

# PHYSICAL METALLURGY LABORATORY

## OBSERVATION BOOK



Name of the Student: .....

Scholar No: .....



**Department of Metallurgical & Materials Engineering**

**VEER SURENDRA SAI UNIVERSITY OF TECHNOLOGY, BURLA**

**Odisha – 768018**

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Name of the Student: .....

Scholar No: .....

LABORATORY COORDINATOR(S)

**A. LAVA KUMAR**

**Assistant Professor**



**Department of Metallurgical & Materials Engineering**

**VEER SURENDRA SAI UNIVERSITY OF TECHNOLOGY, BURLA**

**Odisha - 768018**

## **PREFACE**

This lab manual is designed to suit the needs of the under graduate metallurgist in acquainting with the principles and practices of metallography. The metallographic techniques are stressed, as these practices are essential for a metallurgist. In this observation book an attempt is made to clarify the concepts of crystal structures, specimen preparation techniques, etching techniques. The students shall get clarity about microstructures of carbon steels, cast irons and non-ferrous alloys, in particular to differentiate between various phases present in metals and alloys. The student shall also be trained in grain size measurements, and quantitative metallography.

Instructor will indicate the experiments to be conducted and advise on the nature of the lab report and time of submission. However, a typical format for a lab report is included in this observation book. It is important that all the information necessary to complete the lab report is obtained before students leave the lab.

**Mr.A.Lava Kumar**  
Assistant Professor

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## SAFETY IN THE LABORATORY

All students must read and understand the information in this document with regard to laboratory safety and emergency procedures prior to the first laboratory session. *Your personal laboratory safety depends mostly on you.* Effort has been made to address situations that may pose a hazard in the lab but the information and instructions provided cannot be considered all-inclusive. Students must adhere to written and verbal safety instructions throughout the academic term. Since additional instructions may be given at the beginning of laboratory sessions, it is important that all students arrive at each session on time. With good judgment, the chance of an accident in this course is very small. Nevertheless, research and teaching workplaces (labs, shops, etc.) are full of potential hazards that can cause serious injury and or damage to the equipment. Working alone and unsupervised in laboratories is forbidden if you are working with hazardous substances or equipment. With prior approval, at least two people should be present so that one can shut down equipment and call for help in the event of an emergency. Safety training and/or information should be provided by a faculty member, teaching assistant, lab safety contact, or staff member at the beginning of a new assignment or when a new hazard is introduced into the workplace.

### *Emergency Response*

1. It is your responsibility to read safety and fire alarm posters and follow the instructions during an emergency
2. Know the location of the fire extinguisher, eye wash, and safety shower in your lab and know how to use them.
3. Notify your instructor immediately after any injury, fire or explosion, or spill.
4. Know the building evacuation procedures.

### *Common Sense*

Good common sense is needed for safety in a laboratory. It is expected that each student will work in a responsible manner and exercise good judgment and common sense. If at any time you are not sure how to handle a particular situation, ask your Teaching Assistant or Instructor for advice. **DO NOT TOUCH ANYTHING WITH WHICH YOU ARE NOT COMPLETELY FAMILIAR!!!** It is always better to ask questions than to risk harm to yourself or damage to the equipment.

### *Personal and General laboratory safety*

1. Never eat, drink, or smoke while working in the laboratory. Read labels carefully.
2. Do not use any equipment unless you are trained and approved as a user by your supervisor.
3. Wear safety glasses or face shields when working with hazardous materials and/or Equipment. Wear gloves when using any hazardous or toxic agent.
4. Clothing: When handling dangerous substances, wear gloves, laboratory coats, and safety shield or glasses. Shorts and sandals should not be worn in the lab at any time. Shoes are required when working with the heat treatment furnaces.
5. If you have long hair or loose clothes, make sure it is tied back or confined.

6. Keep the work area clear of all materials except those needed for your work. Extra books, purses, etc. should be kept away from equipment, which requires air flow or ventilation to prevent overheating.
7. Disposal - Students are responsible for the proper disposal of used material if any in appropriate containers.
8. Equipment Failure - If a piece of equipment fails while being used, report it immediately to your lab assistant or tutor. Never try to fix the problem yourself because you could harm yourself and others.
9. If leaving a lab unattended, turn off all ignition sources and lock the doors. Clean up your work area before leaving. Wash hands before leaving the lab and before eating.

### ***Electrical safety***

1. Obtain permission before operating any high voltage equipment.
2. Maintain an unobstructed access to all electrical panels.
3. Wiring or other electrical modifications must be referred to the Electronics Shop or the Building coordinator.
4. Avoid using extension cords whenever possible. If you must use one, obtain a heavy-duty one that is electrically grounded, with its own fuse, and install it safely. Extension cords should not go under doors, across aisles, be hung from the ceiling, or plugged into other extension cords.
5. Never, ever modify, attach or otherwise change any high voltage equipment.
6. Always make sure all capacitors are discharged (using a grounded cable with an insulating handle) before touching high voltage leads or the "inside" of any equipment even after it has been turned off. Capacitors can hold charge for many hours after the equipment has been turned off.
7. When you are adjusting any high voltage equipment or a laser which is powered with a high voltage supply, USE ONLY ONE HAND. Your other hand is best placed in a pocket or behind your back. This procedure eliminates the possibility of an accident where high voltage current flows up one arm, through your chest, and down the other arm.

### ***Mechanical safety***

1. When using compressed air, use only approved nozzles and never directs the air towards any person.
2. Guards on machinery must be in place during operation.
3. Exercise care when working with or near hydraulically- or pneumatically driven equipment. Sudden or unexpected motion can inflict serious injury.

### ***Chemical safety***

1. Treat every chemical as if it were hazardous.
2. Make sure all chemicals are clearly and currently labelled with the substance name, concentration, date, and name of the individual responsible.
3. Never return chemicals to reagent bottles. (Try for the correct amount and share any excess.)

4. Comply with fire regulations concerning storage quantities, types of approved containers and cabinets, proper labelling, etc. If uncertain about regulations, contact the building coordinator.
5. Use volatile and flammable compounds only in a fume hood. Procedures that produce aerosols should be performed in a hood to prevent inhalation of hazardous material.
4. Never allow a solvent to come in contact with your skin. Always use gloves. Never "smell" a solvent!! Read the label on the solvent bottle to identify its contents.
5. Dispose of waste and broken glassware in proper containers. Clean up spills immediately.
6. Do not store food in laboratories.

