EXPERIMENT -1

- 1. **AIM** -: Preparation and study of the Micro Structure of pure metals like Iron, Copper and Aluminum.
- 2. Objectives: a) To Learn the preparation of specimen for microscopic observation.b) To understand what Microscopy is, and how it can be used to observe Microstructure of metals,

3. APPARATUS / EQUIPMENT –

3.1 Abrasive Cut-Off Wheel Machine



- 3 **HP** Motor, 1440 rpm
- Manual model .
- 10" Diameter wheel
- Fully enclosed
- Cutting Capacity 50mm Diameter & 75mm
- Height X 200mm Length.

This Equipment is used to cut the sample pieces form the component of all kind of materials. These sample pieces can be used microscopic observation.

3.2 Specimen Mounting Press

- Manual Operation
- 1" Mould Capacity
- Digital Temperature/ Time control unit
- Pressure gauge for reading pressure
- Alarm indicator for cycle completion.

This Equipment is used to mount specimen in bakelite.

Particularly is useful in hot mounting process.

3.3 Belt Linishing Machine

This Equipment is used for rough Grinding of the Specimen.

3.4 Polishing Machine -

- Variable speeds 100 rpm 800rpm
- High torque PMDC motor with D. C. Drive.
- Water In and Drain
- Corrosion resistant fiber reinforced cover.
- 8" Disc

This Equipment is used to polish the specimen after rough grinding with the help of polishing clothe. It also useful for rough grinding with grinding disc.

3.5 Metallurgical Microscope.



Binocular model

- Eye pieces: 10X, 15X & 2 OX
- Magnification: 10X 500X. This Microscope is used for microscopic observation of specimen with the adjustable magnification.

CONSUMABLES/ RAW MATERIAL -:

Rod or Sample pieces of Pure Iron, Copper and Aluminum, Abrasive Polishing paper 80grit/ 100 grit. Polishing powder

4. THEORY -:

4.1 Metallography:

Metallography is the study of metals by optical and electron microscopes. Structures, which are coarse enough to be discernible by the naked eye or under low magnifications, are termed macrostructures. Useful information can often be gained by examination with the naked eye of the surface of metal objects or polished and etched sections. Those, which require high magnification to be visible, are termed microstructures. Microscopes are required for the examination of the microstructure of the metals. Optical microscopes are used for resolutions down to roughly the wavelength of light (about half a micron) and electron microscopes are used for detail below this level, down to atomic resolution. The most commonly used microscope is the conventional light microscope. In principle, optical microscopes may be used to look through specimens ('in transmission') as well as at them ('in reflection'). Many materials, however, do not transmit light and so we are restricted to looking at the surface of the specimens with an optical microscope (TEM) and to look at the surfaces e.g. Scanning Electron Microscope (SEM) Microscopy can give information concerning a material's composition, previous treatment and properties. Particular features of interest are

(1) Grain size (II) phases present (III) Chemical homogeneity (IV) distribution of phases (V) Elongated structures formed by plastic deformation

4.2 Optical Microscopy:

With optical microscopy, the light microscope is used to study the microstructure; optical illumination systems are its basic elements. For materials that are opaque to visible light (all metals, many ceramics and polymers), only the surface is subject to observation, and the light microscope

must be used in a reflective mode. Contrasts in the image produced result from differences in reflectivity of the various regions of the microstructure. Careful and meticulous surface preparations are necessary to reveal the important details of the microstructure. The specimen surface must first be ground and polished to a smooth and mirror like finish. This is accomplished by using successively finer abrasive papers and powders.

The microstructure is revealed by a surface treatment using an appropriate chemical reagent in a procedure termed etching. The etching reagents depend on the material used and after etching the specimen must be washed with alcohol and ether to remove the grease. The atoms at the grain boundaries are chemically more active, and consequently dissolve more readily than those within the grains forming small grooves. These grooves become discernible when viewed under a microscope because they reflect light at an angle different from that of the grains themselves. When the microstructure of a two-phase alloy is to be examined, an etchant is chosen that produces a different texture for each phase so that the different phases may be distinguished from each other.

5. PROCEDURE:

The preparation of metallic or other materials for microscopic examination and micro structural characterization is in principal very simple. There are four basic processes that you will need to become familiar with: sample cutting and sectioning, metallographic mounting, Surface grinding and surface polishing.

5.1 Sample Cutting and Sectioning

5.1. J. Sectioning

Sectioning means removal of convenient size specimen from large sample with minimal damage to microstructure with the help of abrasive cut off machine. Abrasive cutting wheel/saw is attached to cutting machine and for work piece holding proper vice is provided on machine. The primary concern in this process is to minimize the heating of the sample due to the cutting. For this reason, the cut-off saws that is equipped with either water-cooling systems.

5.2 Mounting

If sample is large enough (about 25 mm square or larger) than do not need to mount it, as it will be able to control the sample during polishing without a mount. For smaller samples there are two basic mounting techniques used in this laboratory. Mounting facilitates handling during preparation and handling. It also avoids damage to polishing wheels during polishing. The most common uses a thermosetting plastic compound (Bakelite) to encapsulate the specimen known as hot-mounting process, and the second uses a room temperature curing epoxy known as cold-mounting process. The Bakelite mounting is by far the most common and easiest. The room temperature curing epoxy mount should only be used for samples that are extremely sensitive to heat.

The Bakelite process uses a sample mounting press that applies a pressure to the Bakelite/sample system during the cure to remove voids and gaps and to fully fill the sample spaces. Bakelite comes in a variety of colors, which can be combined to produce easy sample identification.

5.2.2 The procedure for Hot mounting process.

The following process is used to encapsulate your specimen in Bakelite:

- 1) Place sample face down on the small piston inside the press, and lower the piston into the cylinder by opening slightly the valve on the front of the press.
- 2) Approximately three tablespoons of Bakelite is poured over the sample, and the top of the press gently screwed into place. DO NOT TIGHTEN THE TOP OF THE PRESS. It is only necessary to engage all of the screw threads; you do not have to tightly secure the top.
- 3) The cylindrical heater is plugged in and turned on (the red light should turn on). Place the heater around the mold. The heater is thermostatically controlled and will heat the mold to about 135-150 °C. Close the valve, and pump up the cylinder using the hand lever. As the Bakelite heats, it will begin to flow to fill the void spaces, and the pressure will drop. Maintain the constant.
- 4) When the pressure stops rapidly dropping, the whole mold has reached I50°C. Begin timing for 5-7 minutes to fully cure the Bakelite. Maintain the pressure during the entire heating and cooling cycle.
- 5) At the end of the heating cycle, remove the heater and place the cooling collar on the mold for an additional 6-8 minutes.
- 6) Crack the valve to release the pressure, and unscrew the top of the mold. When the mold top is fully unscrewed (it may not come out due to adhesion with the Bakelite), close the valve and slowly pump up the cylinder to push the sample fully out of the press. Mark the sample on the back.
- 7) Clean any residual Bakelite off of the mold surfaces.

5.2.2 The procedure for Cold Mounting process.

The following process is used to room temperature curing epoxy process:

- 1) Apply mold release agent to mold. Place specimen in the mold.
- 2) Mix epoxy powder and bonding liquid in 1:2 ration in a cup.
- 3) Pour into mold, wait for 10 minutes.
- 4) Eject the mold.

5.3 Sample Surface Polishing

The goal of the surface polishing is to end up with a planar cross section of sample free from scratches or disturbed metal introduced by the cutting and sectioning. This process is a step-wise process that can be broken into three loosely separate parts: grinding, coarse polishing, and final polishing.

