strain.

1.3.3 Hardness

It is the ability of a material to resist to permanent shape change due to external stress. There are various measure of hardness – Scratch Hardness, Indentation Hardness and Rebound Hardness.

1. Scratch Hardness

Scratch Hardness is the ability of materials to the oppose the scratches to outer surface layer due to external force.

2. Indentation Hardness

It is the ability of materials to oppose the dent due to punch of external hard and sharp objects.

3. Rebound Hardness

Rebound hardness is also called as dynamic hardness. It is determined by the height of “bounce” of a diamond tipped hammer dropped from a fixed height on the material.

1.3.4 Hardenability

It is the ability of a material to attain the hardness by heat treatment processing. It is determined by the depth up to which the material becomes hard. The SI unit of hardenability is meter (similar to length). Hardenability of material is inversely proportional to the weldability of material.

1.3.5 Brittleness

Brittleness of a material indicates that how easily it gets fractured when it is subjected to a force or load. When a brittle material is subjected to a stress it observes very less energy and gets fractures without significant strain. Brittleness is converse to ductility of material. Brittleness of material is temperature dependent. Some metals which are ductile at normal temperature become brittle at low temperature.

1.3.6 Malleability

Malleability is a property of solid materials which indicates that how easily a material gets deformed under compressive stress. Malleability is often categorized by the ability of material to be formed in the form of a thin sheet by hammering or rolling. This mechanical property is an aspect of plasticity of material. Malleability of material is temperature dependent. With rise in temperature, the malleability of material increases.

1.3.7 Ductility

Ductility is a property of a solid material which indicates that how easily a material gets deformed under tensile stress. Ductility is often categorized by the ability of material to get stretched into a wire by pulling or drawing. This mechanical property is also an aspect of plasticity of material and is temperature dependent. With rise in temperature, the ductility of material increases.

1.3.8 Creep and Slip

Creep is the property of a material which indicates the tendency of material to move slowly and deform permanently under the influence of external mechanical stress. It results due to long time exposure to large external mechanical stress within limit of yielding. Creep is more severe in material that are subjected to heat for long time. Slip in material is a plane with high density of atoms.

1.3.9 Resilience

Resilience is the ability of material to absorb the energy when it is deformed elastically by applying stress and release the energy when stress is removed. Proof resilience is defined as the maximum energy that can be absorbed without permanent deformation. The modulus of resilience is defined as the maximum energy that can be absorbed per unit volume without permanent deformation. It can be determined by integrating the stress-strain curve from zero to elastic limit. Its unit is joule/m³.

1.3.10 Fatigue

Fatigue is the weakening of material caused by the repeated loading of the material. When a material is subjected to cyclic loading and loading greater than certain threshold value but much below the strength of material (ultimate tensile strength

limit or yield stress limit), microscopic cracks begin to form at grain boundaries and interfaces. Eventually the crack reaches to a critical size. This crack propagates suddenly, and the structure gets fractured. The shape of structure affects the fatigue very much. Square holes and sharp corners lead to elevated stresses where the fatigue crack initiates.

1.4 Fracture Toughness

Fracture is a process of breaking a solid into pieces as a result of stress. Toughness is a ability of material to resist fracture. In Material science, fracture toughness is a property which describes the ability of a material to resist the fracture, and is one of the most important properties of any material for many design applications. The linear-elastic fracture toughness of material is determined from the stress intensity factor (K) at which a thin crack in the material begins to grow. It is denoted K_{IC} .

Fracture toughness is a quantitative way of expressing a material resistance to brittle fracture when a crack is present. A material with high fracture toughness may undergo ductile fracture is as opposed to brittle fracture is characteristic of materials with low fracture toughness. It is an indication of the amount of stress required to propagate a pre-existing flaw. It is a very important material property since the occurrence of flaws is not completely avoidable in the processing, fabrication, or a service of material/component. Flaws may appear as cracks, voids, metallurgical inclusions, weld defects, design discontinuities, or some combination thereof since engineers can never be totally sure that the material is flaw free, it is common practice to assume that a flaw of some chosen size will be present in some number of components and use the linear elastic fracture mechanics (LEFM) approach

to design critical components. This approach uses the flaw size and features, component geometry, loading conditions and the material property called fracture toughness to evaluate the ability of a component containing a flaw to resist fracture.

1.5 Types of fractures

There are two types of fracture process they are:

- Ductile Fracture
- Brittle Fracture

1.5.1 Ductile Fracture

Ductile materials undergo observable plastic deformation and absorb significant energy before fracture. A crack formed as a result of the ductile fracture, propagate slowly and when the stress is increased.

Plastic deformation of a multiphase material cause's formation and coalescence of voids are responsible for the specific appearance of the ductile fracture surface, consisting of numerous spherical micro-cavities (**dimples**), initiating formation of the crack. Tensile specimen fractured by the ductile mechanism is characterized by the cup and cone appearance of the fracture. Single-phase alloys and pure metals are more ductile, than metals, containing second phases or inclusions.

1.5.2 Brittle Fracture

Brittle fracture is characterized by very low plastic deformation and low energy absorption prior to breaking. A crack, formed as a result of the brittle fracture, propagates fast and without increase of the stress applied to the material. The brittle crack is perpendicular to the stress direction. Cleavage cracks pass along crystallographic planes through the grains. Inter crystalline

fracture occurs through the grain boundaries embrittled by segregated impurities, second phase inclusions and other defects. The brittle fractures usually possess bright granular appearance.

Ductile vs. Brittle Failure

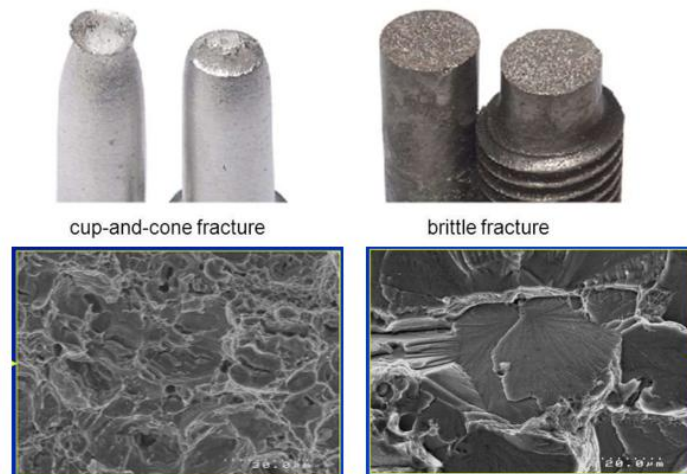


Fig. No.1.1. Types of Fractures.

