



**Parala Maharaja Engineering College, Berhampur**

*A Government Engineering College affiliated to  
Biju Patnaik University of Technology, Odisha, Rourkela, India*

ପାରଳା ମହାରାଜା ଯାନ୍ତ୍ରିକ ମହାବିଦ୍ୟାଳୟ, ବ୍ରହ୍ମପୁର  
(ସରକାରୀ ଯାନ୍ତ୍ରିକ ମହାବିଦ୍ୟାଳୟ)

# **PHYSICAL METALLURGY LABORATORY MANUAL**

**COURSE: B. TECH  
(2<sup>nd</sup> YEAR-3<sup>rd</sup> SEMESTER)  
LOCATION: WORKSHOP-II**



**Department of Metallurgical and  
Materials Engineering**

## **SAFETY IN THE LAB**

- You are only allowed in the laboratory when there is a 'responsible person' present, such as a demonstrator or the laboratory staff.
- Do not touch any equipment or machines kept in the lab unless you are asked to do so.
- A tidy laboratory is generally safer than an untidy one, so make sure that you do not have a confused tangle of electrical cables. Electrical equipment is legally required to be regularly checked, which means it should be safe and reasonably reliable. Do not tamper with or attempt to repair any electrical equipment (in particular, do not rewire mains plug or change a fuse - ask one of the laboratory staff to do it). Never switch off the mains using the master switches mounted on the walls. Please familiarize yourself with the fire exits upon entering the lab. When the alarm sounds, please leave whatever you are doing and make your way quickly, calmly, and quietly out of the lab. You must always follow instructions from your demonstrators and the laboratory staff.
- You must keep walkways clear at all times, and in particular, coats and bags must be stowed away safely and must not pose a trip hazard.
- It is important that you make a point of reading the "Risk Assessment" sheet included in the manuscript of each experiment before you start work on the experiment.
- Please take notice of any safety information given in your scripts. If an experiment or project requires you to wear PPE (personal protective equipment) such as gloves and safety glasses, then wear them.
- Always enter the lab wearing your shoes. It is strictly prohibited to enter the lab without shoes.
- There must be NO smoking, eating, drinking, use of mobile phones or using personal headphones in the laboratory. This last point is not because we dislike your choice of music but because you must remain aware of all activity around you and be able to hear people trying to warn you of problems.
- Keep the lab neat and clean.

## **List of experiments:**

1. Study of the metallurgical microscope.
2. Metallographic preparation of metals and alloys.
3. Microscopic examination of steels and non-ferrous metals, interpretation of microstructures, and microstructure of heat-treated steels.
4. Microscopic examination of cast, wrought, and welded structures; defects and failures in components.
5. Comparison of microstructures of cold-worked and annealed samples.

# EXPERIMENT -1

**Aim:** To study the complete working operation of the Metallurgical Microscope

**Objectives: -**

1. Familiarization with the different components of metallurgical components.
2. Familiarization with the compound optical microscope and metallographic.
3. To study the magnification system and how to increase magnification.

**Introduction: -**

The metallurgical microscope is the most important tool of the metallurgist. It consists of an objective and an eyepiece. Its primary function is to reveal the details of the object. The clarity and the extent to which the details are shown depend on the degree to which these optical systems are created.

**Principle: -** A horizontal beam of light from the light source is reflected using a plane glass reflector downwards through the microscope objective on the surface of the specimen. Some of the incident light reflected from the specimen surface will be magnified and pass through the plane glass reflector and magnified again by the upper lens system of the eye-piece.

**Constructional Details: -** The table-type microscopes consist of

1. Stage: A flat movable table supporting the specimen. This can be moved up or down with knobs.
2. Tubes: - The vertically movable tube containing the eyepiece, objective, and plane reflector. The tube length varies from 160 mm to 250 mm.
3. Rough & fine focus Adjustments - The limbs of the microscope carry the coarse & fine adjustments.
4. Objective – The body tube carries a revolving nose piece carrying the three objectives. This enables a quick change of the objective, which helps for a quick resolution of the structure of metal, and the magnification of lenses is enlarged on the focal length of the lens used

The important properties of an objective are-

- 1) Magnifying Power
- 2) Resolving Power.

It is the property by which an objective shows distinctly represented two small adjacent bonds in the structure of the object. This is usually expressed as several lines per mm that can be separated, which depends on the numerical operator and the wavelength of the light used. Resolution is crucial during the microscopy of the micro constituents of metals, consisting of fine lamination with core resolution, which appears as one uniform area. In contrast, an objective with a higher numerical appearance reveals a deeper nature of the structure.

The total magnification of the microscope may be calculated

as  $M = L \cdot E / F$

Where L- is the distance from the back of the objective to the eyepiece. E- Magnification of Eyepiece. & F- The focal length of the objective.

Eyepiece - It is named, as it is near the eye. It is made up of various Powers such as 5, 10, 15, etc.

**Uses:** -The metallurgical microscope is useful in quality control departments in Industries to observe & study

- 1) Differential phases
- 2) Porosity or defects.