5.3.1. Grinding

The first step in preparing your sample is to ensure that you have a flat surface to begin with. A water-cooled abrasive grinder is available to form a flat initial surface from which to begin. After getting a flat sample on the belt grinder, WASH sample thoroughly. The hand lapping station has four graded abrasive papers to produce a sequentially finer surface finish. Be sure the water is turned on and flowing uniformly over the abrasives. Start with the coarsest grit (240) and, using a firm and

uniform pressure, slowly move the specimen forward and back across the abrasive. This will produce parallel scratches of uniform size. Continue this step until the entire surface of your sample is flat and contains only scratches of the size of 240 grit abrasive. When the sample is flat and the only scratches remaining are those due to the 240 grit abrasive, WASH your sample and your hands thoroughly, and move to the 320 grit abrasive. Repeat this procedure for the 400 grit and the 600 grit abrasive, checking after each step to be sure that only those scratches remain that are due to the smallest grit.

5.3.2 Rough Grin dins

Before proceeding to the first polishing wheel (leftmost wheel), wash sample with water.

- 1) First, apply a small amount of water to the wheel, turn on the motor, and gently clean off the wheel with your fingers.
- 2) Apply a small amount of abrasive slurry to the wheel. This wheel uses an A1203 abrasive in a water suspension. The abrasive particles are 5 micrometers in diameter.
- 3) Carefully place your sample on the wheel while gripping it tightly. Slowly move the sample in a circular motion against the rotation of the wheel. Use a moderate and even pressure. It is important to ensure that you keep the sample flat on the wheel so that the final surface will be completely planar.
- 4) After several minutes on the wheel, hold the sample in one place for a moment. This will provide lots of parallel scratches that you can use to determine if you have removed the damage from the grinding steps.
- 5) Examine the sample under the microscope to determine if all the scratches are the same size.
- 6) Repeat steps 1-5 on the middle polishing wheel. This wheel uses a 0.3 micrometer A1203 abrasive in a water suspension.

5.3.3. Final Polishing

- 1) Repeat steps 1-5 above on the right polishing wheel. This wheel uses a 0.05 micrometer A1203 abrasive in a water suspension. At this point, the sample will be very smooth to the eye and even the oils and dirt on your fingers will scratch it with larger scratches than the abrasive. DO NOT TOUCH THE SAMPLE SURFACE FROM THIS POINT ON.
- 2) The last step in the process is to etch the sample to bring out the microstructure.
- 3) Use a cotton swab and a petri dish for the etching. Gently swab the surface of your sample with the etchant. Roughly spreading the etchant will scratch your surface. Let the etchant stand for15 seconds or so and rinse the sample with water to stop the etching, and rinse again with methanol. Rinse the swab with water and throw into the trash bin.
- 4) Examine specimen under the microscope. You may require several etching steps to bring out the microstructure
- 5) If the sample is over-etched, repeat the final polishing step and re-etch for a shorter time. Samples to be examined at high magnification generally require shorter etching times than those to be viewed at lower magnifications.
- * After last polishing stage the sample looks mirror like.

5.4 Etching

Grains cannot be seen without etching. Cracks, pores and defects are observed without etching. Etchant reacts with atoms and dissolves them. Atoms at grain boundaries dissolve quickly.

Dissolved grain boundaries appear dark.

Steps:

- 1) Apply enchant to polished surface for some time
- 2) Rinse with distilled water

5.5. Metallographic Observation

Observe microstructure. Place specimen on metallograph and adjust magnification, focus and position s adjus micro High magnification - to study phases and Low magnification -to study grain size.

5.5. / <u>Microphotography</u>

In this laboratory, you will report the microstructures of prepared samples in specific formats. You will be expected to sketch the microstructure that you see under the microscope by hand. In sketching the microstructure there are several things to keep in mind. First, the magnification that you use depends upon the scale of the microstructure you are looking for. It is IMPORTANT to know in advance of the lab class what the expected microstructure for your samples are and at what scale they should appear, hi sketching the microstructure, you should indicate only the important features of the structure that you observe-don't make a photographic reproduction of the microstructure. Simple sketches show that you know what the important structures are and have identified them in the cross section.

An example of what is considered to be a good laboratory report sketch of the microstructures is included in the appendix. Your sketch MUST INCLUDE:

- 1) The sample name and composition,
- 2) The metallurgical history of the sample,
- 3) A simple sketch of the important microstructure indicating
 - a) The magnification used (i.e. "MAG= 100 X"),
 - b) Important phases and features noted,
- 4) Etchant used.

6. OBSERVATION:



7. PRECAUTIONS:

There are several general instructions to keep in mind during this part of the process.

- 1) CLEANLINESS!!! Keep the room and the work areas clean, especially the polishing area. Each step in the polishing process uses a finer grade of abrasive, so good results require that both your sample and your hands are free from abrasive at each stage before proceeding to the next stage of the process. Turn the power off and cover the wheels when you are through. Clean up any spilled water and wipe up the countertop.
- 2) Throw away the leftover etchants into the sink with a liberal amount of water. Swabs should be rinsed and thrown into the trash bin IMMEDIATELY AFTER USE. Wash your containers with water, rinse in methanol, and place them on the shelf above the sink.
- 3) Use goggles and gloves while handling chemicals. Contact the T.A. or the Instructor in the case of an acid spill, or if new etchants are needed. Be extra careful when using hydrofluoric acids.
- 4) Do not eat or drink anything while working in the lab. Wash your hands thoroughly with soap before leaving the lab.

8. TROBLESHOOTING:

TROUBLE SHOOTING GUIDE for Grinding and polishing

Symptom Specimen has fine matte like finish	 Cause This layer of particles on the surface 	 Action Clean surface swab 110% cotton swab and alcohol. Clean specimen by flushing specimen and polishing pad with water for last 10-15 seconds of the polishing operation.
Excessive relief in surface finish	Over polishing	 Repeat step prior to final polish and shorten final polishing time
Scratches in specimen	 Improper selection of polishing and Contaminated pad Removal of secondary phases 	 Review application guideline chart for polishing pads or select a softer pad Replace polishing pad Alternate between polishing and etching
Etching of specimen	 Alumina abrasive pH too high 	Use lower pH alt

9. QUESTIONS:

- 1. What is Microscopy?
- 2. Why it is necessary to mount the specimen before grinding and polishing?
- 3. Which different etching agents used for specimen preparations?
- 4. What is the principal of metallurgical Microscope?
- 5. How is the microstructure of pure Iron?
- 6. How is the microstructure of pure Copper?
- 7. How is the microstructure of pure Aluminum?
- 8. How is the microstructure of low carbon steel?
- 9. How is the microstructure of medium carbon steel?
- 10. What is the purpose of Etching?

EXPERIMENT-2

- **1.Aim-:** Preparation and study of the Micro Structure of pure metals Mild Steel, Low Carbon steel and High Carbon Steel.
- 2.Objectives: a.) To Learn the preparation of specimen for microscopic observation.b) To understand what Microscopy is, and how it can be used to observe Microstructure of metals.

3. APPARATUS / EQUIPMENT

3.1 Abrasive Cat-Off Wheel Machine,



- 3 HP Motor, 1440 rpm
- Manual model
- 10" Diameter-wheel
- Fully enclosed
- Cutting Capacity 50mm Diameter & 75mm
- Height X 200mm Length.

This Equipment is used to cut the sample pieces form the component of all kind of materials. These sample pieces can be used microscopic observation.

3.2 Specimen Mounting Press

- Manual Operation
- 1" Mould Capacity
- Digital Temperature/ Time control unit
- Pressure gauge for reading pressure
- Alarm indicator for cycle completion.



This Equipment is used to mount specimen in bakelite. Particularly is useful in hot mounting process.

3.3 Belt Linishing Machine.



This Equipment is used for rough Grinding of the Specimen 3.4 Polishing Machine Machine.



- Variable speeds 100rpm 800rpm
- High torque PMDC motor with D. C. Drive.
- Water In and Drain
- Corrosion resistant fiber reinforced cover.
- 8" Disc

This Equipment is used to polish the specimen after rough grinding with the help of polishing clothe. It also useful for rough grinding with grinding disc.

3.5 Metallurgical Microscope.



Binocular model

- Eyepieces: 10X, 15X&20X
- Magnification: 10X-500X.