1.6 Charpy impact Testing

The Charpy impact testing is also known as the Charpy V-notch test, is a standardized high strain-rate test which determines the amount of energy absorbed by a material during the fracture. This absorbed energy is a measure of a given material's notch toughness and acts as tool to study the temperature-dependent ductile-brittle translation. It is widely applied in industries, since it is to prepare and conduct and results can be obtained quickly and cheaply.

This Test was developed around 1900 by S.B. Russell (1898, American) and George Charpy (1901, French). The Test became known as Charpy test in early 1900s due to the technical contributions and standardization efforts by Charpy. The test was pivotal in understanding the fracture problems of ships during World War 2. Today it is utilized in many industries for testing the materials.

The Charpy test is a three point bend impact test. It requires a specimen containing a machined notch in the center of the face facing away from the impacting device and a sturdy machine that can impart a sudden load to the specimen. The Charpy tester consists of a heavy pendulum which is allowed to strike the specimen at the bottom of its arch (maximum kinetic energy, maximum velocity). As the specimen deforms and fractures a portion of the kinetic energy of the pendulum is transferred to the specimen. The specimen is broken and the two pieces of the fractured specimen are knocked clear of the testing machine while the pendulum continues its swing to a somewhat lower position than it was released from. The difference in these heights and the mass of the pendulum determines how much energy was absorbed by the specimen.

Most impact testers have a gage that reports this energy so that it doesn't have to be computed. Current applications of the Charpy impact test include comparisons of heat to heat variations of steel, evaluation of material behavior during either intentional or accidental high rates of loading, evaluation of the effect of irradiation on the embrittlement of steel, evaluation of the effects of microstructure and fabrication on toughness and studies of the fundamental aspects of deformation in bcc materials. Together the tensile test and Charpy impact test form a fairly complete evaluation of the mechanical properties of a material. However, it should be noted that the Charpy test is not a simulation of an alloy in service. The results of the Charpy tests are useful indications of how the material might behave in service.

The apparatus consists of a pendulum of known mass and length that is dropped from a known height to impact a notched specimen of a material.

The energy transferred to the material can be inferred by comparing the difference in the height of the hammer before and after the fracture. The notch in the sample affects the results of the impact test, thus it is necessary for the notch to be a regular dimensions and geometry. The size of the sample can also affects the results, since the dimensions determine whether or not the material is in plain stain. The difference can greatly affect the conclusion made.

The “Standard methods for the Notched Bar Impact Testing of Metallic Materials” can be found in ASTM23, ISO 143-1 or EN 10045-1, where all the aspects of the test and equipment used are described in detail.

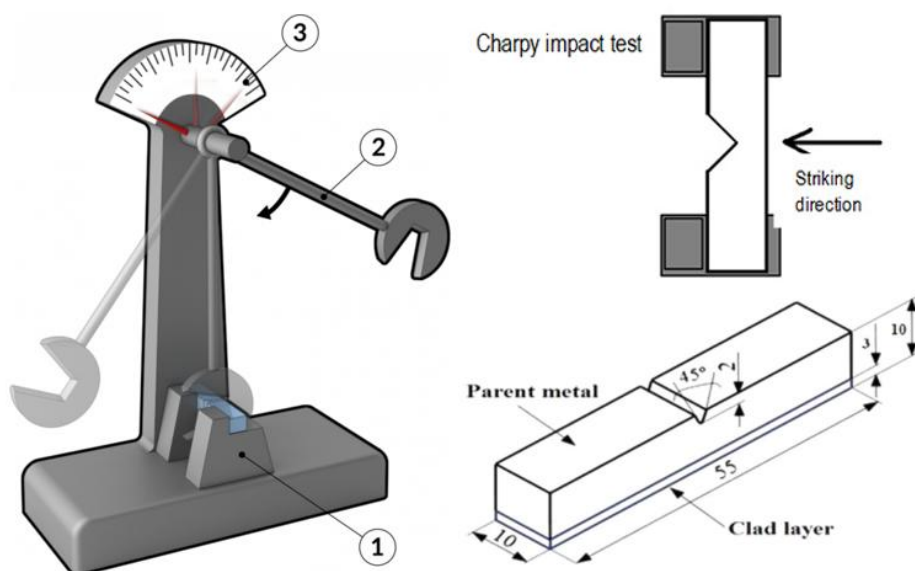


Fig. No.1.2. Charpy Impact Test Equipment and Specimen Measurements

1.7 Cryogenic temperatures

Cryogenics is basically coming from the kryo which means very cold; from Greek language this word has come and genics means to produce. So, basically cryogenic means, science and technology associated with generation of low temperature below 123 K. Cryogenic treatment is an inexpensive one time permanent treatment affecting the entire section or bulk of the component unlike coatings. The treatment is an add on process over conventional heat treatment in which the samples are cooled down to prescribed cryogenic temperature for a long time and then heated back to room temperature. It is believed that life of cutting tool get substantially extended due to cryogenic treatment. However, researchers have been skeptical about the process because it imparts no apparent visible change. Moreover mechanism is also unpredictable and research articles are also not sufficient to support the treatment. So in general cryogenic treatment is still in the dormant level.

Over the past few years there has been an increase in interest in the application of cryogenic temperature to different materials. Some literature says that the cryogenic treatment can improve the life span would depend a lot on the cutting conditions. Hence various research works are being carried out to study the effects of this treatment on the performance of various cutting tools so that it could be added to the regular heat treatment cycle for the components the production sector manufacture. However for evaluating the performance of the cutting tools it is very necessary to study the effect of cutting parameters (cutting speed, depth of cut and feed) on the tool wear. This necessitates planning experiments in advance so that maximum benefit can be derived from data obtained from organized sets of experiment. Designs of experiment (DOE) is one such approach that has proved to be a powerful technique in getting a quantitative relationship among the variables (in the form equations). One important benefit of DOE is that this not only evaluates the significant effect of each of the individual factors (parameters) but also determines the interaction effects among all the factors. When an interaction is large the

corresponding main effect cease to have much meaning. Hence, it is very important to determine the interaction effects of various process variables to fully evaluate the performance of the tools.