All these have a great effect on the mechanical properties of a material. List of Modern Microscopes –

- i) Watson Royal Microscope.
- ii) Van Lanes Hock Microscope.
- iii) Glass-LED Microscope.
- iv) Baker series Microscope.
- v) Leitz Microscope.

## Conclusion: -

Proper selection of magnification is very much essential because –

- a. To travel, inclusions in steel, low magnification is used.
- b. To travel grain size, grain structure, twin boundaries, etc., medium magnification is used.
- c. To travel particular e.g., coarse perlite, fine martensite, bainite, etc., high magnification is necessary.

Therefore, a proper study of metallurgical microscopes is necessary.



Fig- Metallurgical Microscope

## **EXPERIMENT-2**

**Aim-** Preparation of metallic specimens for metallographic study by grinding, polishing, and etching.

### **Objectives-**

1. Familiarization with the procedure for preparation of material specimens for microscopic examination.
2. Familiarization with the compound optical microscopic and metallographic.
3. Examination of surface characteristics of engineering materials.
4. Grain size determination of metals.
5. To study the structural characteristics or constitution of metal or alloy in relation to its physical and mechanical properties.

### **Introduction-**

There are two examination methods-

1. Macroscopy
2. Microscopy

In macroscopy, the examination of the structural or chemical characteristics of metal or an alloy is done by the unaided eye or with the aid of a low-power microscope or binocular, usually under 10X.

In microscopy, similar examination is done with prepared metal specimens, employing magnifications with an optical microscope of from 100X to as high as 200X. Apart from the observation of microstructural details in a metal or alloy, other defects such as grain boundaries, twins, and precipitates can be observed readily in microscopic examination.

The preparation of metallurgical specimens generally can be divided into a series of stages: sectioning/sampling, mounting, grinding, polishing & etching.

## **Procedure of Test**

### **1] Sectioning / Sampling-**

The choice of sample for microscopic study may be very important. If a failure is to be investigated, the sampling should be chosen as close as possible to the area of the failure & should be compared with one taken from the normal section. If the material is soft, such as nonferrous metals or alloy & non-heat-treated steels, the section is obtained by manual hack sawing /power sawing. If the material is hard, the section may be obtained by the use of abrasive cutoff wheels. This wheel is a thin disk of suitable cutting abrasive rotating at high speed. The specimen should be kept cool during the cutting operation.

### **2] Rough Grinding –**

Whenever possible, the specimen should be of a size & shape that is convenient to handle. A soft sample may be made flat by slowly moving it up to & back across the surface of a flat, smooth file. The soft-hard material may be rough ground on a belt sander with the specimen kept cool by frequent dipping in water during the grinding operation. In all grinding & polishing operations, the specimen should be moved perpendicular to the existing scratches. This will facilitate recognition of the stage when the deeper scratches are replaced by shallower one's characteristic of the finer abrasives. The rough grinding is continued until the surface is flat & free from wire brushes & all scratches due to the hacksaw or cut-off wheel are no longer visible.

### **3] Intermediate Polishing –**

After the previous processes, the specimen is polished on a series of emery paper containing successively finer abrasives (Si-C). The first paper is usually no. 1, then 1/0, 2/0, 3/0, & finally 4/0. The intermediate polishing operation using emery paper is usually done dry. However, in certain cases, such as the preparation of soft material, Silicon Carbide has a greater removal rate & as it is resin bonded, can be used with a lubricant, which prevents overshooting of the sample, minimizes shearing of soft metals & also provides a rising action to flush away surface removal product so the paper won't be clogged.

#### **4] Fine polishing –**

The time consumed & the success of fine polishing depend largely on the case that we exercised during the previous polishing processes. The final approximation to the flat, scratch-free surface is obtained by the use of a wet rotating wheel covered with a special cloth that is charged with carefully sized abrasive particles. A wide range of abrasives is available for final polishing, while many will do a satisfying job, these appear to be a presence of gamma form of aluminum-oxides ( $\text{Al}_2\text{O}_3$ ), for ferrous & copper-based materials, & Cerium oxide for Aluminium, Magnesium & their alloys, other final polishing abrasives often used are diamond, chromium oxide & magnesium oxide, etc. A choice of proper polishing cloth depends upon the particular material being polished & the purpose of the metallographic study. Many cloths are available in varying lap or pile, from those having no pile, such as silk, to those of intermediate pile, such as broadcloth, billiard cloth, canvas cloth & finally to a deep pile such as velvet synthetic clothes are also available for the general purpose of which two under the trivial names of Gamal & micro cloth are most widely used.