This Microscope is used for microscopic observation of specimen with the adjustable magnification.

CONSUMABLES/ RAW MATERIAL -:

Rod or Sample pieces of Pure Iron, Copper and Aluminum, Abrasive Polishing paper 80grit/ 100 grit. Polishing powder

4. THEORY -:

Mild Steel, Low Carbon steel and High Carbon Steel are types of ferrous materials and are most important to the engineering application because of their wide range of properties and verity of applications. Theoretically, steels are the alloys of iron and carbon in which the carbon content is between 0.008 to 2.0 per cent. The structures and properties can be discussed with the help of Fe-C equilibrium diagram.

4.1 Iron -Iron Carbide Equilibrium Diagram



Fig 5 Fe-Fe₃C Phase Diagram

Figure 5 shows the equilibrium diagram for combinations of carbon in a solid solution of iron. The diagram shows iron and carbons combined to form Fe-Fe₃C at the 6.67%C end of the diagram. The left side of the diagram is pure iron combined with carbon, resulting in steel alloys. Three significant regions can be made relative to the steel portion of the diagram. They are the

eutectoid E, the hypoeutectoid A, and the hypereutectoid B. The right side of the pure iron line is carbon in combination with various forms of iron called alpha iron (ferrite), gamma iron (austenite), and delta iron. The black dots mark clickable sections of the diagram.

Allotropic changes take place when there is a change in crystal lattice structure. From 2802°-2552°F the delta iron has a **body-centered** cubic lattice structure. At 2552°F, the lattice changes from a body-centered cubic to a face-centered cubic lattice type. At 1400°F, the curve shows a plateau but this does not signify an allotropic change. It is called the Curie temperature, where the metal changes its magnetic properties.

Two very important phase changes take place at 0.83%C and at 4.3% C. At 0.83%C, the transformation is eutectoid, called **pearlite**. gamma (austenite) \longrightarrow alpha + Fe₃C (cementite) At 4.3% C and 2066°F, the transformation is eutectic, called **ledeburite**. L(liquid) --> gamma (austenite) + + Fe₃C (cementite)

Definitions

Eutectoid:	A eutectoid system occurs when a single-phase solid transforms directly to a two- phase solid.	
Hypereutectoid: Hypereutectoid systems exist below the eutectoid temperature.		
Hypoeutecto	id: Hypoeutectoid systems exist above the eutectoid temperature.	
Ferrite:	Body-centered cubic iron or an iron alloy based on this structure.	
Austenite:	Face-centered cubic iron or an iron alloy based on this structure.	
Delta iron:	The body-centered cubic phase which results when austenite is no longer the most Stable form of iron. Exists between 2802 and 2552 degrees F. has BCC lattice structure and is magnetic.	
Body-center	ed: A structure in which every atom is surrounded by eight adjacent atoms, whether the atom is located at a corner or at the center of a unit cell.	
Face-centere	d: A structure in which there is an atom at the corner of each unit cell and one in the center of each face, but no atom in the center of the cube.	
Pearlite:	A lamellar mixture of ferrite and carbide formed by decomposing austenite of Eutectoid composition.	
Cementite:	The second phase formed when carbon is in excess of the solubility limit.	
Ledeburite:	Eutectic of cast iron. It exists when the carbon content is greater than 2 percent. It contains 4.3 percent carbon in combination with iron.	

4.2 Classification of Steel : The steels are classified by various methods and each method is based on a definite criteria as follows,

- i) Amount of carbon
 - *a) Low carbon steels* (0.008 0.3%C)
 - *b) Medium carbon steels* (0.30 0.60%C)
 - c) High carbon steels (0.60 2.00%C)
- ii) Amount of alloying elements and carbon
- iii) Amount of deoxidation
- iv) Method of manufacture v) Form and use.

5. PROCEDURE:

The preparation of metallic or other materials for microscopic examination and micro structural characterization is in principal very simple. There are four basic processes that you will need to become familiar with: sample cutting and sectioning, metallographic mounting, Surface grinding and surface polishing.

5.1 Sample Cutting and Sectioning

5.1.1. Sectioning

Sectioning means removal of convenient size specimen from large sample with minimal damage to microstructure with the help of abrasive cut off machine. Abrasive cutting wheel/saw is attached to cutting machine and for work piece holding proper vice is provided on machine. The primary concern in this process is to minimize the heating of the sample due to the cutting. For this reason, the cut-off saws that is equipped with either water-cooling systems.

5.2 Mounting

If sample is large enough (about 25 mm square or larger) than do not need to mount it, as it will be able to control the sample during polishing without a mount. For smaller samples there are two basic mounting techniques used in this laboratory. Mounting facilitates handling during preparation and handling. It also avoids damage to polishing wheels during polishing. The most common uses a thermosetting plastic compound (Bakelite) to encapsulate the specimen known as hot-mounting process, and the second uses a room temperature curing epoxy known as cold-mounting process. The Bakelite mounting is by far the most common and easiest. The room temperature curing epoxy mount should only be used for samples that are extremely sensitive to heat.

The Bakelite process uses a sample mounting press that applies a pressure to the Bakelite/sample system during the cure to remove voids and gaps and to fully fill the sample spaces. Bakelite comes in a variety of colors, which can be combined to produce easy sample identification.

5.2.2 The procedure for Hot mounting process.

The following process is used to encapsulate your specimen in Bakelite:

- 1) Place sample face down on the small piston inside the press, and lower the piston into the cylinder by opening slightly the valve on the front of the press.
- 2) Approximately three tablespoons of Bakelite is poured over the sample, and the top of the press gently screwed into place. DO NOT TIGHTEN THE TOP OF THE PRESS. It is only necessary to engage all of the screw threads; you do not have to tightly secure the top.
- 3) The cylindrical heater is plugged in and turned on (the red light should turn on). Place the

heater around the mold. The heater is thermostatically controlled and will heat the mold to about 135-150 °C. Close the valve, and pump up the cylinder using the hand lever. As the Bakelite heats, it will begin to flow to fill the void spaces, and the pressure will drop. Maintain the constant.

- 4) When the pressure stops rapidly dropping, the whole mold has reached 150°C. Begin timing for 5-7 minutes to fully cure the Bakelite. Maintain the pressure during the entire heating and cooling cycle.
- 5) At the end of the heating cycle, remove the heater and place the cooling collar on the mold for an additional 6-8 minutes.
- 6) Crack the valve to release the pressure, and unscrew the top of the mold. When the mold top is fully unscrewed (it may not come out due to adhesion with the Bakelite), close the valve and slowly pump up the cylinder to push the sample fully out of the press. Mark the sample on the back.
- 7) Clean any residual Bakelite off of the mold surfaces.

5.2.2 The procedure for Cold Mounting process.

The following process is used to room temperature curing epoxy process:

- 1) Apply mold release agent to mold. Place specimen in the mold.
- 2) Mix epoxy powder and bonding liquid in 1:2 ration in a cup.
- 3) Pour into mo Id... wait for 10 minutes.
- 4) Eject the mold.

5.3 Sample Surface Polishing

The goal of the surface polishing is to end up with a planar cross section of sample free from scratches or disturbed metal introduced by the cutting and sectioning. This process is a step-wise process that can be broken into three loosely separate parts: grinding, coarse polishing, and final polishing.

5.3.1. Grinding

The first step in preparing your sample is to ensure that you have a flat surface to begin with. A water-cooled abrasive grinder is available to form a flat initial surface from which to begin. After getting a flat sample on the belt grinder, WASH sample thoroughly. The hand lapping station has four graded abrasive papers to produce a sequentially finer surface finish. Be sure the water is turned on and flowing uniformly over the abrasives. Start with the coarsest grit (240) and, using a firm and uniform pressure, slowly move the specimen forward and back across the abrasive. This will produce parallel scratches of uniform size. Continue this step until the entire surface of your sample is flat and contains only scratches of the size of 240 grit abrasive. When the sample is flat and the only scratches remaining are those due to the 240 grit abrasive, WASH your sample and your hands thoroughly, and move to the 320 grit abrasive. Repeat this procedure for the 400 grit and the 600 grit abrasive, checking after each step to be sure that only those scratches remain that are due to the smallest grit.