A cryogenic treatment is the process of treating work pieces to cryogenic temperatures (i.e. below $-190\text{ }^{\circ}\text{C}$ ($-310\text{ }^{\circ}\text{F}$)) in order to remove residual stresses and improve wear resistance on steels. The process has a wide range of applications from industrial tooling to the improvement of musical signal transmission. Some of the benefits of cryogenic treatment include longer part life, less failure due to cracking, improved thermal properties, better electrical properties including less electrical resistance, reduced coefficient of friction, less creep and walk, improved flatness, and easier machining.

1.8 Wear behavior and Hardness

The wear of steel components used on machinery, which is operating in a wide range of industrial circumstances can cause sudden breakdowns, serious inefficiencies, and significant financial losses. These losses can be reduced by means of cryogenic treatment on steels. Wear is responsible for catastrophic failure of some machine components. This process occurs between hard particles and the working surface. Methods to enhance the life of the component are based on application of wear resistant materials or formation of hard, wear-resistant surface material. The wear rate of steels depends on their chemical constituents and conventional heat treatment.

Hardness is generally considered as resistance to penetration. The harder the materials, the greater the resistance to penetration. Hardness is directly related to the mechanical properties of the material. Factors influencing hardness include microstructure, grain size, strain hardening, etc. Generally as hardness increases so does yield strength and ultimate tensile strength (UTS), thus specifications often

require the results of hardness tests rather than tensile tests. The most popular methods are Brinell, Vickers and Rockwell hardness tests for metals and alloys.

Charpy bar specimens are used most commonly in the United States, while the izod specimen is favored in Great Britain. The charpy specimen has a square cross section (10×10 mm) and contains a 45° V notch, 2mm deep with a 0.25 mm root radius. The specimen is supported as a beam in a horizontal position and loaded behind the notch by the impact of a heavy swinging pendulum.

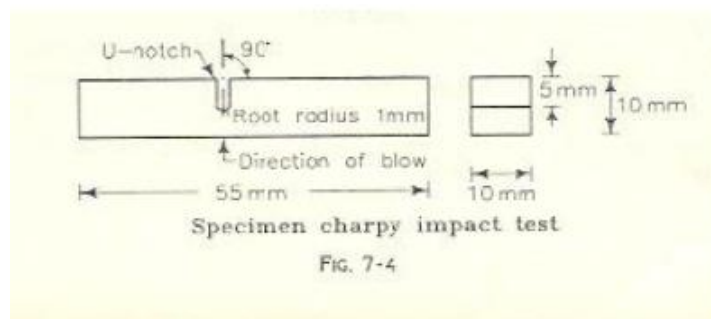


Fig. No.1.3. Specimen Charpy

In the Rockwell test, a diamond cone or a hard steel ball is employed as the indenter

depending on the hardness of materials. Diamond cone or Brale indenter with cone angle of 120° is used to test hard materials and the balls of sizes between 1.6 mm ($1/16$ ") and 12.7 mm ($1/2$ ") are used in testing softer materials.

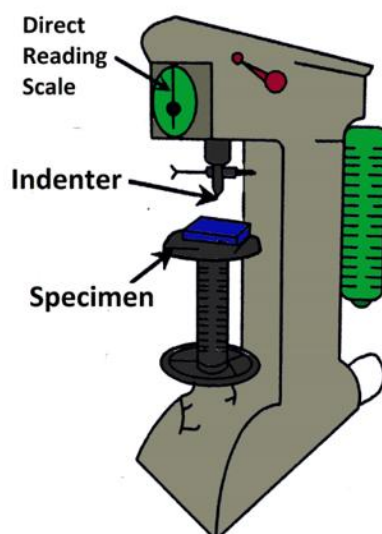


Fig. No.1.4. Rockwell Hardness

Rockwell tests differ from other indentation hardness tests in that the depth of indentation determines the hardness rather than the indentation size (see Figure 2). Therefore,

surface condition of specimens is very

important in Rockwell testing because of its high dependency on the accuracy in indentation depth measurements. In order to establish a reference position a minor load of 10 kgf. Is first applied, and the major load is then applied. Additional penetration due to major load is measured and readings are obtained from a calibrated scale (dial) directly, which has a maximum value of 100, depending on the depth of penetration. Most commonly used Rockwell hardness scales are given in Table-I with typical applications. The hardness numbers are designated HRX, where X indicates the scale used (i.e. 50 HRC for 50 points on the C scale of dial). It should be noted that a Rockwell hardness number is meaningless unless the scale is not specified.

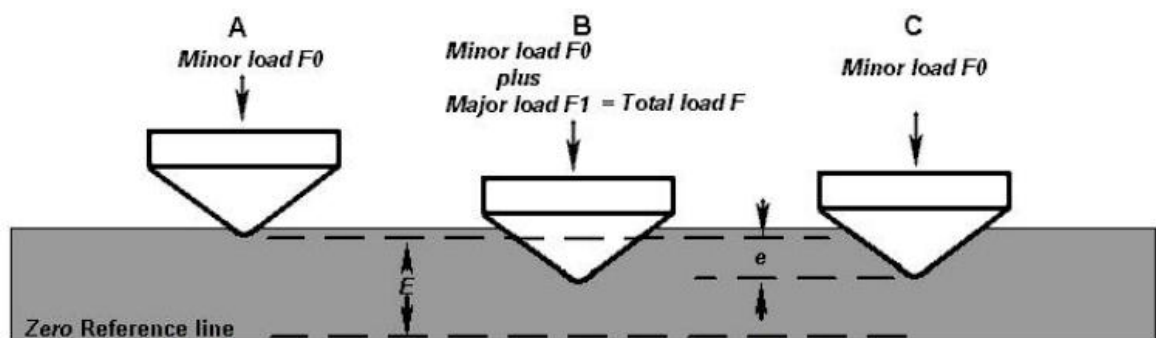


Figure 2. Schematic showing the principle of Rockwell Hardness measurement

Rockwell Hardness Number; $HR = E - e$

Fig. No.1.5. Depth of Indenter Diamond

Where 'E' is a constant depending on the form of indenter and 'e' is the permanent increase in depth of penetration due to major load F measured in units of 0.002 mm.