### ***Additional Safety Guidelines***

1. Never do unauthorized experiments.
2. Keep your lab space clean and organized.
3. Do not leave an on-going experiment unattended.
4. Never taste anything. Never pipette by mouth; use a bulb.
5. Never use open flames in laboratory unless instructed by TA.
6. Check your glassware for cracks and chips each time you use it. Cracks could cause the glassware to fail during use and cause serious injury to you or lab mates.
7. Maintain unobstructed access to all exits, fire extinguishers, electrical panels, emergency showers, and eye washes.
8. Do not use corridors for storage or work areas. Do not store heavy items above table height. Any overhead storage of supplies on top of cabinets should be limited to lightweight items only.
9. Also, remember that a 36" diameter area around all fire sprinkler heads must be kept clear at all times.
10. Be careful when lifting heavy objects. Only shop staff may operate forklifts or cranes.
11. Clean your lab bench and equipment, and lock the door before you leave the laboratory.

**I \_\_\_\_\_ Scholar No \_\_\_\_\_ have read and understood the laboratory safety procedures and policies. I am responsible for following these procedures while in the laboratories.**

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**(Signature with date)**

# PHYSICAL METALLURGY LAB

1<sup>st</sup> semester - 2017

## LOGBOOK MARK SHEET

Name:

Section:

Scholar Number:

Group Number:

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S.No	Experiment Title	Marks obtained	Instructor Sign.
1.	Study of Crystal Structures and Metallurgical Microscope	...../10	
2.	Specimen Preparation techniques for Metallographic Analysis	...../10	
3.	Fe-Fe <sub>3</sub> C Phase Diagram	...../10	
4.	Microstructural Analysis of Low carbon and Medium carbon steels	...../10	
5.	Microstructural Analysis of Cast Iron	...../10	
6.	Microstructural Analysis of Al-alloys	...../10	
7.	Standard test Methods for Estimation of Grain Size	...../10	
	<b>Total Marks obtained</b>		

**Unauthorized absences will be credited with a mark of 0/10.** Your completed laboratory logbook (with this sheet firmly stuck in and fully completed) must be submitted to your instructor at the end of each experiment. Your instructor will make an assessment and transmit the mark.

**A. LAVA KUMAR**  
Laboratory Coordinator



## EXPERIMENT 1

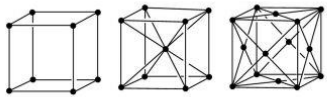

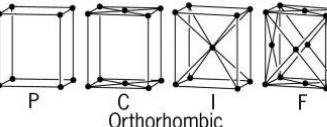
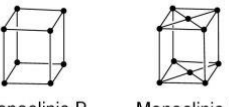



### STUDY OF CRYSTAL STRUCTURES & METALLURGICAL MICROSCOPE

**Objective:** To study the different crystal structures by using ball, ball and stick or wire models.

**Theory:** Crystal structure is one of the most important aspects of materials science and engineering as many properties of materials depend on their crystal structures. The solid are either crystalline or non-crystalline. The majority of engineering materials, such as ceramics, and most minerals, some plastics and all metals are crystalline in structure.

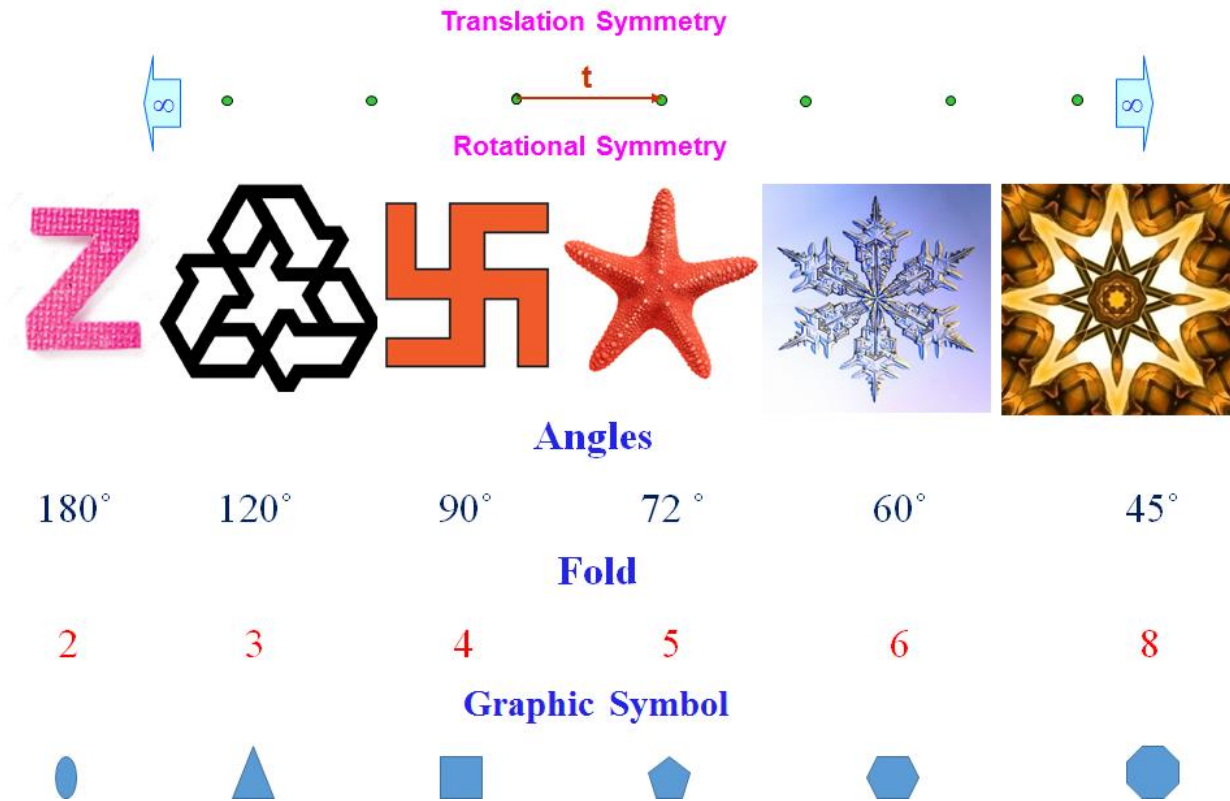
**Crystal structure:** A regular and repetitious pattern in which atom or group of atoms of crystalline materials are well arrange themselves is known as crystal structure. All crystalline solids may be classified into seven crystal systems or 14 crystal structures based on the symmetry aspects.