5.3.2 Rough Grinding

Before proceeding to the first polishing wheel (leftmost wheel), wash sample with water.

- 1) First, apply a small amount of water to the wheel, turn on the motor, and gently clean off the wheel with your fingers.
- 2) Apply a small amount of abrasive slurry to the wheel. This wheel uses an AI203 abrasive in a water suspension. The abrasive particles are 5 micrometers in diameter.
- 3) Carefully place your sample on the wheel while gripping it tightly. Slowly move the sample in a circular motion against the rotation of the wheel. Use a moderate and even pressure. It is important to ensure that you keep the sample flat on the wheel so that the final surface will be completely planar.
- 4) After several minutes on the wheel, hold the sample in one place for a moment. This will provide lots of parallel scratches that you can use to determine if you have removed the damage from the grinding steps.
- 5) Examine the sample under the microscope to determine if all the scratches are the same size.
- 6) Repeat steps 1-5 on the middle polishing wheel. This wheel uses a 0.3 micrometer A1203 abrasive in a water suspension.

5.3.3. Final Polishing

- 1) Repeat steps 1-5 above on the right polishing wheel. This wheel uses a 0.05 micrometer A1203 abrasive in a water suspension. At this point, the sample will be very smooth to the eye and even the oils and dirt on your fingers will scratch it with larger scratches than the abrasive. DO NOT TOUCH THE SAMPLE SURFACE FROM THIS POINT ON.
- 2) The last step in the process is to etch the sample to bring out the microstructure.
- 3) Use a cotton swab and a petri dish for the etching. Gently swab the surface of your sample with the etchant. Roughly spreading the etchant will scratch your surface. Let the etchant stand for 15 seconds or so and rinse the sample with water to stop the etching, and rinse again with methanol. Rinse the swab with water and throw into the trash bin.
- 4) Examine specimen under the microscope. You may require several etching steps to bring out the microstructure.
- 5) If the sample is over-etched, repeat the final polishing step and re-etch for a shorter time. Samples to be examined at high magnification generally require shorter etching times than those to be viewed at lower magnifications.
- * After last polishing stage the sample looks mirror like.

5.4 Etching

Grains cannot be seen without etching. Cracks, pores and defects are observed without etching. Etchant reacts with atoms and dissolves them. Atoms at grain boundaries dissolve quickly.

Dissolved grain boundaries appear dark.

Steps:

1) Apply enchant to polished surface for some time

2) Rinse with distilled water

Enchants:

- 1) 50/50 HCI: equal parts hydrochloric acid (HCI) mixed with water.
- 2) Alcoholic Ferric Chloride: 5 grams FeC13; 2 ml concentrated HCI acid; 95 ml methyl alcohol.
- 3) Aqueous Ferric Chloride: 10 grams FeC13; 20 ml concentrated HCI acid; 80 ml water.
- 4) Ammonia/Hydrogen peroxide: 1 part strong ammonia; 1 part hydrogen peroxide; 2 parts water; FRESHLY MADE.
- 5) Mixed acids: 95 ml water; 1.5 ml concentrated HCI acid; 2.5 ml concentrated nitric (HN03) acid; 0.5 ml hydrofluoric (HF) acid.
- 6) 2% Nital: 2 ml concentrated HN03; 98 ml methyl alcohol.

5.5. Metallographic Observation

Observe microstructure, Place specimen on metallograph and adjust magnification, focus and position s adjust micro High magnification - to study phases and Low magnification -to study grain size.

5.5.1 <u>Microphotography</u>

In this laboratory, you will report the microstructures of prepared samples in specific formats. You will be expected to sketch the microstructure that you see under the microscope by hand. In sketching the microstructure there are several things to keep in mind. First, the magnification that you use depends upon the scale of the microstructure you are looking for. It is IMPORTANT to know in advance of the lab class what the expected microstructure for your samples are **and** at what scale they should appear. In sketching the microstructure, you should indicate only the important features of the structure that you observe-don't make a photographic reproduction of the microstructure. Simple sketches show that you know what the important structures are and have identified them in the cross section.

An example of what is considered to be a good laboratory report sketch of the microstructures is included in the appendix. Your sketch MUST INCLUDE:

- 1) The sample name and composition,
- 2) The metallurgical history of the sample,
- 3) A simple sketch of the important microstructure indicating
 - a) The magnification used (i.e. "MAG= 100 X"),
 - b) Important phases and features noted,
- 4) Etchant used.

6. OBSERVATION:



7.PRECAUTIONS:

There are several general instructions to keep in mind during this part of the process.

- 1) CLEANLINESS!!! Keep the room and the work areas clean, especially the polishing area. Each step in the polishing process uses a finer grade of abrasive, so good results require that both your sample and your hands are free from abrasive at each stage before proceeding to the next stage of the process. Turn the power off and cover the wheels when you are through. Clean up any spilled water and wipe up the countertop.
- 2) Throw away the leftover etchants into the sink with a liberal amount of water. Swabs should be rinsed and thrown into the trash **bin** IMMEDIATELY AFTER USE. Wash your containers with water, rinse in methanol, and place them on the shelf above the sink.
- 3) Use goggles and gloves while handling chemicals. Contact the T.A. or the Instructor in the case of an acid spill, or if new etchants are needed. Be extra careful when using hydrofluoric acids.
- 4) Do not eat or drink anything while working in the lab. Wash your hands thoroughly with soap before leaving the lab.

Symptom	Cause	Action
Specimen has fine matte like finish	This layer of particles on the surface	 Clean surface swab 110% cotton swab and alcohol. Clean specimen by flushing specimen and polishing pad with water for last 10-15 seconds of the polishing operation.
Excessive relief in surface finish	Over polishing	 Repeat step prior to final polish and shorten final polishing time
Scratches in specimen	 Improper selection of polishing and Contaminated pad Removal of secondary phases 	 Review application guideline chart for polishing pads or select a softer pad Replace polishing pad Alternate between polishing and etching
Etching of specimen	 Alumina abrasive pH too high 	Use lower pH alt

7. QUESTIONS:

- i) Why alloying elements are added to steels?
- ii) How negative effects of sulphur in steels will be neutralized?
- iii) What is the composition of stain less steel?
- iv) What are the important characteristics of Tool steels?
- v) What is the composition of H.S.S.?
- vi) What makes High Carbon high chromium steel suitable for making dies?
- vii) Show the heat treatment cycles, on Time-temperature diagrams for different types of steels?
- viii) Compare the properties of alloy steels with and without heat treatment?

EXPERIMENT - 3

- AIM: 1. Study of the Microstructures of Cast Iron.
- 2. **Objectives:** To understand what Microscopy is, and how it can be used to a.) observe Microstructure of metals like cast iron.
- b) To conduct typical engineering microscopic observation and be able to recognize the microstructure of typical type of cast iron metal.

c) To learn the differences in microstructure of different type of cast irons.

- 3. **APPERATUS:** Metallurgical Microscope, Standard Specimens of Cast Iron materials.
- Metallurgical Microscope. 3.1



Binocular model Eye pieces: 10X,15X & 20X Magnification: 10X-500X.