Table No. 1.1. Measurements of Indenters of Rockwell Hardness**Rockwell Hardness Scales**

Scale	Indenter	Minor Load F_0 kgf	Major Load F_1 kgf	Total Load F kgf	Value of E
A	Diamond cone	10	50	60	100
B	1/16" steel ball	10	90	100	130
C	Diamond cone	10	140	150	100
D	Diamond cone	10	90	100	100
E	1/8" steel ball	10	90	100	130
F	1/16" steel ball	10	50	60	130
G	1/16" steel ball	10	140	150	130
H	1/8" steel ball	10	50	60	130
K	1/8" steel ball	10	140	150	130
L	1/4" steel ball	10	50	60	130
M	1/4" steel ball	10	90	100	130
P	1/4" steel ball	10	140	150	130
R	1/2" steel ball	10	50	60	130
S	1/2" steel ball	10	90	100	130
V	1/2" steel ball	10	140	150	130

1.9 Ductile-Brittle Transition

Ductile-brittle transition is a limiting state between ductile and brittle behaviour of a material. This transition between ductile and brittle behaviour is defined by a temperature, called ductile-brittle transition temperature t_{db} . A ductile material shows brittle nature below this temperature whereas a brittle material possess ductile nature above this temperature.

Conditions Responsible for Brittle Fracture of Ductile Metals:

- I. A low or decreasing temperature,
- II. High rate of straining,
- III. Large grain size of material,
- IV. High stress concentration,

- V. Rough surface conditions, and
- VI. Tri-axial stress conditions.

Factors Affecting the Transition Temperature:

- I. Fine grained materials possess lower transition temperature than the coarse grained materials.
- II. Transition temperature is raised due to stress concentration such as on sharp notches.
- III. Effect of higher straining rate is to cause increased transition temperature.
- IV. Most of the ductile BCC metals behave as brittle materials at low temperatures and at a very high rate of straining, whereas many FCC metals behave as ductile materials at very low temperatures.
- V. It is because a higher yield stress σ_y is required to move dislocations in BCC metals than FCC metals.
- VI. This σ_y increases rapidly when temperature lowers down but this is not the case with the stress required to propagate a crack σ_f .
- VII. Steel structures such as oil rigs, ships and bridges are generally fail in winter than in summer due to ductile-brittle transition effect.
- VIII. Ductile to brittle transition temperature for metals is around $0.1-0.2 T_m$ while for ceramics is $0.5-0.7 T_m$.

T_m = Melting temperature

Chapter 2

Literature Review

Sahadev Shivaji Satur, Gorakshanth Shivaji Kale, Sujit Haridas Merad produced a paper discussing the importance of the transition states and temperatures of different metals. And they also briefly described the ways we can follow to observe this transition and how to deal with it.

Susheel Kaila (2010) pointed out that cryogenics is an exciting, important and inexpensive method to increase the life of the steel component. It improves abrasive wear resistance, erosion and corrosion resistance and stabilizes the strength characteristics of the steels.

Bensely et al (2005) studied the wear resistance of case carburized En353 steel after shallow and deep cryogenic treatments. The shallow cryogenic treatment samples are treated at -80°C for 5 hours and the deep cryogenic treatment samples are treated at -196°C for 24 hours. It is concluded that the improvement over conventional heat treatment was 85% and 372% for SCT and DCT respectively. In addition to the well-known effect of converting retained austenite to martensite, DCT induces precipitation and finer distribution for carbides which are solely responsible for the improved wear resistance.

Hasim et al (2002) pointed out that the cryogenic treatment of materials are gaining importance in recent days because of their potential to produce steel components that find enormous application in industries, nuclear power plants, fertilizer plants, medical, aerospace and avionics. This is due to the fact that materials treated under cryogenic environments attain superior properties that call for operation under severe environments as indicated by Charles and Arunachalam (2006). It is a one-time homogenous

process that provides significant extension in the performance and productive life of steel.

Maria Arockia Jaswin et al (2010) investigates the wear resistance improvement in En 52 and 21-4N valve steels through shallow and deep cryogenic treatment using a reciprocatory friction and wear monitor as per the MAST standard: G-133. The shallow cryogenic treatment is carried out at -80°C and the deep cryogenic treatment is carried out at -196°C. It has been observed that the wear resistance of En 52 and 21-4N has improved by 81.15% and 13.49% respectively, due to shallow cryogenic treatment, 86.54% and 22.08% respectively, due to deep cryogenic treatment, when compared to the conventional heat treatment. The microstructural study suggests that the improvement in wear resistance and hardness is attributed to the conversion of retained austenite into martensite, along with precipitation and distribution of the carbides brought in by the cryogenic treatment.

Barron Randall (1974) and Harish et al (2009) studied that deep cryogenic treatment of SAE 52100 bearing steel enhances wear resistance. Dong et al (1998) studied the effect of DCT with respect to the microstructure of T1 high speed steels. It is proved that deep cryogenic treatment can enhance wear resistance by the precipitation of nano-sized eta-carbides in the primary martensite.

Mahdi Koneshlou et al (2011) studied the effects of shallow cryogenic treatments on microstructure and mechanical properties of H13 hot work tool steel. Shallow cryogenic treatment at -72 °C and deep cryogenic treatment at -196 °C are applied and it is found that by applying the shallow cryogenic treatment, the retained austenite is transformed to martensite. As the temperature is decreased more retained austenite is transformed to martensite. The deep cryogenic

treatment at a very low temperature and long sample holding times also appear to result in the precipitation of more uniform and very fine carbide particles. However tempering of the deep cryogenically treated samples improve the wear properties of the H13 tool steel. Stratton (2007) put forward the following material processing route for the cryogenic treatment of steels to attain maximum wear resistance.