**Table 1: The 14 Bravais lattices**

Bravais lattice cells	Axes and interaxial angles	Examples
 <p style="text-align: center;">Cubic P    Cubic I    Cubic F</p>	<p>Three axes at right angles; all equal: <math>a = b = c; \alpha = \beta = \gamma = 90^\circ</math></p>	<p>Copper (Cu), silver (Ag), sodium chloride (NaCl)</p>
 <p style="text-align: center;">Tetragonal P    Tetragonal I</p>	<p>Three axes at right angles; two equal: <math>a = b \neq c; \alpha = \beta = \gamma = 90^\circ</math></p>	<p>White tin (Sn), rutile (TiO<sub>2</sub>), <math>\beta</math>-spodumene (LiAlSi<sub>2</sub>O<sub>6</sub>)</p>
 <p style="text-align: center;">P    C    I    F Orthorhombic</p>	<p>Three axes at right angles; all unequal: <math>a \neq b \neq c; \alpha = \beta = \gamma = 90^\circ</math></p>	<p>Gallium (Ga), perovskite (CaTiO<sub>3</sub>)</p>
 <p style="text-align: center;">Monoclinic P    Monoclinic C</p>	<p>Three axes, one pair not at right angles, of any lengths: <math>a \neq b \neq c; \alpha = \gamma = 90^\circ \neq \beta</math></p>	<p>Gypsum (CaSO<sub>4</sub> • 2H<sub>2</sub>O)</p>
 <p style="text-align: center;">Triclinic P</p>	<p>Three axes not at right angles, of any lengths: <math>a \neq b \neq c; \alpha \neq \beta \neq \gamma \neq 90^\circ</math></p>	<p>Potassium chromate (K<sub>2</sub>CrO<sub>7</sub>)</p>
 <p style="text-align: center;">Trigonal R (rhombohedral)</p>	<p>Rhombohedral: three axes equally inclined, not at right angles; all equal: <math>a = b = c; \alpha = \beta = \gamma \neq 90^\circ</math></p>	<p>Calcite (CaCO<sub>3</sub>), arsenic (As), bismuth (Bi)</p>
 <p style="text-align: center;">Trigonal and hexagonal C (or P)</p>	<p>Hexagonal: three equal axes coplanar at 120°, fourth axis at right angles to these: <math>a_1 = a_2 = a_3 \neq c;</math> <math>\alpha = \beta = 90^\circ, \gamma = 120^\circ</math></p>	<p>Zinc (Zn), cadmium (Cd), quartz (SiO<sub>2</sub>) [P]</p>

**Symmetry:** Symmetry refers to certain pattern or arrangement. A body is symmetrical when it is reproduced by certain operation. Symmetry operations are as follows:

- Translation
- Rotation
- Reflection
- Inversion



## METALLURGICAL MICROSCOPE

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Optical microscopes are categorized on a structure basis according to the intended purpose. An upright microscope (left photo) which observes a specimen (object to be observed) from above is widely known as the most common type with a multitude of uses. An inverted microscope (right photo) which observes a specimen from beneath is used for observing the mineralogy and metallurgy specimens, etc.



### Microscope Parts and Functions

1. **Eyeiece:** The eyepiece (sometimes called the 'ocular') is the lens of the microscope closest to the eye that you look through. It is half of the magnification equation (eyepiece power multiplied by objective power equals magnification), and magnifies the image made by the objective lens. Sometimes called the virtual image. Eyepieces come in many different powers. One can identify which power any given eyepiece is by the inscription on the eyecup of the lens, such as "5x", "10x", or "15X". Oculars are also designed with different angles of view; the most common is the wide field (W.F.).
2. **Eyeiece Holder:** This simply connects the eyepiece to the microscope body, usually with a setscrew to allow the user to easily change the eyepiece to vary magnifying power.
3. **Body:** The main structural support of the microscope which connects the lens apparatus to the base.
4. **Nose Piece:** This connects the objective lens to the microscope body. With a turret, or rotating nose piece as many as five objectives can be attached to create different powers of magnification when rotated into position and used with the existing eyepiece.
5. **Objective:** The lens closest to the object being viewed which creates a magnified image in an area called the "primary image plane". This is the other half of the microscope magnification equation (eyepiece power times objective power equals magnification). Objective lenses have many designs and qualities which differ with each manufacturer. Usually inscribed on the barrel of the objective lens is the magnification power and the numerical aperture (a measure of the limit of resolution of the lens).
6. **Focusing Mechanism:** Adjustment knobs to allow coarse or fine (hundredths of a millimeter) variations in the focusing of the stage or objective lens of the microscope.

7. Stage: The platform on which the prepared slide or object to be viewed is placed. A slide is usually held in place by spring-loaded metal stage clips. More sophisticated high-powered microscopes have mechanical stages which allow the viewer to smoothly move the stage along the X (horizontal path) and Y (vertical path) axis. A mechanical stage is a must for high-power observing.
8. Illumination Source: The means employed to light the object to be viewed. The simplest is the illuminating mirror which reflects an ambient light source to light the object. Many microscopes have an electrical light source for easier and more consistent lighting. Generally electrical light sources are either tungsten or fluorescent, the fluorescent being preferred because it operates at a cooler temperature. Most microscopes illuminate from underneath, through the object, to the objective lens. On the other hand, stereo microscopes use both top and bottom illumination.
9. Base: The bottom or stand upon which the entire microscope rests or is connected.
10. Photography unit with CMOS or CCD sensor able to make pictures via microscope.

## **OBSERVATIONS**

1. **Draw a neat sketch of crystal structures (14 Bravais Lattices)**



*Identify the parts of the metallurgical microscope given below and write down how to operate it.*









## EXPERIMENT 2

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### SPECIMEN PREPARATION FOR METALLOGRAPHIC ANALYSIS

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**Objective:** Preparation of a Specimen for metallographic examination.

#### FOR METALS

When preparing samples for microscopy, it is important to produce something that is representative of the whole specimen. It is not always possible to achieve this with a single sample. Indeed, it is always good practice to mount samples from a material under study in more than one orientation. The variation in material properties will affect how the preparation should be handled, for example very soft or ductile materials may be difficult to polish mechanically.

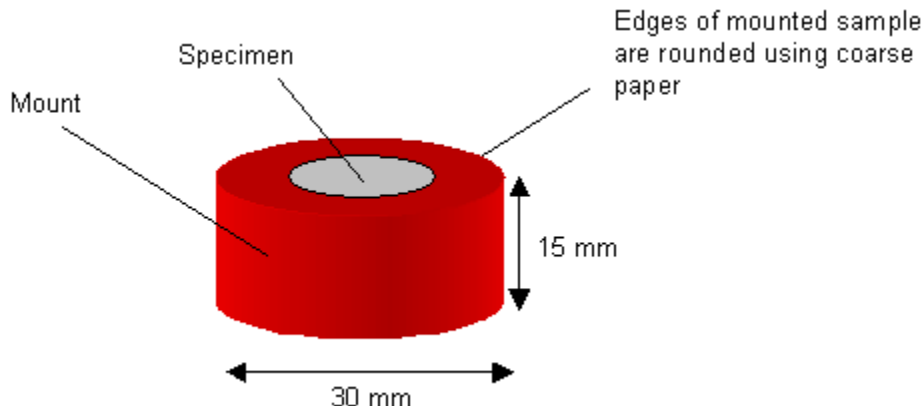
#### Cutting a specimen

It is important to be alert to the fact that preparation of a specimen may change the microstructure of the material, for example through heating, chemical attack, or mechanical damage. The amount of damage depends on the method by which the specimen is cut and the material itself.