#### **5] Etching –**

The purpose of etching is to make the many structural characteristics of the metal or alloy visible. The process should be such that the various parts of the microstructure may be differentiated. This is to subject the polished surface to chemical action. The alloys are composed of two or more phases. The composition is revealed during etching by a preferential attack of one or more of the constituents by the reagent because of the difference in the chemical composition of the phases. In a uniform single-phase alloy, contact is obtained, and the grain boundaries are made visible because of the difference in the rate at which various grains are attacked by the reagent. This difference in the rate of attack by the reagent is mainly associated with the angle of the different grain structure section to the plane of the polished surface. Because of the chemical attack of the chemical reagent, the grain boundary appears as valleys in the polished surface. Light from the microscope hitting the side of these valleys will be reflected, but the microscope makes the grain boundaries appear as dark lines. The selection of the appropriate etching reagent is determined by the metal or alloys & the specific structure desired for viewing.

<b>Etching Reagent</b>	<b>Composition</b>	<b>Use</b>
Nitric Acid	White nitric acid, 1 to 5 ml ethyl or methyl alcohol' 100 ml (95%)	Carbon steels and cast iron
Picric acid	Picric acid '49 ethyl or methyl alcohol.'	Carbon steels
Ferric chloride and hydrochloric	Ferric chloride and hydrochloric acid 50 ml water 100 ml	Structure of austenitic nickel stainless steel
Ammonium hydroxide and hydrogen	Ammonium hydroxide 90 parts water, 5 parts hydrogen peroxide, 2 to 5	Copper and its alloys
Palmerton reagent	Chromic oxide 200 gm sodium sulphate 15 gm water	Zinc and its alloys
Ammonium molybdenum	Molybdic acid 100 gm, ammonium hydroxide 140ml, water 240 ml, filters, add to nitric acid S.P.gr. 60 ml	Lead and its alloys
Hydrofluoric acid	Hydrofluoric acid 0.5 ml	Aluminum and its alloy

### **Result and Conclusion: -**

Correct sample techniques, if followed properly, will only reveal the microstructure of a metallic specimen. Otherwise, it will be most difficult to distinguish between different phases in the microstructure, the fine scratches on the meta surface, and grain boundaries, etc.

## EXPERIMENT-3

**Aim:** Preparation and study of the Micro Structure of pure metals, Mild Steel, Low Carbon steel, and High Carbon Steel.

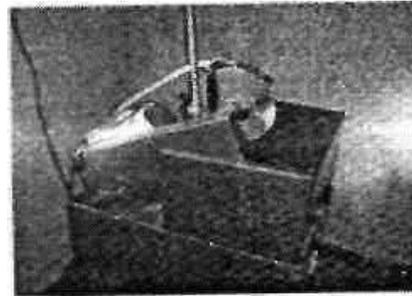
### Objectives:

- a) To Learn the preparation of specimens for microscopic observation.
- b) To understand what Microscopy is, and how it can be used to observe the Microstructure of metals.

### APPARATUS / EQUIPMENT

#### *Abrasive Cat-Off Wheel Machine*

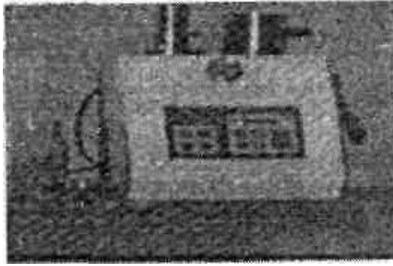
- 3 HP Motor, 1440 rpm
- Manual model
- 10” Diameter-wheel
- Fully enclosed
- Cutting Capacity 50mm Diameter & 75mm
- Height X 200mm Length.



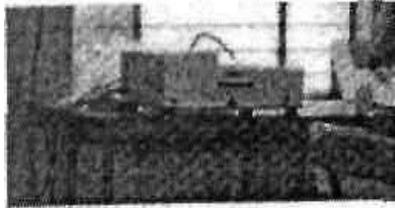
This Equipment is used to cut the sample pieces from the components of all kinds of materials. These sample pieces can be used for microscopic observation.

#### *Specimen Mounting Press*

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

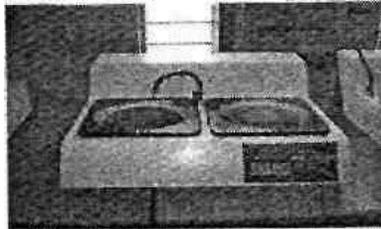


This Equipment is used to mount specimens in Bakelite. Particularly is particularly useful in the hot mounting process.



### **Belt Finishing Machine.**

This Equipment is used for rough Grinding of the Specimen 3.4 Polishing 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 clothes. It is also useful for rough grinding with grinding discs.

### **Metallurgical Microscope.**



Binocular model

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

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

## CONSUMABLES/ RAW MATERIAL -:

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

## THEORY -:

Mild Steel, Low Carbon steel, and High Carbon Steel are types of ferrous materials and are most important to engineering applications because of their wide range of properties and variety of applications. Theoretically, steels are the alloys of iron and carbon in which the carbon content is between 0.008 to 2 %. The structures and properties can be discussed with the help of the Fe-C equilibrium diagram.