> This Microscope is used specimen with the adjustable

Fig. 1

4. THEORY:

4.1 Cast Iron

Cast irons typically contain 2-4 wt% of carbon with a high silicon concentrations and \mathbf{a} greater concentration of impurities than steels. The carbon equivalent (CE) of a cast iron helps to distinguish the grey irons which cool into a microstructure containing graphite and the white irons where the carbon is present mainly as cementite. The carbon equivalent is defined as: St4-P

$$CE(wt\%) = C + \frac{Si + P}{3}$$

The term cast iron, like the term steel, identifies a large family of ferrous alloys. Cast irons are multi component ferrous alloys. They contain major (iron, carbon, silicon), minor (<0.01%), and often alloying (>0.01%) elements. Cast iron has higher carbon and silicon contents than steel. Because of the higher carbon content, the structure of cast iron, as opposed to that of steel, exhibits a rich carbon phase. Depending primarily on composition, cooling rate and melt treatment, cast iron can solidify according to the thermodynamically metastable Fe-Fe₃C system or the stable Fc- Gr system. When the metastable path is followed, the rich carbon phase in the eutectic is the iron carbide; when the stable solidification path is followed, the rich carbon phase is graphite. Referring only to the binary Fe-Fe₃C or Fe-Gr system, cast iron can be defined as an iron-carbon alloy with more than 2% C. Important notice is that silicon and other alloying elements may considerably change the maximum solubility of carbon in austenite (g). Therefore, in exceptional cases, alloys with less than 2% C can solidify with a eutectic structure and therefore still belong to the family of cast iron.

4.2 Types of Cast Iron

On the basis of Microstructure cast irons are classified as follows.



4.2.1 White Cast iron

Composition of the iron is appropriate or the cooling rate of the metal is sufficiently rapid during solidification, the metal will solidify with the C combined with iron as iron carbide. This compound, also called cementite, is hard and brittle and dominates the microstructure of white iron. Thus, white iron is hard and brittle and has a white crystalline fracture because it is essentially free of graphite.

White iron has a high compressive strength and excellent wear resistance, and it retains its hardness for limited periods even up to a red heat. It can be produced in selected areas of a casting—such as. on the periphery of a cam—by causing localized rapid solidification of the iron. White iron at the surface of a casting is called chill. It is produced by making that portion of the mold—where the white iron is desired—of-a material that can extract heat very rapidly, such as iron or graphite. White iron does not have the easy castability of other irons because its solidification temperature is generally higher, and it solidifies with C in its combined form as iron carbide. Application includes rollers of rolling mills, Dies of metal extrusion and where high wear resistance is necessary.



Graphite Flakes



4.2.1 Gray Cast Iron

When the composition of the molten iron and its cooling rate are appropriate, the C in the iron separates during solidification and forms separate graphite flakes that are interconnected within each eutectic cell. The graphite grows edgewise into the liquid and forms the characteristic flake shape. When gray iron is broken, most of the fracture occurs along the graphite, thereby

accounting for the characteristic gray color of the fractured surface. Because the large majority of the iron castings produced are of gray iron, the generic term, cast iron, is often improperly used to mean gray iron specifically.

The properties of gray iron are influenced by the size, amount and distribution of the graphite flakes, and by the relative hardness of the matrix metal around the graphite. These factors are controlled mainly by the C and Si contents of the metal and the cooling rate of the casting. Slower cooling and higher C and Si contents tend to produce more and larger graphite flakes, a softer matrix structure and lower strength. The flake graphite provides gray iron with unique properties such as excellent machinability at hardness levels that produce superior wear-resisting characteristics, the ability to resist galling and excellent vibration damping.

The amount of graphite present, as well as its size and distribution, are important to the properties of the iron. Whenever possible, it is preferable to specify the desired properties rather than the factors that influence them.4.2.2

Nodular cast Iron (Ductile iron, S. G Iron)

Ductile iron also referred to as nodular iron or spheroidal graphite iron, was patented in 1948. After a decade of intensive development work in the 1950s, ductile iron had a phenomenal nine-fold increase in use as an engineering material during the 1960s, and the rapid increase in commercial application continues today. An unusual combination of properties is obtained in ductile iron because the graphite occurs as spheroids rather than as individual flakes as in gray iron. This mode of solidification is obtained by adding a very small, but specific, amount of Mg to molten iron of a proper composition. The base iron is severely restricted in the allowable contents of certain minor elements that can interfere with the graphite spheroid formation. The added Mg reacts with the sulfur and oxygen in the molten iron and changes the way the graphite is formed. Control procedures have been developed to make the processing of ductile iron dependable. The high C and Si content of ductile iron provide the casting process advantages, but the graphite spheroids have only a nominal influence on the mechanical properties of the metal. Ductile iron, like malleable iron, exhibits a linear stress-strain relation, a considerable range of yield strengths and, as its name implies, ductility. Castings are made in a wide range of sizes with sections that can be either very thin or very thick.

The different grades are produced by controlling the matrix structure around the graphite either ascast or by subsequent heat treatment. Only minor compositional differences exist among the regular grades, and these adjustments are made to promote the desired matrix microstructures. Alloy additions may be made to ductile iron to assist in controlling the matrix structure as-cast or to provide response to heat treatment. Special analysis ductile irons and high-alloy ductile irons provide unusual properties for special applications.

4.2.3 Malleable Cast Iron

This type of iron is characterized by having the majority of its C content occur in the microstructure as irregularly shaped nodules of graphite. This form of graphite is called temper carbon because it is formed in the solid state during heat treatment. The iron is cast as a white iron of a suitable chemical composition. After the castings are removed from the mold, they are given an extended heat treatment starting at a temperature above 1650°F (900°C). This causes the iron carbide to dissociate and the free carbon precipitates in the solid iron as graphite. The rapid

solidification rate that is necessary to form the white iron limits the metal thickness in the casting that is practical for the malleable iron process.

A wide range of mechanical properties can be obtained in malleable iron by controlling the matrix structure around the graphite. Pearlitic and martensitic matrices are obtained both by rapid cooling through the critical temperature and with alloy additions. Malleable irons containing some combined carbon in the matrix often are referred to as pearlitic malleable, although the microstructure may be martensitic or a spheroidized pearlite.



Fig. 4 Microstructure Nodular Cast Iron



Fig. 5. Microstructure Malleable Cast Iron



Fig. 6 Iron-Carbon diagram (cast iron portion)

4. PROCEDURE:

In this laboratory, you will report the microstructures of prepared samples in specific formats. You will be expected to sketch the microstructure that you see under the microscope by hand. In sketching the microstructure there are several things to keep in mind. First, the magnification that you use depends upon the scale of the microstructure you are looking for. It is IMPORTANT to know in advance of the lab class what the expected microstructure for your samples are and at what scale they should appear. In sketching the microstructure, you should indicate only the important features of the structure that you observe-don't make a photographic reproduction of the' microstructure. Simple sketches show that you know what the important structures are and have identified them in the cross section.

An example of what is considered to be a good laboratory report sketch of the microstructures is included in the appendix. Your sketch MUST INCLUDE:

- 1) The sample name and composition,
- 2) The metallurgical history of the sample,
- 3) A simple sketch of the important microstructure indicating
 - a) The magnification used (i.e. "MAG= 100 X"),
 - b) Important phases and features noted,
- 4) Etchant used.

5. **OBSERVATIONS:**



6. **PRECAUTIONS:**

There are several general instructions to keep in mind during this part of the process.

- 6.1 CLEANLINESS!!! Keep the room and the work areas.
- 6.2 Don't touch etched and polished surface of the specimen.
- 6.3 Don't touch the lances of eyepiece with dirty hand.
- 6.4 Use clean clothes only to clean the lenses of eyepieces.
- 6.5 Handle the microscope with gently.
- 6.6 Return the standard specimen to the Lab Technician after observation.
- 6.7 Switch off the microscope after the observation.

7, Review Questions:

- i) What are the important alloys of Copper & Zinc?
- ii) What is composition of Muntz metal?
- iii) What is the composition of Cartridge Brass?
- iv) What is a Bronze?
- v) What is composition of Gunmetal?
- vi) What are the important applications of Gunmetal?
- vii) What is a Bell metal?.
- viii) What is melting point of Tin?
- ix) What is use of Babbit metals? Explain why?
- x) What is the microstructure of Tin based Babbit?