Cutting with abrasives may cause a large amount of damage, whilst the use of a low-speed diamond saw can cause fewer problems. There are many different cutting methods, although some are used only for specific specimen types.

#### Mounting

Mounting of specimens is usually necessary to allow them to be handled easily. It also minimises the amount of damage likely to be caused to the specimen itself. The mounting material used should not influence the specimen as a result of chemical reaction or mechanical stresses. It should adhere well to the specimen and, if the specimen is to be electro polished (an Electrolytic process) or examined under a Scanning Electron Microscope, then the mounting material should also be electrically conducting.

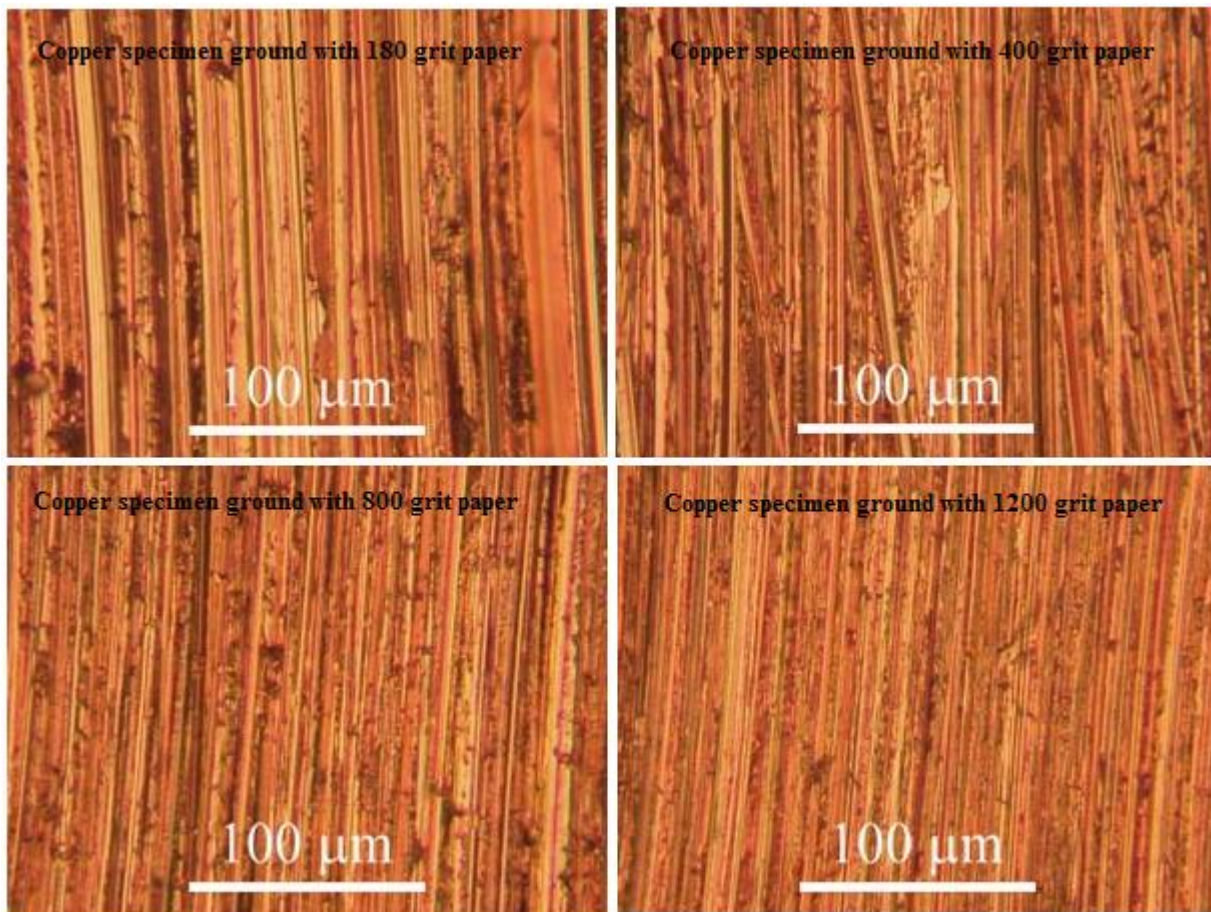


Specimens can be hot mounted (at around 200°C) using a mounting press, either in a thermosetting plastic (*e.g.* phenolic resin), or a thermos softening plastic (*e.g.* acrylic resin). If hot mounting will alter the structure of the specimen a cold-setting resin can be used, *e.g.* epoxy,

acrylic or polyester resin. Porous materials must be impregnated by resin before mounting or polishing, to prevent grit, polishing media or etchant being trapped in the pores, and to preserve the open structure of the material. A mounted specimen usually has a thickness of about half its diameter, to prevent rocking during grinding and polishing. The edges of the mounted specimen should be rounded to minimise the damage to grinding and polishing discs.

## Grinding

Surface layers damaged by cutting must be removed by grinding. Mounted specimens are ground with rotating discs of abrasive paper flushed with a suitable coolant to remove debris and heat, for example wet silicon carbide paper. The coarseness of the paper is indicated by a number: the number of grains of silicon carbide per square inch. So, for example, 180 grit paper is coarser than 1200.



The grinding procedure involves several stages, using a finer paper (higher number) for each successive stage. Each grinding stage removes the scratches from the previous coarser paper. This is more easily achieved by orienting the specimen perpendicular to the previous scratches, and watching for these previously oriented scratches to be obliterated. Between each grade the specimen is washed thoroughly with soapy water to prevent contamination from coarser grit present on the specimen surface. Typically, the finest grade of paper used is the 1200, and once the only scratches left on the specimen are from this grade, the specimen is thoroughly washed

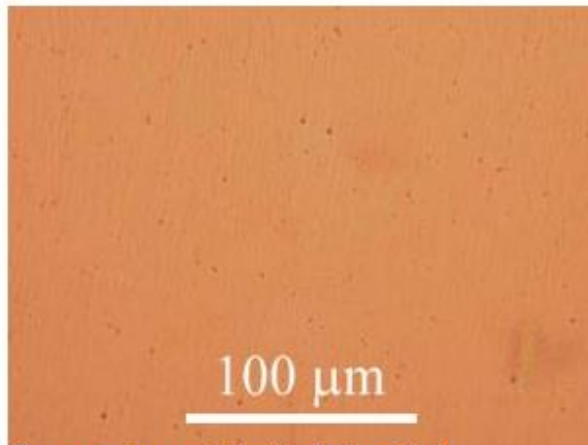
with water, followed by alcohol and then allowed to dry. It is possible to determine the start point for grinding using the following empirical relationship where the width of the largest scratch is measured under a microscope:

$$\text{Paper grit size} = \frac{16000}{\text{Width of largest scratch (in microns)}}$$