2.1 Effect of Temperature on Mechanical Properties:

At higher temperatures the yield strength is lowered and the fracture is more ductile in nature. On the opposite end, at lower temperatures the yield strength is greater and the fracture is more brittle in nature. This relationship with temperature has to do with atom vibrations. As temperature increases, the atoms in the material vibrate with greater frequency and amplitude. This increased vibration allows the atoms under stress to slip to new places in the material (i.e. break bonds and form new ones with other atoms in the material). This slippage of atoms is seen on the outside of the material as plastic deformation, a common feature of ductile fracture.

When the temperature decreases however, the exact opposite is true. Atom Vibration decreases and the atoms do not want to slip to new locations in the material. So when the stress on material becomes high enough, the atoms just break their bonds and do not form new ones. This decrease in slippage causes little plastic deformation before fracture. Thus, we have a brittle type fracture. At moderate temperatures (with respect to the material) the material exhibits characteristics of both types of fracture. In conclusion, temperature determines the amount of brittle or ductile fracture that can occur in a material.

Another factor that determines the amount of brittle fracture that occurs in a material is dislocation density. The higher the dislocation density, the more brittle the fracture will be in material. The idea behind this theory is that plastic deformation comes from the movement of dislocations. As dislocations increase in a material due to stresses above the materials yield point, it becomes increasingly difficult for the dislocations to move because they pile into each other. So a material that already has a high dislocation density can be deformed but so much before it fractures becomes into more brittle manner. The last factor is grain size. As grains get smaller in material, the fracture becomes more brittle. This phenomenon is due to the fact in smaller grains, dislocations have less space to move before they hit a grain boundary. When dislocations cannot move very far before fracture, then plastic deformation decreases. Thus, the material's fracture is more brittle.

Crack initiation and propagation are essential to fracture. The manner through which the crack propagates through the material gives great insight into the mode of fracture. In ductile materials (ductile fracture), the crack moves slowly and is accompanied by a large amount of plastic deformation. The crack will usually not extend unless an increased stress is applied. In brittle fractures, cracks spread very rapidly with very little or no plastic deformation. The cracks that propagate in a brittle material will continue to grow and increase in magnitude once they are initiated. Another important mannerism of crack propagation is the way in which the advancing crack travels through the material. A crack that passes through the grains within the material is undergoing Transgranular fracture. However, a crack that propagates along the grain boundaries is termed an Intergranular fracture. On both macroscopic

and microscopic levels, ductile fracture surface have distinct features. Macroscopically, ductile fracture surfaces have larger necking regions and an overall rougher appearance than a brittle fracture surface.

2.2 Ductile – Brittle transition

The ductile-brittle transition is exhibited in bcc metals, such as low carbon steel, which become brittle at low temperature or at very high strain rates. FCC metals, however, generally remain ductile at low temperatures. In metals, plastic deformation at room temperature occurs by dislocation motion. The stress required to move a dislocation depends on the atomic bonding, crystal structure, and obstacles such as solute atoms, grain boundaries, precipitate particles and other dislocations. If the stress required moving the dislocation is too high, the metal will fail instead by the propagation of cracks and the failure will be brittle. Thus, either plastic flow (ductile failure) or crack propagation (brittle failure) will occur, depending on which process requires the smaller applied stress. In FCC metals, the flow stress, i.e. the force required to move dislocations, is not strongly temperature dependent. Therefore, dislocation movement remains high even at low temperatures and the material remains relatively ductile. In contrast to FCC metal crystals, the yield stress or critical resolved shear stress of bcc single crystals is markedly temperature dependent, in particular at low temperatures. The temperature sensitivity of the yield stress of bcc crystals has been attributed to the presence of interstitial impurities on the one hand, and to a temperature dependent Peierls Nabarro force on the other. However, the crack propagation stress is relatively independent of temperature. Thus the mode of failure changes from plastic flow at high temperature to brittle fracture at low temperature.

well known in bcc metals. As temperature decreases, a metal's ability to absorb energy of impact decreases. Thus its ductility decreases. At some temperature the ductility may suddenly decrease to almost zero. This transition is often more abrupt than the transition determined by the energy absorbed. This temperature is called the nil-ductility transition temperature (NDTT). The NDTT is lower than the fracture energy transition temperature and is generally more narrowly defined. The difference between these two transition temperatures is related to the high rate of loading during impact testing rate sensitive metals. Increased loading rates cause the yield stress to increase while increasing temperature causes ductility to increase.

Chapter 3

Experimental details

3.1 Specimen preparation

(a) Material

Mild steel specimen of dimensions (10 × 10 × 55 mm) were polished using emery papers of 1/0, 2/0, 3/0, 4/0 followed by cloth polishing using 5 μm Al₂O₃ slurry.

(b) Muffle furnace

A muffle furnace (sometimes retort furnace in historical usage) is a furnace in which the subject material is isolated from the fuel and all of the products of combustion, including gases and flying ash. After the development of high-temperature heating elements and widespread electrification in developed countries, new muffle furnaces quickly moved to electric designs.



Fig. 3.1 Muffle furnace (range 1000 °C)

Today, a muffle furnace is (usually) a front-loading box-type oven or kiln for high-temperature applications such as fusing glass, creating enamel coatings, ceramics and soldering and brazing articles. They are also used in many research facilities, for

3.2 Impact testing:

In an impact test, a specially prepared notched specimen is fractured by a single blow from a heavy hammer. Impact load is produced by a swinging of an impact weight (hammer) from a height h . release of the weight from the height swings the weight through the arc of a circle, which strikes the specimen to fracture at the notch. The drop angle of pendulum for Charpy is 140° and for Izod is 90° . Also, the initial potential energy for Charpy is 300 joules and for Izod is 170 joules with a least count of 2 joules. When the striker impacts the specimen, the specimen will absorb energy until it yields. At this point, the specimen will begin to undergo plastic deformation at the notch. The test specimen continues to absorb energy and work hardens at the plastic zone at the notch. When the specimen can absorb no more energy, fracture occurs.