EXPERIMENT-4

- 1. **AIM:** Study of the Microstructures of Non Ferrous Metals.
- 2. **Objectives:** a.) To understand what Microscopy is, and how it can be used to observe Microstructure of non-ferrous metals.
- b) To conduct typical engineering microscopic observation and be able to recognize the microstructure of typical type of copper, aluminum and its alloys.
- c) To learn the differences in microstructure of different type of cast irons.
- APPERATUS: Metallurgical Microscope, Specimens of Non Ferrous Metals.
 3.1 Metallurgical Microscope.



Binocular model

- Eye pieces: 10X, 15X & 20X
 - Magnification: 10X 500X. This Microscope is used for microscopic observation of specimen with the adjustable magnification.

4. THEORY:

4.1 COPPER AND COPPER ALLOYS

4.1.1 COPPER -: The properties of copper are high electrical and thermal conductivity, good. Corrosion resistance, machinability, strength and ease of fabrication.

Copper properties:-

- Its melting pt is 1083C
- Specific gravity is 8.9.
- Has high electrical and thermal conductivity.
- Its good ductility and malleability r due to its FCC structure.
- Cu containing 0.3% As is called as ARSENICAL Cu
- Cu containing 0.6% Al is called as FREE CUTTING Cu.

4.1.2 COPPER ALLOYS.

There are two types of Cu alloys popularly known:- BRASS AND BRONZE.

BRASS:- Cu + Zn = Brass.

• Brass are again divided as alpha brass and alpha + beta brass.

// TYPES OF ALPHA BRASS //

1) CAP BRASS:-

- Consists of 2-5% Zn'
- Very very ductile alloy.
- Zn is used as deoxidiser for deoxidation of Cu.
- Used for cap of detonation..

2) GUILDING METAL:-

- Zn-(5-15%).
- Has color of gold.
- Uses-artificial jewellary, condenser tubes, coils, needles, etc.

3) CARTRIDGE BRASS:-

- Known as (70-30) brass
- Has max ductility and malleability of all brasses.
- Applications=radiator fins, lamp fixtures, rivets, springs, etc.

4) ADMIRALITY BRASS: -

- 1% Sn is added to cartridge brass to improve corrosion resistance.
- Uses in condenser tubes and heat exchangers.
- Containing 22%Zn,2%Al,0.04%Sn is used in marine applications.

//(ALPHA +BETA) BRASS//: -

- Contains up to (32-40)%Zn.
- Hard, strong and less corrosion resistance compared to alpha brasses.

1) MUNTZ METAL:-

- Contains 40%Zn.
- Alloy becomes single phase at about 700C
- Also called as (60-40)brass.
- Uses in utensils, shafts, nuts, bolts, condenser tubes, etc.

2) NAVAL BRASS:-

- ADDING 1%Sn to muntz metal inc corrosion resistance to marine environment
- Also called as TOBIN BRONZE.

3) LEADED BRASS: -.

- Lead added in small amount (1-3)%to improve machinability.
- Appears as globules in microstructure.

// BRONZE // (Cu-Sn alloy)

1) Al BRONZE: -

- An alloy **of Cu** and Ni.
- Max solubility of Cu in Ni is 9.4%
- Has good ductility, strength, toughness, corrosion resistance and fatigue resistance.
- Called as IMITATION GOLD.
- Applications "-components of valves, pump castings, park plug body.

2) TIN BRONZE:-

- ALLOY OF Cu nd Sn.
- Tin has affinity towards oxyzen. the tin oxide formed reduces ductility nd malleability. Zn or P are added to deoxidize these.

COINAGE BRONZE

Contains 5%Sn and 1%Zn.

- It is a ductile metal, having better formability and strength..
- Application=manufacturing of coins.

GUN METEL

- Contains 10%Sn and 2% Zn.
- Known as (10-2) bronze.

1) PHOSPHOROUS BRONZE:-

- Alloy of Cu and Zn with P.
- Phosphorous is a strong deoxidizer and helps in inc fluidity.
- Phosphorous content is max upto 1% depending on application,
- (2.5-8)% Zn,(0.1 -0.35)%P known as WROUGHT PHOSPHOROUS BRONZE.
- (5-13)% Zn and (0.3-1)%P is known as CAST PHOSPHOROUS BRONZE.

2) MONEL METAL:-

- ALLOY OF Cu and Ni.
- Cupronickel metal alloy consists of 15-30%Ni.
- Cu and Ni are completely soluble into each other in both solid in liquid solution.
- Application—blades of turbine, coins, bullet envelops.

ALUMINIUM

Properties:

- Light in wt.
- Castability and formability arc better.
- Corrosion resistance of Al is excellent.
- Powerful deoxidiser.
- Carries more electricity than Cu.
- Costlier metal in the family of light metal alloys.
- Has good electrical and thermal conductivity.

A1 ALLOYS

1) Al-Si-Cu: -

- LM2,LM6, LM8, LM13.
- Used for production of castings, due to their excellent fluidity nd casting characteristics.
- Higher Si content better are the mechanical properties.

2) Al-Mg (MAGNALIUM ALLOY):-

- LM5NDLM10.
- Used in marine environments. Due to its high strength nd resistant to corrosion.
- Has good surface finish.

3) Al-Cu (DURALIUM): -

- LM11 (CU-4.5%)
- Susceptible to hot tearing. Applications are casted components used in aircrafts.

4) Y ALLOY (HIGH STRENGTH AI ALLOY):-

- LM14.
- CONTAINS 4%Cu, 2% Ni, and 1.5%Mg.
- Application=heavy-duty petrol engine, piston block, etc.

5) RR350 (HINDUMINIUM):-

- Contains 5%Cu, 1.5%Ni with small amounts of Mn, Ti, Sb, Co, Zr.
- Used in aero engines and other continuous elevated temp service applications upto 300°C.

4. PROCEDURE:

In this laboratory, you will report the microstructures of prepared samples in specific formats. You will be expected to sketch the microstructure that you see under the microscope by hand. In sketching the microstructure there are several things to keep in mind. First, the magnification that you use depends upon the scale of the microstructure you are looking for. It is IMPORTANT to know in advance of the lab class what the expected microstructure for your samples are and at what scale they should appeal'. In sketching the microstructure, you should indicate only the important features of the structure that you observe-don't make a photographic reproduction of the microstructure. Simple sketches show that you know what the important structures are and have identified them in the cross section.

An example of what is considered to be a good laboratory report sketch of the microstructures is

- included in the appendix. Your sketch MUST INCLUDE:
- 1) The sample name and composition,
- 2) The metallurgical history of the sample,
- 3) A simple sketch of the important microstructure indicating
 - a) The magnification used (i.e. "MAG= 100 X"),
 - b) Important phases and features noted,
- 4) Etchant used.

5. **OBSERVATION:**

6. PRECAUTIONS:

There are several general instructions to keep in mind during this part of the process.

- 6.8 CLEANLINESS!!! Keep the room and the work areas.
- 6.9 Don't touch etched and polished surface of the specimen.
- 6.10 Don't touch the lances of eyepiece with dirty hand.
- 6.11 Use clean clothes only to clean the lenses of eyepieces.
- 6.12 Handle the microscope with gently.
- 6.13 Return the standard specimen to the Lab Technician after observation.
- 6.14 Switch off the microscope after the observation.

7, Review Questions:

- i) What are the important alloys of Copper & Zinc?
- ii) What is composition of Muntz metal?
- iii) What is the composition of Cartridge Brass?
- iv) What is a Bronze?
- v) What is composition of Gunmetal?
- vi) What are the important applications of Gunmetal?
- vii) What is a Bell metal?.
- viii) What is melting point of Tin?
- ix) What is use of Babbit metals? Explain why?
- x) What is the microstructure of Tin based Babbit?

EXPERIMENT – 5

1. **AIM:** Study of the Microstructures of Heat Treated Steels.