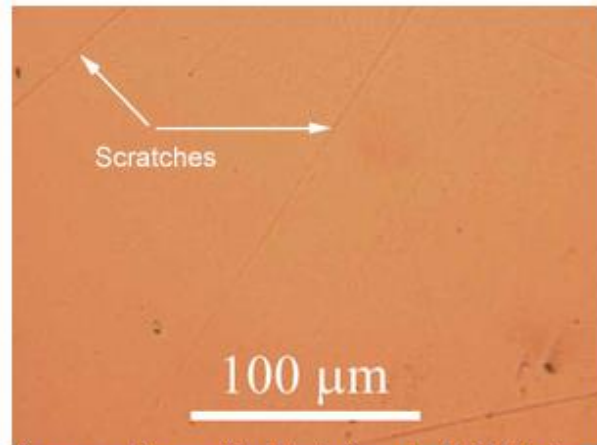
1. This prevents putting more damage into the sample than already exists; the coarsest grades of paper are often not useful.
2. Cleaning specimens in an ultrasonic bath can also be helpful, but is not essential.
3. The series of photos below shows the progression of the specimen when ground with progressively finer paper.

### Polishing

Polishing discs are covered with soft cloth impregnated with abrasive diamond particles and an oily lubricant. Particles of two different grades are used : a coarser polish - typically with diamond particles 6 microns in diameter which should remove the scratches produced from the finest grinding stage, and a finer polish – typically with diamond particles 1 micron in diameter, to produce a smooth surface. Before using a finer polishing wheel the specimen should be washed thoroughly with warm soapy water followed by alcohol to prevent contamination of the disc.



**Copper specimen polished to 6 micron level**



**Copper specimen polished to 1 micron level. Ideally there should be no scratches after polishing, but it is often hard to completely remove them all**

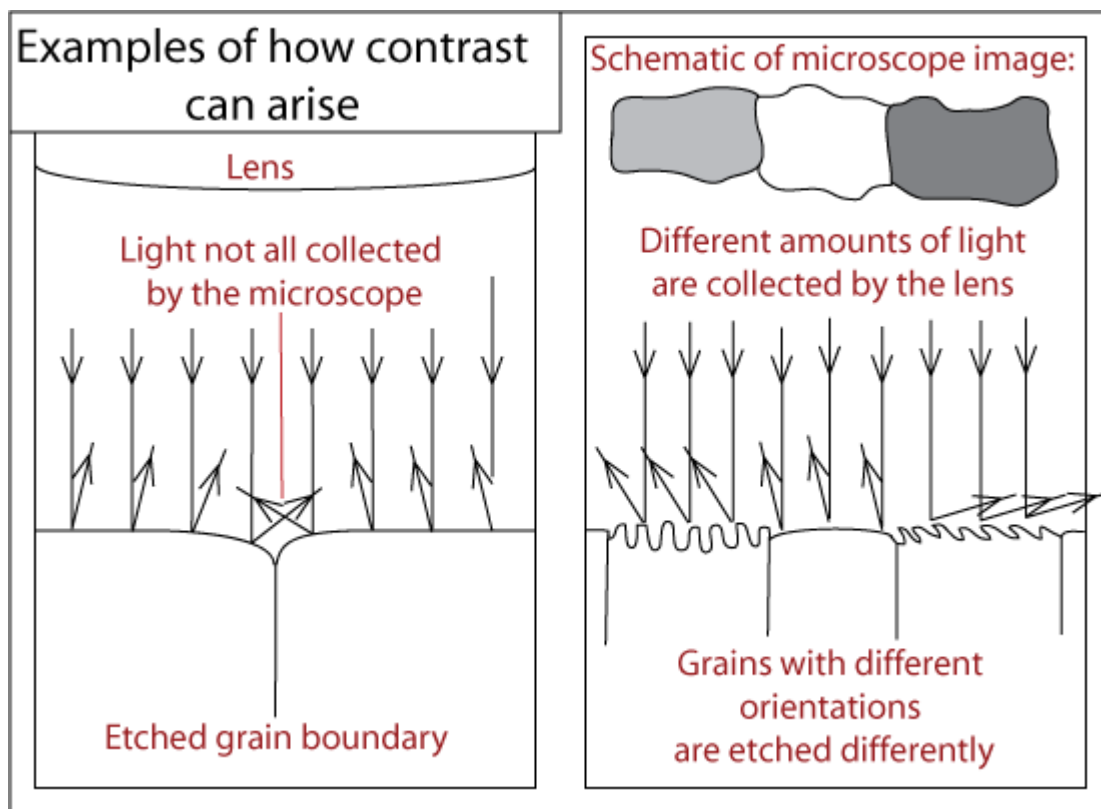
Mechanical polishing will always leave a layer of disturbed material on the surface of the specimen, if the specimen is particularly susceptible to mechanical damage (or excessive force is used in the grinding and polishing stages) debris can become embedded in the surface and plastic deformation may exist below the surface. Electro polishing or chemical polishing can be used to remove this, leaving an undisturbed surface.

## Etching

Etching is used to reveal the microstructure of the metal through selective chemical attack. It also removes the thin, highly deformed layer introduced during grinding and polishing.

In alloys with more than one phase, etching creates contrast between different regions through differences in topography or reflectivity. The rate of etching is affected by crystallographic orientation, the phase present and the stability of the region. This means contrast may arise through different mechanisms – therefore revealing different features of the sample.

In all samples, etchants will preferentially attack high energy sites, such as boundaries and defects.