## Iron -Iron Carbide Equilibrium Diagram

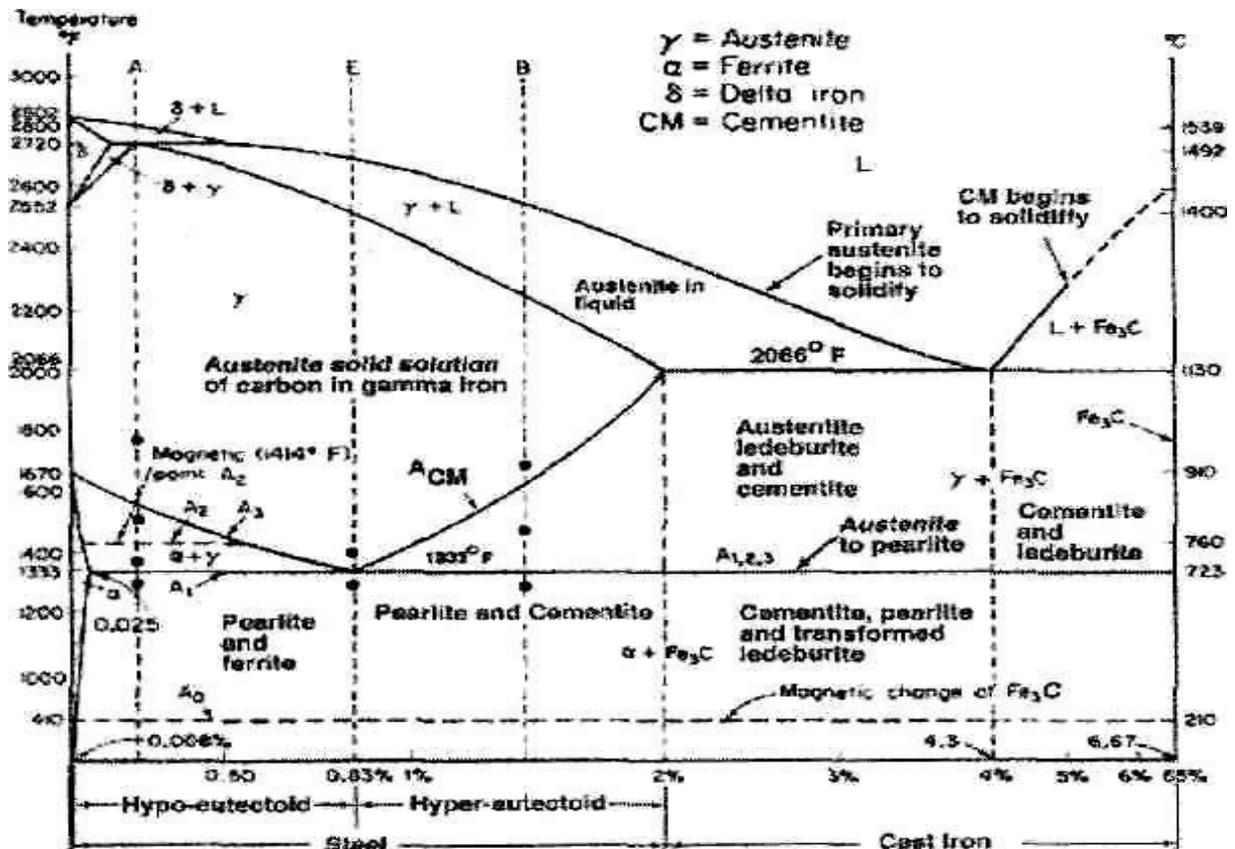


Fig. Fe-Fe<sub>3</sub>C Phase Diagram

The figure shows the equilibrium diagram for combinations of carbon in a solid solution of iron. The diagram shows iron and carbon combined to form Fe-Fe<sub>3</sub>C 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 hypereutectoid 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 4.3% C. At 0.83%C, the transformation is eutectoid, called pearlite.



At 4.3% C and 2066°F, the transformation is eutectic, called ledeburite.



## Definitions

**Eutectoid:** A eutectoid system occurs when a single-phase solid transforms directly into a two-phase solid.

**Hypereutectoid:** Hypereutectoid systems exist below the eutectoid temperature.

**Hypoeutectoid:** 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 that results when austenite is no longer the most stable form of iron. Exists between 2802°F and 2552°F. has a BCC lattice structure and is magnetic.

**Body-centered:** A structure in which every atom is surrounded by eight adjacent atoms, whether the atom is located at a corner or the center of a unit cell.

**Face-centered:** 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 is formed when carbon is more than the solubility limit.

**Ledeburite:** Eutectic of cast iron. It exists when the carbon content is greater than 2 %. It contains 4.3 % carbon in combination with iron.

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

- 1) 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)
- 2) Number of alloying elements and carbon
- 3) Amount of deoxidation
- 4) Method of manufacture
- 5) Form and use.

**PROCEDURE:** The preparation of metallic or other materials for microscopic examination and microstructural characterization is, in principle, 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.