2 **OBJECTIVE.**

- a. To observe microstructure of heat traded steels.
- b. To study the changes in macrostructure due to heat Treatment.
- c. To study the one of the heat treatment process
- 3. APPERATUS:

1) Specimens of High Carbon Steel subjected

- II) Metallurgical Microscope. **Binocular model**
- Eye pieces: 10X, 15X & 20X
- Magnification: 10X-500X.

This Microscope is used for microscopic observation of Specimen with the adjustable magnification.

4. THEORY:

4.1 **Heat Treatment** is *the* controlled heating and cooling of metals to alter their physical and mechanical properties without changing *the product shape*. Heat treatment is sometimes done inadvertently due to manufacturing processes that either heat or cool the metal such as welding or forming.

Heat Treatment is often associated with *increasing the strength of material*, but it can also be used to *alter certain manufacturability* objectives such as improve machining, improve formability, and restore ductility after a cold working operation. Thus it is a very enabling manufacturing process that can not only help other manufacturing process, but can also improve product performance by increasing strength or other desirable characteristics.

Steels are particularly suitable for heat treatment, since they respond well to heat treatment and the commercial use of steels exceeds that of any other material. Steels are heat treated for one of the following reasons: <u>Softening</u>, <u>Hardening</u>, <u>Material Modification</u>

4.2 Heat Treatment Processes:

4.2.1 Annealing

The process consists of heating the steel to above A3 temperature for hypo-eutectoid steels and above A1 temperature for hypereutectoid steels by 30-50°C, holding at this temperature for a definite period and slow cooling to below A1 or to room temperature in the furnace. Due to slow cooling, eutectoid phase transformation occurs very nearly in accordance with conditions represented by Fe-C phase diagram.

Purpose of Annealing -:

- To relive the internal stresses induced due to cold working, welding.
- To reduce hardness and to increase ductility.
- To refine the grain size.

- To make the material homogeneous in respect of chemical composition.
- To increase machinability
- To increase the uniformity of phase distribution and to make the material isotropic in respect to mechanical properties.

Types of Annealing -:

i.Full Annealing	iv.	Box annealing
ii.Spheroidized Annealing	v.	Isothermal Annealing
iii.Bright Annealing		

4.2.2. Normalizing

The process consists of heating the steels above the upper critical temperature (A3) for hypoeutectoid steels, and above Acm for hypoeutectoid steels by 30 to 50°C, holding long enough at this temperature for homogeneous austenitization and cooling to room temperature in air. Due to air cooling which is slightly fast as compared to furnace cooling employed to annealing, normalized components show slightly different structure and properties than annealed components. After normalizing, microstructure shows more peralite than observed in annealed components.

Purpose -:

- This process is used to eliminate the cementite network which formed due to slow cooling in the temperature range from Acm to **al**.
- To relieve internal stresses.
- To make the material homogeneous in respect of chemical composition.
- To increase machinability
- To increase the uniformity of phase distribution and to make the material isotropic in respect to mechanical properties.

4.2.3 Hardening:

The hardening process consists of heating the steel to above A3 temperature for hypoeutectoid steels and above A1 temperature.

Draw hardening cycle.

4.2.4 Tempering:

Tempering is fallowed to reduce the effect of retained austenite in which the hardened steel is reheated below the lower critical temperature and cooled slowly.

4.3 Time — Temperature — Transformation Diagram.

Draw the Time — Temperature — Transformation Diagram.

5 **PROCEDURE:**

In this laboratory, you will report the microstructures of prepared samples in specific formats. You will be expected to sketch the microstructure that you see under the microscope by hand, in sketching the microstructure there are several things to keep in mind. First, the magnification that you use depends upon the scale of the microstructure you are looking for. It is IMPORTANT to know in advance of the lab class what the expected microstructure for your samples are and at what scale they should appear. In sketching the microstructure, you should indicate only the important features of the structure that you observe-don't make **a** photographic reproduction of the microstructure. Simple sketches show that you know what the important structures are and have identified them in the cross section.

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- 2) The metallurgical history of the sample,
- 3) A simple sketch of the important microstructure indicating
 - a) The magnification used (i.e. "MAG= 100 X"),
 - b) Important phases and features noted,
- 4) Etchant used.

6. **OBSERVATION:**



7. **Review Questions:**

- i) What is the Annealing temperature range of Hypo eutectoid steels?
- ii) What is the hardening temperature range of Hyper eutectoid steels?
- iii) Why hardened steel specimens are subjected to tempering?
- iv) What is the normalizing temperature range of Hyper eutectoid steels?
- v) How the soaking time in furnace is decided? Mention the times required for 1 cm thickness, 5 cm thickness, 10 cm thickness etc.
- vi) Explain the properties of Hypo eutectoid, eutectoid, Hyper eutectoid steels, before and after heat treatments?
- vii) Slow Time Temperature diagram for different types of plain carbon steels?

EXPERIMENT - 6

1. AIM: To find out the Hardness of various treated and untreated steels.

2. **OBJECTIVES:**

- 1. To understand what hardness is, and how it can be used to indicate some properties of materials.
- 2. To conduct typical engineering hardness tests and be able to recognize commonly used hardness scales and numbers.
- 3. To learn the advantages and limitations of the common hardness test methods.

3. EQUIPMENTS*



Rockwell Standard Hardness Tester Maximum Test height - 295mm Depth of the throat - 150mm Diamond Dial Indentor- 120 deg cone angle Maximum load -] 50 Kgf

Rockwell Calibration Test Blocks (C scale), Hardness Test Specimens of various metal.

4. THEORY:

A commonly accepted engineering definition of hardness is the resistance to indentation. Resistance to indentation is a function of the mechanical properties of the material, primarily its elastic limit and to a lesser extent, its work-hardening tendency, and the modulus of elasticity. For a given composition with a known history it is possible to relate the elastic limit (for practical purposes, the yield strength) to the tensile strength, ductility, and toughness. Hence, the hardness tests can provide information from which many important mechanical properties can be derived. Since the hardness test can be conducted easily and quickly, they are very popular and are used to control processing and for inspection and acceptance of materials and components.

The common hardness tests rely upon the slow application of a fixed load to an indenter, which is forced into the smooth surface of the specimen. Upon removal of the load either the area or the depth of penetration is measured as an indication of resistance to the load. Three types of tests are discussed below,

4.1 Rockwell Tests:

The Rockwell tests depend upon the measurement of the differential depth of a permanent deformation caused by the application and removal of differential loads. Various penetrator and load combinations are used to adapt different Rockwell tests to materials of varying hardness and thickness. The penetrators include a cone-shaped diamond, known as a Brale, and hard steel balls from 1/16-inch to 1/2 -inch in diameter.

4.1.1 Standard Rockwell Test:

The Standard Rockwell tests use a light load of 10 Kg to seat the penetrator firmly in the surface of the specimen. This load is known as the minor load. After the application of the minor load, the depth gauge is zeroed and a larger load, known as major load, is applied and then removed. While the minor load still acts, the depth of permanent penetration is measured. The depth gauge, which measures the penetration, is calibrated to read in hardness numbers directly rather than in inches. Major loads for Standard Rockwell tests are 60, 100 or 150 Kg. The diamond penetrator is marked as "C-Brale".

4.1.2. Superficial Rockwell Test:

Superficial Rockwell tests are used for measuring the hardness of thin specimens and specimens which have only a thin hardened surface layer) known as a case) on a soft base (known as a core). The ball penetrators available for superficial testing are the same as for standard testing. The diamond brale is marked as "N-Brale". The loads for superficial testing are much lower than for standard testing, being 3 Kg for the minor load and 15, 30 or 45 Kg for the major load.

The wide range of combinations of penetrators and loads permit application of Rockwell Test to an equally wide range of materials of varying hardness. The diamond penetrator makes it practical to test the hardest steels and the large balls permit testing of soft metals and even plastics. Generally the Rockwell test is considered to be nondestructive because the light loads and small penetrators produce very small impressions. However, because of the small impressions several readings should be taken to obtain a representative result. Furthermore, the smaller the impression, the greater is the care necessary in preparing the surface. Apart from any special effort required for surface preparation, the Rockwell test is easier and more quickly performed than the Brinell test.