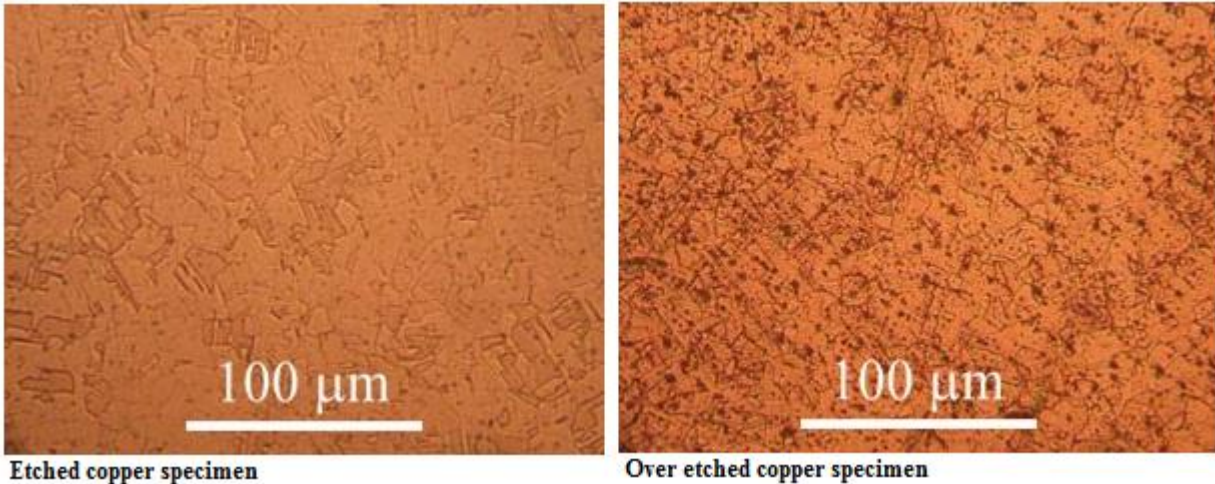


The specimen is etched using a reagent. For example, for etching stainless steel or copper and its alloys, a saturated aqueous solution of ferric chloride, containing a few drops of hydrochloric acid is used. This is applied using a cotton bud wiped over the surface a few times (Care should be taken not to over-etch - this is difficult to determine, however, the photos below may be of some help). The specimen should then immediately be washed in alcohol and dried.

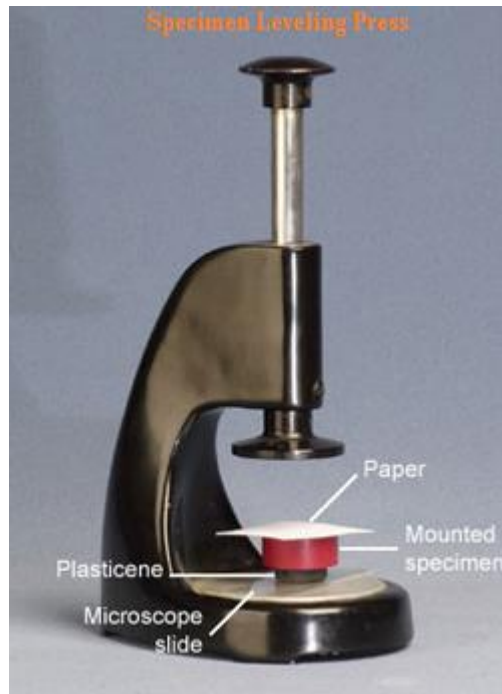
Following the etching process there may be numerous small pits present on the surface. These are etch pits caused by localised chemical attack and, in most cases, they do not represent features of the microstructure. They may occur preferentially in regions of high local disorder, for example where there is a high concentration of dislocations.



If the specimen is over etched, i.e. etched for too long, these pits tend to grow, and obscure the main features to be observed. If this occurs it may be better to grind away the poorly etched surface and re-polish and etch, although it is important to remember what features you are trying to observe – repeatedly grinding a very thin sample may leave nothing to see.



Ideally the surface to be examined optically should be flat and level. If it is not, the image will pass in and out of focus as the viewing area is moved across the surface. In addition, it will make it difficult to have the whole of the field of view in focus - while the centre is focused, the sides will be out of focus. By using a specimen levelling press (shown below) this problem can be avoided, as it presses the mounted specimen into plasticene on a microscope slide, making it level. A small piece of paper or cloth covers the surface of the specimen to avoid scratching.



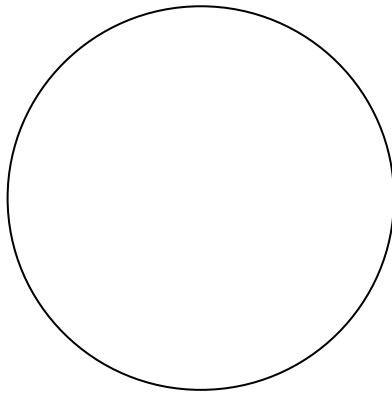
The following table lists the most commonly used etchants.

Etchant	Composition	Conc.	Conditions	Comments
<b>Kalling's No. 1</b>	Distilled water CuCl <sub>2</sub> Hydrochloric acid Ethanol	33 ml 1.5 gm 33 ml 33 ml	Immersion etching at 20 degrees Celcius	For etching martensitic stainless steels. Martensite will be dark and the ferrite will be colored.
<b>Kalling's No. 2</b>	CuCl <sub>2</sub> Hydrochloric acid Ethanol	5 grams 100 ml 100 ml	Immersion etching at 20 degrees Celcius	For etching duplex stainless steels and Ni-Cu alloys and superalloys.
<b>Kellers Etch</b>	Distilled water Nitric acid Hydrochloric acid Hydrofluoric acid	190 ml 5 ml 3 ml 2 ml	10-30 second immersion. Use only fresh etchant	Excellent for aluminum and alloys - immersion for 10-20 seconds ; titanium alloys immersion for 10-20 seconds.
<b>Kroll's Reagent</b>	Distilled water Nitric acid Hydrofluoric acid	92 ml 6 ml 2 ml	15 seconds	Excellent for titanium and alloys. Swab specimen up to 20 seconds.
<b>Nital</b>	Ethanol Nitric acid	100 ml 1-10 ml	Seconds to minutes	Most common etchant for Fe, carbon and alloys steels and cast iron - Immerse sample up from seconds to minutes; Mn-Fe, MnNi, Mn-Cu, Mn-Co alloys - immersion up to a few minutes.
<b>Marble's Reagent</b>	CuSO <sub>4</sub> Hydrochloric acid Water	10 grams 50 ml 50 ml	Immerse or swab for 5-60 seconds.	For etching Ni, Ni-Cu and Ni-Fe alloys and superalloys. Add a few drops of H <sub>2</sub> SO <sub>4</sub> to increase activity.
<b>Murakami's</b>	K <sub>3</sub> Fe(CN) <sub>6</sub> KOH Water	10 grams 10 grams 100 ml	Pre-mix KOH and water before adding K <sub>3</sub> Fe(CN) <sub>6</sub>	Cr and alloys (use fresh and immerse); iron and steels reveals carbides; Mo and alloys uses fresh and immerse; Ni-Cu alloys for alpha phases use at 75°C; W and alloys use fresh and immerse; WC-Co and complex sintered carbides.
<b>Picral</b>	Ethanol Picric acid	100 ml 2-4 grams	Seconds to minutes Do not let etchant crystallize or dry - <b>explosive</b>	Recommended for microstructures containing ferrite and carbide.
<b>Vilella's Reagent</b>	Glycerol Nitric acid Hydrochloric acid	45 ml 15 ml 30 ml	Seconds to minutes	Good for ferrite-carbide structures (tempered martensite) in iron and steel

# OBSERVATION

**Sample No:**

*Draw the microstructure in the circle given below and write down the phases*



Magnification used \_\_\_\_\_

The phases present in the microstructure and the approximate % of major phases are

	Phases Present	Percentage
➤	-----	-----
➤	-----	-----
➤	-----	-----
➤	-----	-----

Etchant used \_\_\_\_\_

From the observation of microstructure, the given sample is .....