### **Sample Cutting and Sectioning**

#### **Sectioning:**

Sectioning means the removal of a convenient-sized specimen from a large sample with minimal damage to microstructure with the help of an abrasive cut-off machine. Abrasive cutting wheel/saw is attached to the cutting machine, and for work piece holding proper vice is provided on the 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 are equipped with a water-cooling system.

## Mounting

If a sample is large enough (about 25 mm square or larger) then 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 commonly used thermosetting plastic compound (Bakelite) to encapsulate the specimen, known as the hot-mounting process, and the second uses a room-temperature curing epoxy known as the 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 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.

### The procedure for the Hot mounting process

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

- 1) Place the sample face down on the small piston inside the press, and lower the piston into the cylinder by opening the valve on the front of the press.
- 2) Approximately three tablespoons of Bakelite are poured over the sample, and the top of the press is 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. Clean any residual Bakelite off of the mold surfaces.

### **The procedure for the Cold Mounting process.**

The following process is used for the room temperature curing of the epoxy:

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

### **Sample Surface Polishing**

The goal of the surface polishing is to end up with a planar cross-section of the 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.

#### **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 the 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.

## Rough Grinding

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

## 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 it into the trash bin.
- 4) Examine the 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 the last polishing stage, the sample looks mirror-like.

## Etching

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

Steps:

- 1) Apply the etchant to the polished surface for some time.
- 2) Rinse with distilled water.

## Etchants:

- 1) **50/50 HCL**: equal parts hydrochloric acid (HCL) mixed with water.
- 2) **Alcoholic Ferric Chloride**: 5 grams  $FeCl_3$ ; 2 ml concentrated HCl acid; 95 ml methyl alcohol.
- 3) **Aqueous Ferric Chloride**: 10 grams  $FeCl_3$ ; 20 ml concentrated HCl 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 HCl acid; 2.5 ml concentrated nitric ( $HN0_3$ ) acid; 0.5 ml hydrofluoric (HF) acid. 2% Nital: 2 ml concentrated  $HN0_3$ ; 98 ml methyl alcohol.

## Metallographic Observation

Observe microstructure, place the specimen on the metallography, and adjust magnification, focus, and positions. Adjust micro-High magnification - to study phases, and Low magnification -to study grain size.

## Microphotography

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 on 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 is 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, a 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 the magnification used (i.e., "MAG= 100 X"). Important phases and features noted
- 4) Etchant used.

## EXPERIMENT-4

**Aim:** Study of the Microstructures of Cast Iron.

### Objectives:

- To understand what Microscopy is, and how it can be used to observe the Microstructure of metals like cast iron.
- To conduct typical engineering microscopic observation and be able to recognize the microstructure of a typical type of cast iron metal.
- To learn the differences in the microstructure of different types of cast irons.

### APPERATUS:

Metallurgical Microscope, Standard Specimens of Cast Iron Materials.

#### Metallurgical Microscope.

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



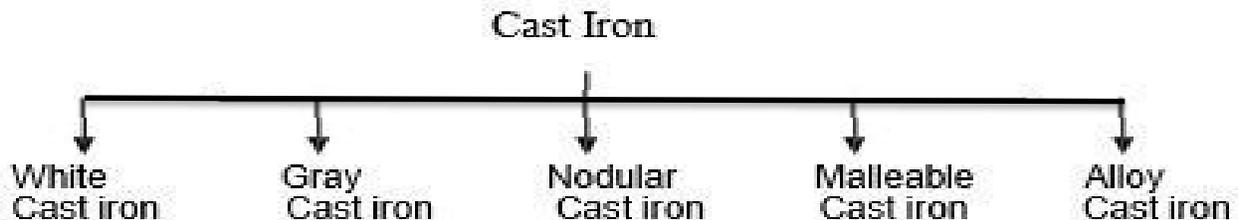
## **THEORY:**

### **Cast Iron**

Cast irons typically contain 2-4 wt.% of carbon with a high silicon concentration and a greater concentration of impurities than steels. The carbon equivalent (CE) of 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 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 a higher carbon and silicon content 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-Fe<sub>3</sub>C system or the stable Fe-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-Fe<sub>3</sub>C 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.

### **Types of Cast Iron**

Based on Microstructure cast irons are classified as follows.



## White Cast iron:

If the composition of the iron is appropriate or the cooling rate of the metal is sufficiently rapid during solidification, the metal will solidify with carbon 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. The application includes rollers of rolling mills, dies of metal extrusion, and where high wear resistance is necessary.