5. BRINELL TEST:

The Brinell test relies on mechanical or hydraulic loads as large as 3000 Kg. acting through a 10 mm hard steel or carbide ball. In order to compensate for variations in the response of materials to the application of the load, the time for which the load is applied is specified. For hard materials such as steel, a 30-second loading period is adequate. Softer metals and alloys such as brass or aluminum require about 60 seconds. After the load is removed, the diameter of the impression made by the ball is measured in millimeters. The Brinell hardness number, abbreviated as BHN, is the quotient of the load, P (kg), divided by the area, A, of the impression:

$$BHN = \frac{P}{(D - (D^2 - d^2)^{1/2})pD/2}$$

Where D is the diameter of the ball penetrator (mm) and d is the diameter of the impression (mm). In practice, the **BHN** is read directly from a table listing different values of d for various values of load, P.

The Brinell test makes a large impression on the surface of the piece tested. Unless such a large impression can be tolerated, and often it cannot, the test is destructive. However, the large impression is advantageous because it gives a more representative result than would a smaller impression, which would be more sensitive to local soft or hard inhomogeneties. The size of the impression also renders the test less sensitive to the presence of rough surface finish and mill scale than is the case when tests are used which rely on small indentations.

6. **PROCEDURE:**

For carrying out test the following procedure should be adopted very carefully, any negligence may lead damage to the Indentor.

- 1. Adjust the weights on plunger (3) or dashpot according to the Rockwell scale required as shown in chart on machine by load selection disc (13).
- 2. Keep the lever (9) at position 'A'.
- 3. Place specimen securely on testing table.
- 4. Turn the hand wheel (10) clockwise so that specimen will push the Indentor and show a reading on dial gauge (6) as small pointer at set (red spot) and long pointer close to 'O' of outer scale (i.e. 'B' 30 inner scale).
- 5. Turn the lever (9) from position A to B slowly so that the total load is brought into action without any jerks.
- 6. The long pointer of dial gauge retches a steady position when indentations complete. Then take- back the lever (9) to 'A' position slowly (sudden return of lever (9) from 'B' to 'A' may show erratic readings). The weights (4) are thereby lifted off, only the initial load remaining active.
- 7. Read the figure against the long pointer. This is the direct reading of the Rockwell Hardness of specimen. Use black or red scale as per the selection of Rockwell scale. Black scale foe

"C" scale and red for 'B' scale.

- 8. Turn back the hand wheel (10) and remove the specimen piece. Carry on the same procedure for further tests.
- 9. The first hardness value so obtained may not be correct. All standards recommended neglecting first two readings to ensure that specimen. The Indentor and the anvil are seating correctly, further reading will be correct.

Standard Calibration.

- 1. Understand thoroughly the operation of each machine, and check its operation before proceeding.
- 2. Check the calibration of the Rockwell Machines with Standard Calibration Test Blocks for the scale selected.



- 3. Using the appropriate scale
 - (a) Check the hardness of each test specimen on a Rockwell Test Machine.
 - (b) Tabulate the results.
 - (c) Convert all readings to either R_B or Re values.
- 4. Using the hardness conversion chart, find the Tensile Strength of the steel samples.

7. **OBSERVATION:**

The following is a sample hardness data as presented in a laboratory report. Use the same format in your report.

Material	Rockwell Hardness Scale, Major Load, Type of Penetrator	Rockwell Hardness Number	Tensile Strength
Example 1 (steel)			
Example 2 (steel)			
Example 3 (steel)			

EXPERIMENT -7

JOMINY END QUENCH TEST

- 1. **AIM:** To determine the Hardenability of a given steel.
- 2. **Objectives:** a) To understand the concept of Hardeanability.
 - b) To study methods of determination of Hardeanability.

3. **APPARATUS:**

Jominy test apparatus, furnace, Rockwell hardness tester and a grinder.



JOMINEY END QUENCH TEST APPARATUS



ROCKWELL HARDNESS TESTER

4. THEORY :

Jominy end quench test is used to determine Hardenability of steels. The process of increasing the hardness of steel is known as Hardening. Specific specimen with standard dimensions, used for the test is given in fig. 8.1. The hardness of hardened bar is measured along its length.

4.1 Hardenability

The depth up to which steel can be hardened is defined as Hardenability. A steel having high hardness need not have high Hardenability. Hardenability may be defined as susceptibility to hardening by quenching. A material that has high Hardenability is said to he hardened more uniformly throughout the section than one that has lower Hardenability.

M.A. Gross man devised a method to decide Hardenability,

4.1.1 Critical diameter:

The size of the bar in which the zone of 50% martensite occurs at center is taken as critical diameter. This is a measure of Hardenability of steel for a particular quenching medium employed.

4.1.2. Severity of Quench:

The severity of quench is indicated by heat transfer equivalent

H = 17k

f- Heat transfer factor of quenching medium and the turbulance of the bath.

k = Thermal conductivity of bar material.

The most rapid cooling is possible with severity of quench as infinity.

4.1.3 Ideal Critical Diameter :

The Hardenability of steel can be expressed as the diameter of bar that will form a structure composed of 50% martensite at the center when quenched with H = infinity. This diameter is defined as ideal critical diameter.

5. Description of Apparatus:

Jominy end quench apparatus is shown in fig. 8.2.

The apparatus consists of a cylindrical drum. At the top of the drum provision is made for fixing the test specimen. A pipe line is connected for water flow, which can be controlled by means of a stop cock.

6. **PROCEDURE:**

- a. Out of the given steel bar. the standard sample is to be prepared as per the dimensions, shown in the fig. 8.1
- b. The austenitising temperature and time for the given steel is to be determined depending on its chemical composition.
- c. The furnace is setup on the required temperature and sample is kept in the furnace.
- d. The sample is to be kept in the furnace for a predetermined time (based on chemical composition of steel) then it is taken out of the furnace and is kept fixed in the test apparatus.
- e. The water flow is directed onto the bottom end of the sample. The water flow is adjusted such that it obtains shape of umbrella over bottom of sample.
- f. The quenching is to be continued for approximately 15 minutes.
- g. A flat near about 0.4 mm deep is grounded on the specimen.
- h. The hardness of the sample can be determined at various points starting from the quenched end and the results are tabulated,
- i. The graph is plotted with hardness values versus distance from quenched end. From the results and graph plotted the depth of hardening of the given steel sample can be determined.

The Hardenability of the specimen is found by observing the structure under the microscope. As detailed above the diameter at which the percentage of martensite is 50" indicates Hardenability of material. More this diameter, high will be the hardenability. Now the important factor is the relationship between size are diameter of a steel bar quenched in an ideal quenching medium which has the same cooing rate at it center as a given position along the surface of a Jominy bar. This information is furnished in fig.

8.4. Its importance is associated with the fact that if position on the Jommy bar where the structure is' 50% martensite is known then the curve shown in fig. 8.4 makes it possible to determine ideal critical diameter.

7. **OBSERVATION TABLE**

S.No	Distance from quenched end	Hardness

8. **RESULTS:**

9. CONCLUSIONS:

10 PRECAUTIONS:

- 1. The specimen is to be handled carefully while transferring from furnace to test apparatus.
- 2. Proper water flow (at high pressure) over the bottom end of specimen is to be ensured.

10. REVIEW QUESTIONS:

- i) What is the difference between Hardness & Hardenability?
- ii) What is severity of quench?
- iii) What is critical diameter?
- iv) What is the ideal critical diameter?
- v) What is the quenching medium employed in the test?
- vi) What are the important precautions to be observed in the test?
- vii) Why a flat is to be ground on the test specimen?
- viii) What is the equipment used to measure the hardness of specimen in the experiment?