Any other observation .....

### **Answer the following Questions**

1. Why specimen preparation is important in metallurgy?
2. Why must metallographic samples be washed and carefully dried before proceeding from one grinding or polishing operation to the next?
3. State the principle of metallurgical microscope?
4. What is the purpose of etching metallographic samples?
5. Why etchants are different for different metals?





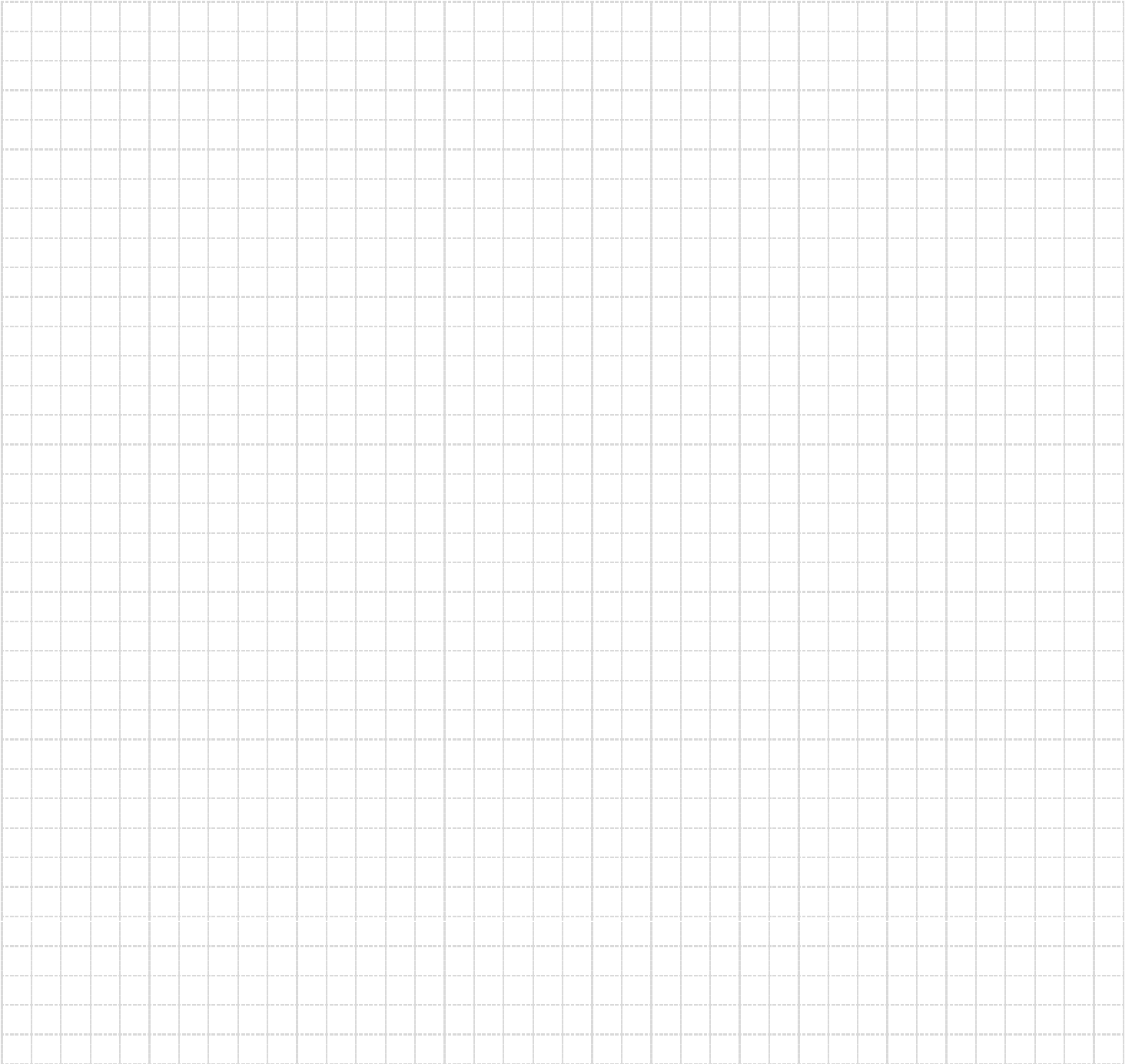
EXPERIMENT 3

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**IRON-IRON CARBIDE PHASE DIAGRAM**

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**1. Draw Fe-Fe<sub>3</sub>C Phase Diagram and identify the phases**



The following phases are involved in the transformation, occurring with iron-carbon alloys:

- ❖  **$\alpha$ -ferrite** – Interstitial solid solution of carbon in  $\alpha$ -iron.  $\alpha$ -ferrite has BCC crystal structure. It is a low temperature phase. The solubility of carbon in  $\alpha$ -Fe is low. Maximum solubility of carbon is 0.025% at 723°C. Ferrite is a soft phase with a hardness of 80-150 HV.
- ❖ **Austenite** – Interstitial solid solution of carbon in  $\gamma$ -iron. Austenite has FCC crystal structure, permitting high solubility of carbon – up to 2.1% at 1147 °C. Austenite does not exist below 723°C and maximum carbon concentration at this temperature is 0.83%. Austenite is normally a metastable phase however, it can be made into a stable phase by alloying elements. Hardness of Austenite is 200-250 HB.
- ❖  **$\delta$ -ferrite** – Solid solution of carbon in  $\delta$ -iron. Maximum concentration of carbon in  $\delta$ -ferrite is 0.1% at 1493°C i.e. at temperature of the peritectic transformation. The crystal structure of  $\delta$ -ferrite is BCC.
- ❖ **L** - Liquid solution of carbon in iron;
- ❖ **Cementite** – Iron carbide, intermetallic compound, having fixed composition Fe<sub>3</sub>C. It has a complex orthorhombic crystal structure. Cementite is a hard and brittle substance, influencing on the properties of steels and cast irons. The hardness of cementite is 800-1400 HV.
- ❖ **Graphite** – Free carbon, soft phase. It has hexagonal crystal structure. Improves machinability and damping properties. Reduces shrinkage and reduces strength severely, depending upon the shape.
- ❖ **Pearlite** – A metastable lamellar aggregate of ferrite and cementite formed due to eutectoid transformation. Provides strength to the steel or cast iron with reduced brittleness. Imparts good machinability. The hardness of pearlite phase is approximately 230 HV.
- ❖ **Ledeburite**– Massive eutectic phase composed of austenite and cementite. Austenite on cooling transforms into cementite and pearlite. Hard and virtually un-machinable.