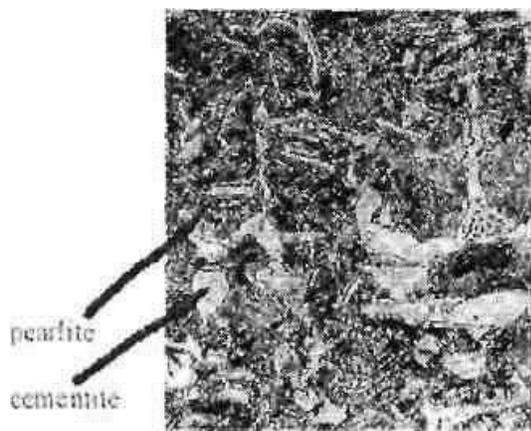


Fig. Microstructure of white cast iron

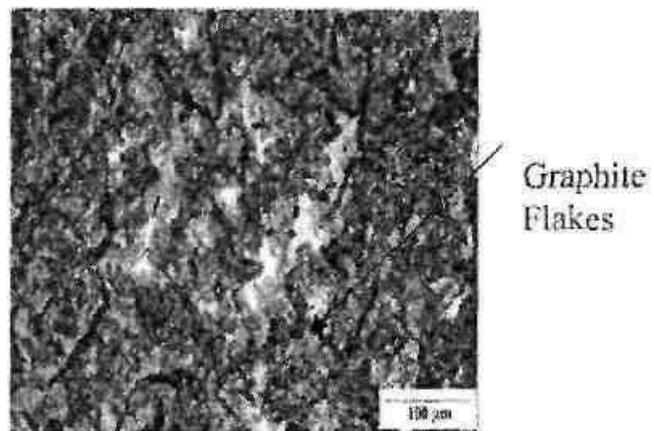


Fig. Microstructure of Gray cast iron

## 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 its 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.

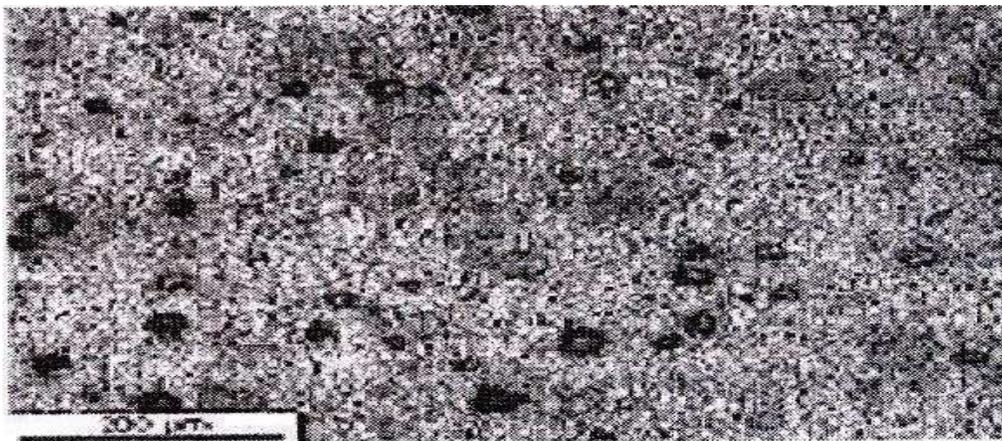
### **Nodular cast Iron (Ductile iron, S. G Iron)**

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 provides 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 as-cast 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 a response to heat treatment. Special analysis of ductile irons and high-alloy ductile irons provides unusual properties for special applications.

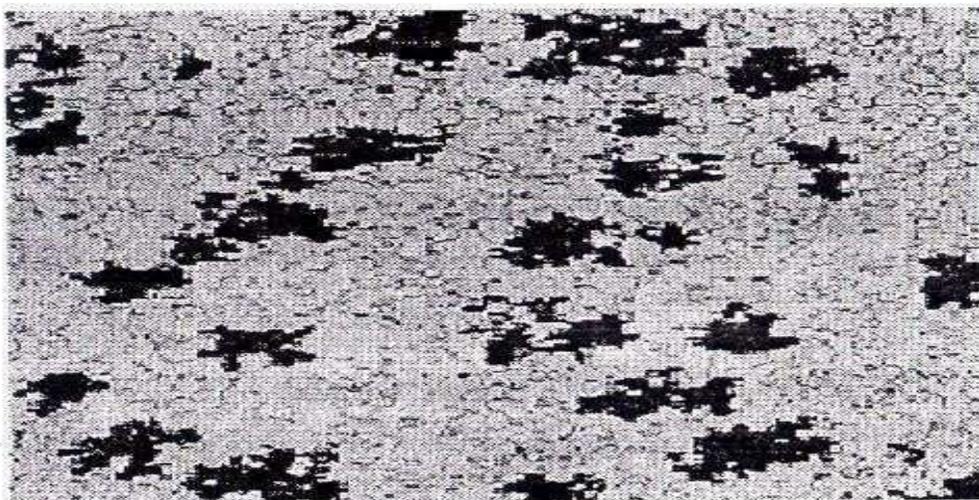
### **Malleable Cast Iron**

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 which 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**