Fig 3.2 Typical Photograph of impact test machine

Kinetic energy of the hammer at the time of impact is $mv^2/2$, which is equal to the relative potential energy of the hammer before its release. (mgh), where m is the mass of the hammer and $v = \sqrt{2gh}$ is its tangential velocity at impact, g is gravitational acceleration (9.806 m/s^2). Energy used can be measured from the scale given. The difference between potential energies is the fracture energy. In test machine this value indicated by the pointer on the scale

$$\text{The energy absorbed} = WL (\cos B - \cos A)$$

Where W = Weight of the hammer,

L = Equivalent length of the pendulum,

B = Maximum angle made by the hammer with the vertical after delivering the blow.

A = Maximum angle made by the hammer with the vertical before delivering the blow.

3.3 Cryogenic Treatment

Cryogenic treatment of metals to improve wear characteristics is a relatively new engineering field and little has been reported on the basic mechanism by which the technique operates. Over the past few years there has been increase in interest in the application of cryogenic treatment to different materials. Research has shown that cryogenic treatment increase product life, and in most cases provides additional qualities to the product such as stress relieving. It has been reported that cryogenic treatment can double the service life of HSS tools.



Fig 3.3 Typical Image of Liquid Nitrogen container

Given below is the list of common fluids used in cryogenic applications

Table No. 3.1. List of Cryogenic Fluids and Boiling Points

Cryogenic fluid	Boiling Point	
	In Kelvin (K)	In Celsius (°C)
Helium – 3	3.19	-269.96
Neon	27.09	-252.88
Hydrogen	20.27	-252.88
Nitrogen	77.09	-196.06
Air	78.80	-194.35

Chapter 4

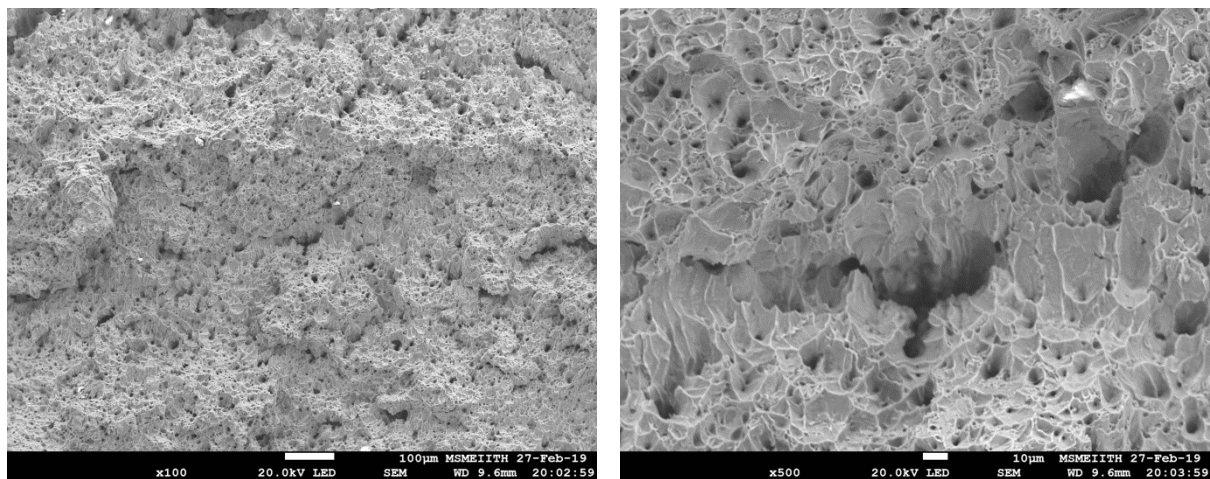
Results and discussions

4.1 Microstructural investigation of Impact test specimen

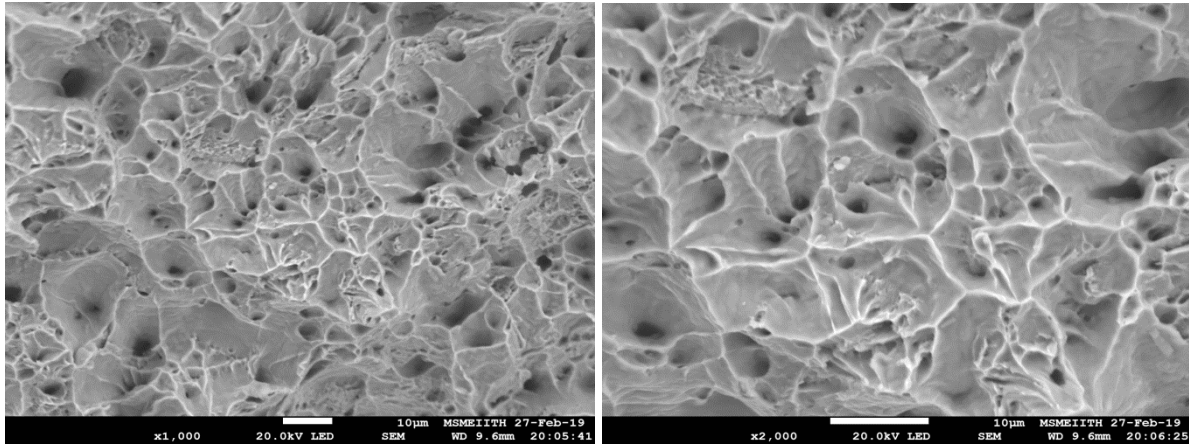
The impact test specimens were characterized prior to their exposure to cryogenic treatment. The photo graph of impact test specimen (10 ×10×55 mm) is shown in fig 4.1. Fractography of mild steel specimen at room temperature with different magnifications are shown in fig 4.2



Fig.4.1 Typical photograph of impact test specimen



**Fig.4.1 (a). Scanning Electron Microscope (SEM) image of mild steel specimen at ×100.
(b) SEM image of mild steel specimen at ×500**



(c) SEM image of mild steel specimen at $\times 1000$. (d) SEM image of mild steel specimen at $\times 2000$

4.2 Determination of Impact Energy through Charpy test

A specially prepared notched specimen is fractured by a single blow from a heavy hammer. Impact load is produced by a swinging of an impact weight (hammer) from a height. Release of the weight from the height swings the weight through the arc of a circle, which strikes the specimen to fracture at the notch. Here it is interesting to note that height through which hammer drops determines the velocity and height and mass of a hammer combined determine the energy. This energy value called impact toughness or impact value, which will be measured, per unit area at the notch. Typical images are shown in below for mild steel material at four different temperature conditions.