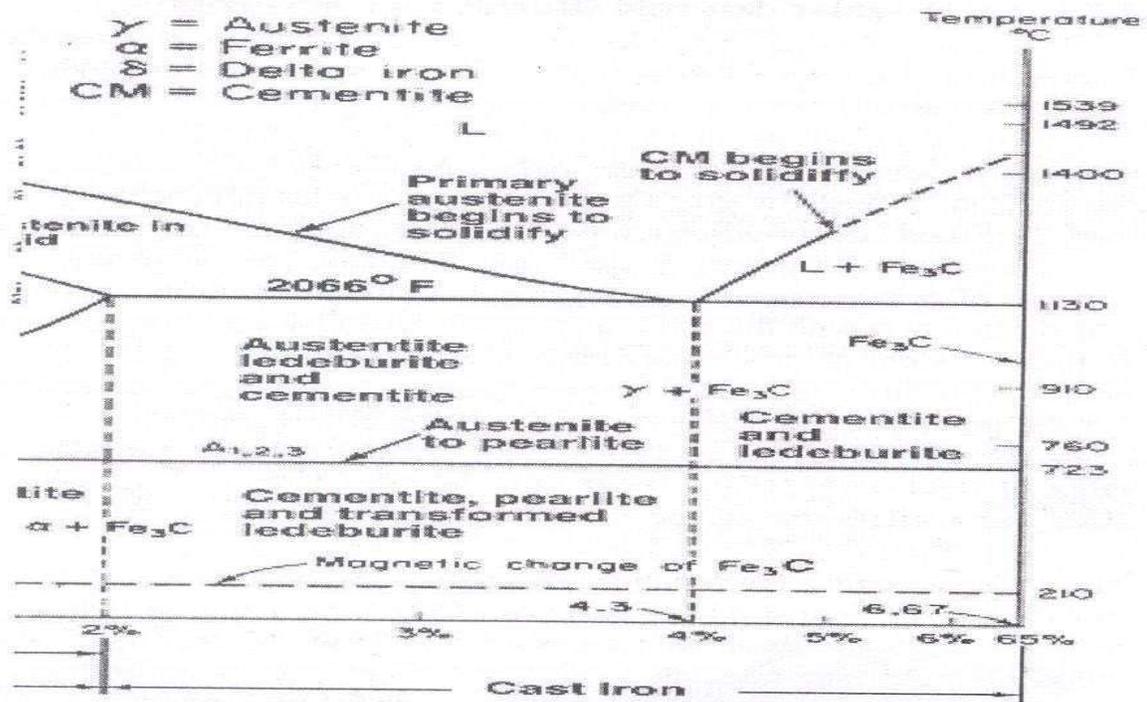


Fig. 6 Iron-Carbon diagram (cast iron portion)

## 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 on 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 is 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.

## EXPERIMENT-5

**AIM:** Study of the Microstructures of Non-Ferrous Metals.

### OBJECTIVES:

- To understand what Microscopy is, and how it can be used to observe the microstructure of non-ferrous metals.
- To conduct typical engineering microscopic observation and be able to recognize the microstructure of typical types of copper, aluminum, and its alloys.
- To learn the differences in the microstructure of different types of cast irons.

### APPARATUS:

Metallurgical Microscope, Specimens of Non-Ferrous Metals.

#### Metallurgical Microscope.



#### Binocular model

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

## **THEORY:**

### **COPPER AND COPPER ALLOYS**

**COPPER** -: Copper is a face-centered cubic (FCC metal) with very high ductility, and electrical and thermal conductivity. This is very soft too. It is used in electrical applications, industrial machinery, and hearth for arc furnaces. In the alloyed form, it also finds enormous importance. One such application is duralumin (Al-4.5% Cu), which is a common material for airplane bodies. When it is alloyed with Zn, it is called brass. 70:30 Brass is very common in making pipelines for water.

Copper properties: -

- Its melting point is 1083°C.
- 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 ARSONICAL Cu.
- Cu containing 0.6% Al is called FREE-CUTTING Cu.

### **COPPER ALLOYS:**

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

**BRASS: - Cu + Zn = Brass.**

Brass is again divided into *alpha brass* and *alpha + beta brass*.

#### **TYPES OF ALPHA BRASS**

### **CAP BRASS: -**

- Consists of 2-5% Zn.
- Very ductile alloy.
- Zn is used as a deoxidizer for the deoxidation of Cu.
- Used for the cap of detonation.

2) **GUILDING METAL:** -

- Zn-(5-15%).
- Has the colour of gold.
- Uses artificial jewelry, 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) **ADMIRALTY 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 resistant compared to alpha brasses.

1) **MUNTZ METAL:** -

- Contains 40%Zn.
- Alloy becomes single phase at about 700 °C
- Also called (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 the marine environment
- Also called TOBIN BRONZE.

3) **LEADED BRASS:**

- Lead was added in a 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 IMITATION GOLD.
- Applications “-components of valves, pump castings, park plug body.

#### **2) TIN BRONZE: -**

- ALLOY OF Cu and Sn.
- Tin has an affinity towards oxygen. the tin oxide formed reduces ductility and malleability. Zn or P is added to deoxidize these.

### **COINAGE BRONZE**

Contains 5% Sn and 1% Zn.

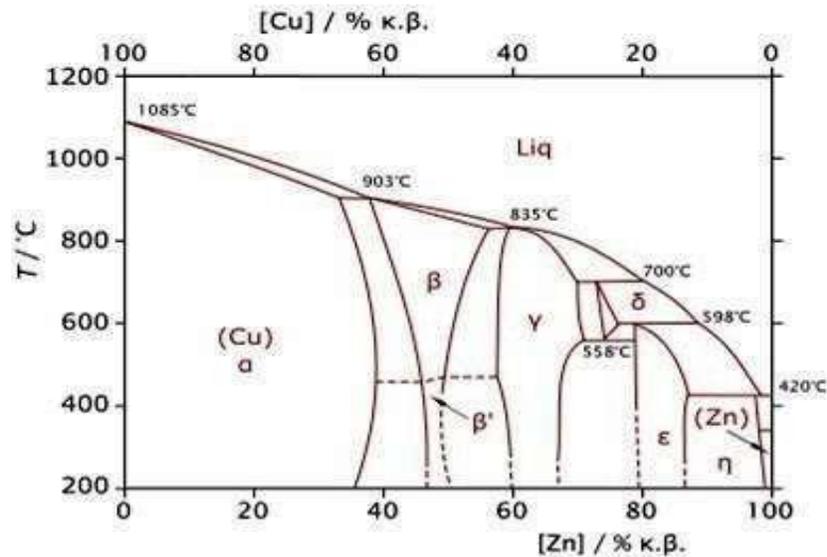
- It is a ductile metal, having better formability and strength.
- Application: manufacturing of coins.

#### **1) PHOSPHOROUS BRONZE: -**

- Alloy of Cu and Zn with P.
- Phosphorus is a strong deoxidizer and helps in increasing fluidity.
- Phosphorous content is max up to 1% depending on the 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 in each other in both solid in liquid solutions.
- Application—blades of turbine, coins, bullet envelopes.



Phase diagram of the Cu-Zn system

### ALUMINIUM Properties:

- Light in weight.
- Cast ability and formability are better.
- Corrosion resistance of Al is excellent.
- Powerful deoxidizer.
- Carries more electricity than Cu.
- Costlier metal in the family of light metal alloys.
- Has good electrical and thermal conductivity.

### Al ALLOYS

#### 1) Al-Si-Cu: -

- LM2, LM6, LM8, LM13.

- Used for the production of castings, due to their excellent fluidity and casting characteristics.
- Higher Si content improves the mechanical properties.

### 2) **Al-Mg (MAGNALIUM ALLOY): -**

- LM5NDLM10.
- Used in marine environments. Due to its high strength and resistance to corrosion.
- Has a good surface finish.

### 3) **Al-Cu (DURALIUM): -**

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

### 4) **Y ALLOY (HIGH STRENGTH Al 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, and Zr.
- Used in aero engines and other continuous elevated temp service applications up to 300°C.

## **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 on 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 is 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.

## EXPERIMENT-6

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

### OBJECTIVE:

- a. To observe the microstructure of heat-treated steels.
- b. To study the changes in macrostructure due to heat Treatment.
- c. To study one of the heat treatment processes.

### APPARATUS:

- I) Specimens of High Carbon Steel subjected
- II) Metallurgical Microscope.



Binocular model

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

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

## **THEORY:**

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 the material, but it can also be used to alter certain manufacturability objectives, such as improving machining, improving formability, and restoring ductility after a cold working operation. Thus, it is a very enabling manufacturing process that can not only help another 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: Softening, Hardening, or material modification

### **Heat Treatment Processes:**

#### **- *Annealing***

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

Purpose of Annealing:

- To relieve the internal stresses induced due to cold working and welding.
- To reduce hardness and to increase ductility.
- To refine the grain size.
- To make the material homogeneous in terms of chemical composition.
- To increase machinability
- To increase the uniformity of phase distribution and to make the material isotropic with respect to mechanical properties.

### *Types of Annealing -:*

- a) Full Annealing
- b) Spheroidized Annealing
- c) Bright Annealing
- d) Box annealing
- e) Isothermal Annealing

### - ***Normalizing***

The process consists of heating the steels above the upper critical temperature ( $A_3$ ) for hypoeutectoid steels, and above  $A_{cm}$  for hypereutectoid 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 in annealing, normalized components show slightly different structure and properties than annealed components. After normalizing, the microstructure shows more pearlite than observed in annealed components.

#### ***Purpose -:***

- This process is used to eliminate the cementite network that formed due to slow cooling in the temperature range from  $A_{cm}$  to  $a_1$ .
- To relieve internal stresses.
- To make the material homogeneous in chemical composition.
- To increase machinability.
- To increase the uniformity of phase distribution and to make the material isotropic with respect to mechanical properties.

- **Hardening:**

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

Draw a hardening cycle.

- **Tempering:**

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

***Time — Temperature — Transformation Diagram.***

***PROCEDURE:***

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  - b) Important phases and features noted,
- Etchant used.