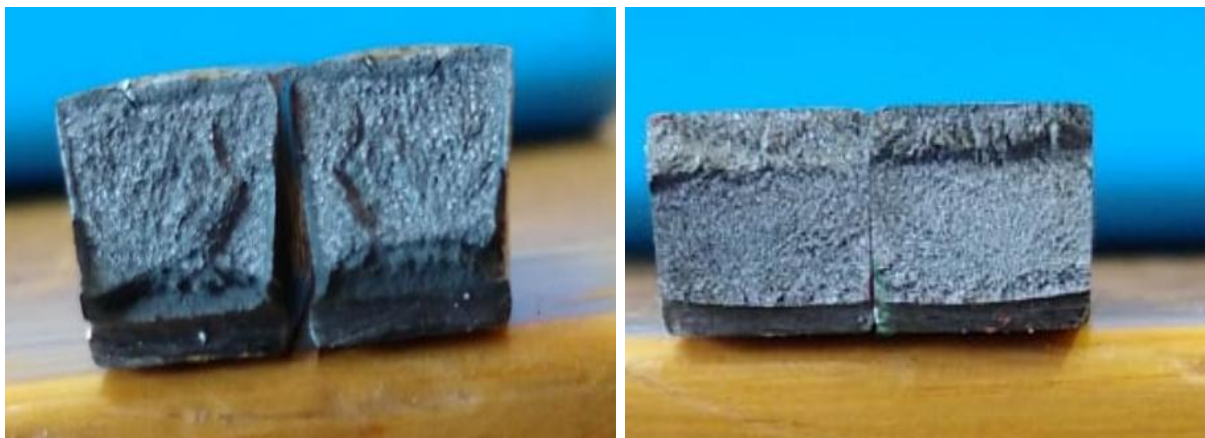


Fig., 4.2 (a) Room Temperature (b) 0°C Temperature



(c) Liquid Nitrogen for 30 Minutes (d) Furnace heating at 450 °C for 30 Minutes

Table No. 4.1. Charpy Impact Test Results

Temperature	Charpy Impact Energy (in J)
-70	13
-50	22
-20	76
0	104
27	92
250	118

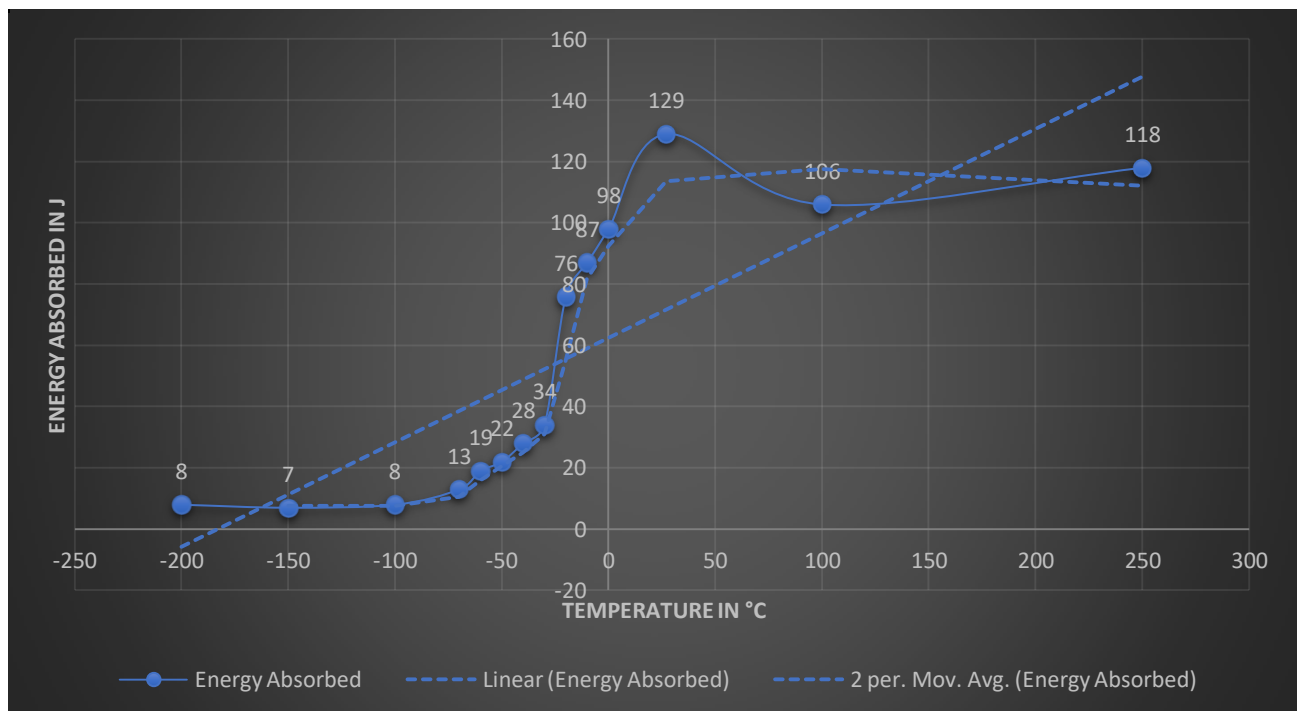


Fig 4.2(e) Energy Absorbed vs Temperature Graph

With increasing in temperature the energy absorbed by the mild steel increases. Since with increasing in temperature material will enable more number of slip systems, material with more slip system will behave like a ductile in nature. Ductile material exhibit good amount of plasticity when compared with the materials which are at low temperatures. So as we are increasing temperature from $-100\text{ }^{\circ}\text{C}$ to $450\text{ }^{\circ}\text{C}$ energy absorbed by the material will increases.

4.3 Correlation between experimental and metallographic results

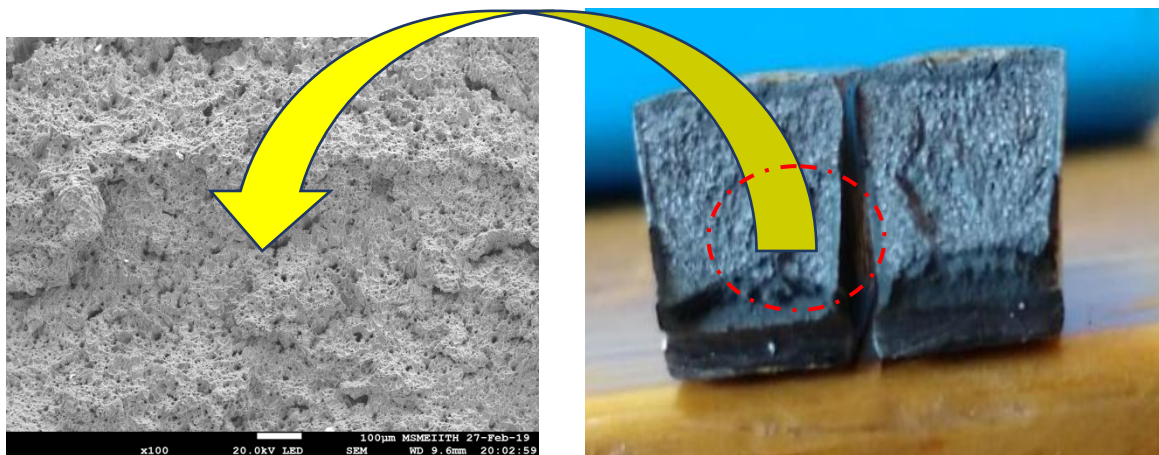


Fig 4.3 Typical SEM image of impact test specimen at Room temperature with microstructural validation

Fig 4.3 SEM image clearly showing fine micro structure at $\times 100$ (magnification) for mild steel material at room temperature. Fine microstructure materials will exhibit good amount of strength since it will possess high intermolecular energy between the atoms.

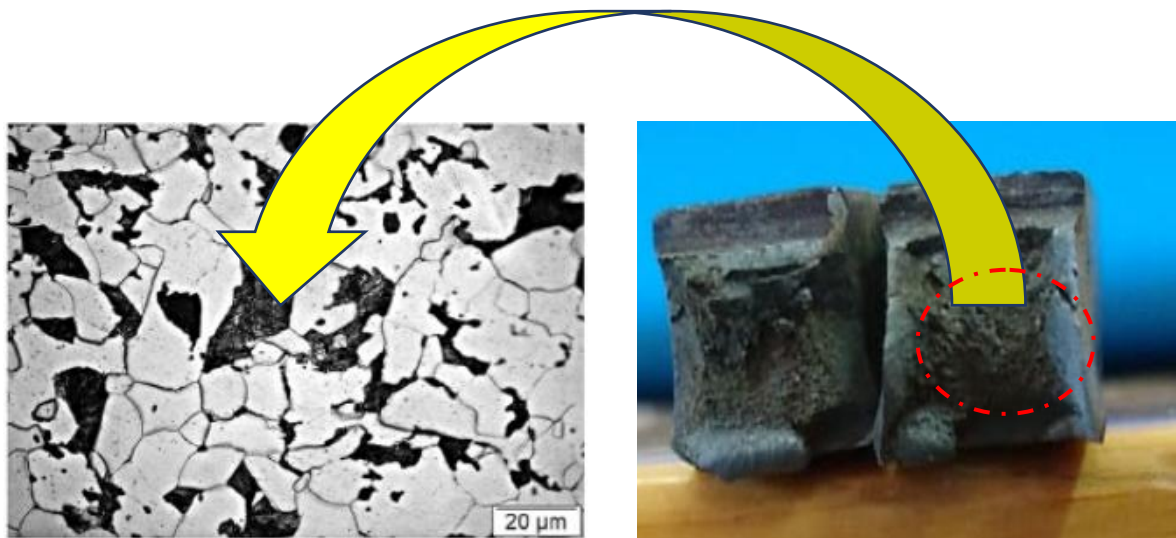


Fig 4.4 Typical Optical Microscope image of impact test specimen at Cryogenic temperature ($-196.06\text{ }^{\circ}\text{C}$) with microstructural validation

Cryogenic or super chilling of most metals and alloys and some plastics, reduces wear and stress to a far greater extent than untreated metals and alloys.

Most medium carbon steels and low alloy steels undergo transformation to 100 % Martensitic at room temperature. However, high carbon and high alloy steels have retained austenite at room temperature. To eliminate retained austenite, the temperature has to be lowered. In cryogenic treatment the material is subjected to deep freeze temperatures of low as $-196.06\text{ }^{\circ}\text{C}$, but usually $-75\text{ }^{\circ}\text{C}$ is sufficient. The austenite is unstable at this temperature, and the whole structures become Martensitic as shown in figure 4.6. This is the main reason to use cryogenic treatment at high carbon & high alloy steels.



Fig.4.5 Microscopic Structures of Steels

Chapter 5

Conclusions

- With increase in temperature the energy absorbed by the mild steel increases. Because when temperature increases material will active more number of slip systems, material with more number slip systems will give good amount of toughness. Generally Tough materials will afford high amount of impact loads. So as we are increasing temperature from -100 °C to 450 °C energy absorbed by the material will increase.
- Materials which are treated at low temperatures will tend to behave like a BCC crystal structure in nature. Generally BCC materials at low temperatures will be brittle in nature. Since brittle materials are hard in nature will exhibit more amount of hardness, brittle materials will possess greater resistance to penetration. So with increasing in temperature from -100 to 450 °C hardness number has been decreased.
- The properties of materials change, when cooled to cryogenic temperatures. Cryogenics is a treatment that you will never wear off the process like coating. But you will be able to sharpen, dress, or modify your tooling without damaging the process. The process also relieves residual stresses some forms of plastics, this has been proven by field studies conducted on product in high impact scenarios where stress fractures are evident.
- From the experimental results and graph we can conclude that for mild steel energy absorbed by the specimen is very low at -196.96 °C and high at the furnace heated 450 °C.