The following phase transformations occur with iron-carbon alloys:

Alloys, containing up to 0.51% of carbon, start solidification with formation of crystals of  $\delta$ -ferrite. Carbon content in  $\delta$ -ferrite increases up to 0.1% in course solidification, and at 1493°C remaining liquid phase and  $\delta$ -ferrite perform peritectic transformation, resulting in formation of austenite.

Alloys, containing carbon more than 0.51%, but less than 2.1%, form primary austenite crystals in the beginning of solidification and when the temperature reaches the curve  $A_{CM}$  primary cementite starts to form.

Iron-carbon alloys, containing up to 2.1% of carbon, are called steels.

Alloys, containing from 2.1% to 6.67% of carbon, experience eutectic transformation at 2097 °F (1147 °C). The eutectic concentration of carbon is 4.3%.

In practice only hypoeutectic alloys are used. These alloys (carbon content from 2.1% to 4.3%) are called cast irons. When temperature of an alloy from this range reaches 1147 °C, it contains primary austenite crystals and some amount of the liquid phase. The latter decomposes by eutectic mechanism to a fine mixture of austenite and cementite, called ledeburite.

All iron-carbon alloys (steels and cast irons) experience eutectoid transformation at 723°C. The eutectoid concentration of carbon is 0.8%.

When the temperature of an alloy reaches 723°C, austenite transforms to pearlite (fine ferrite-cementite structure, forming as a result of decomposition of austenite at slow cooling conditions).

### **Critical temperatures**

- ❖ **Upper critical temperature (point)  $A_3$**  is the temperature, below which ferrite starts to form as a result of rejection from austenite in the hypoeutectoid alloys.
- ❖ **Upper critical temperature (point)  $A_{CM}$**  is the temperature, below which cementite starts to form as a result of rejection from austenite in the hypereutectoid alloys.
- ❖ **Lower critical temperature (point)  $A_1$**  is the temperature of the austenite-to-pearlite eutectoid transformation. Below this temperature austenite does not exist.
- ❖ **Magnetic transformation temperature  $A_2$**  is the temperature below which  $\alpha$ -ferrite is ferromagnetic.

### **Phase compositions of the iron-carbon alloys at room temperature**

- ❖ Hypoeutectoid steels (carbon content from 0 to 0.8%) consist of primary (proeutectoid) ferrite (according to the curve  $A_3$ ) and pearlite.
- ❖ Eutectoid steel (carbon content 0.8%) entirely consists of pearlite.
- ❖ Hypereutectoid steels (carbon content from 0.8 to 2.1%) consist of primary (proeutectoid) cementite (according to the curve  $A_{CM}$ ) and pearlite.

- ❖ Cast irons (carbon content from 2.1% to ~4.3%) consist of proeutectoid cementite rejected from austenite according to the curve  $A_{CM}$ , pearlite and transformed ledeburite (ledeburite in which austenite transformed to pearlite).

### The Lever Rule

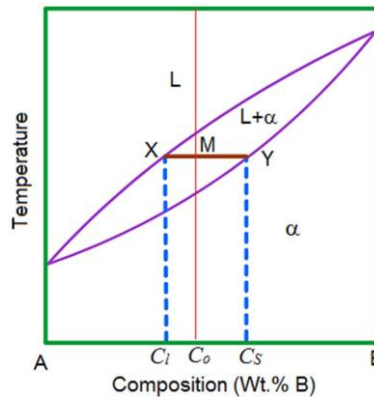
Lever rule derived from mass balance gives relative amounts of the co-existing phases. The tie-line is treated as a lever arm, with the fulcrum at the overall composition. For the arm to be horizontal, the weight to be hung at each end must be proportional to the arm length on the other side of the fulcrum. The “weight” at each end corresponds to the amount of the phase at that end.

#### Procedure to find equilibrium relative amounts of phases

- A tie-line is constructed across the two phase region at the temperature of the alloy to intersect the region boundaries.
- The relative amount of a phase is computed by taking the length of tie-line from overall composition to the phase boundary for the other phase, and dividing by the total tie-line length. From figure 3, relative amounts of liquid and solid phases is given respectively by

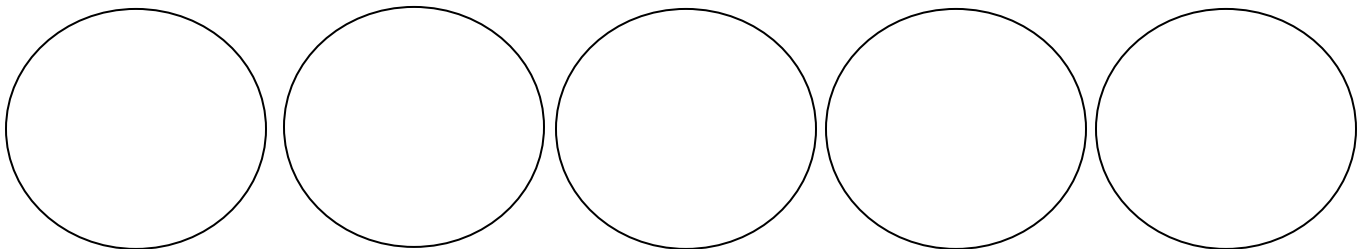
$$C_L = \frac{MY}{XY}, \quad C_S = \frac{UC}{UV}, \quad C_S + C_L = 1$$

The lever rule cannot be applied at an invariant temperature, where three phases are in equilibrium. It can, however, be applied just above or below the invariant line.



### OBSERVATIONS

**Theoretically draw the microstructures of Ferrite, Austenite, Hypo-eutectoid, Pearlite, Hyper-eutectoid steels.**



### Answer the following Questions

1. Why  $\beta$  phase is not appeared in the Fe-Fe<sub>3</sub>C phase diagram?
2. Why A<sub>CM</sub> line is steeper than A<sub>3</sub> line?
3. Why does proeutectoid phase (ferrite or cementite) form along austenite grain boundaries?
4. Compute the mass fractions of  $\alpha$  ferrite and cementite in pearlite?
5. In a hypo-eutectoid steel, both eutectoid and proeutectoid ferrite exist. Explain difference between them. What will be the carbon concentration in each?



## EXPERIMENT 4

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### MICROSTRUCTURAL ANALYSIS OF LOW & MEDIUM CARBON STEELS

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#### 1. Objective

1. To be familiar with metallographic techniques such as grinding, polishing of carbon steels
2. To be familiar with etching techniques used in different type of steels
3. To study the microstructure of carbon steels and low alloy steels

#### 2. AISI-SAE Classifications of Steels

*Major classification of steel as per AISI, SAE and ASME*

AISI- American Iron and Steel Institute

SAE- Society of Automotive Engineers

ASME- American Society of Mechanical Engineers

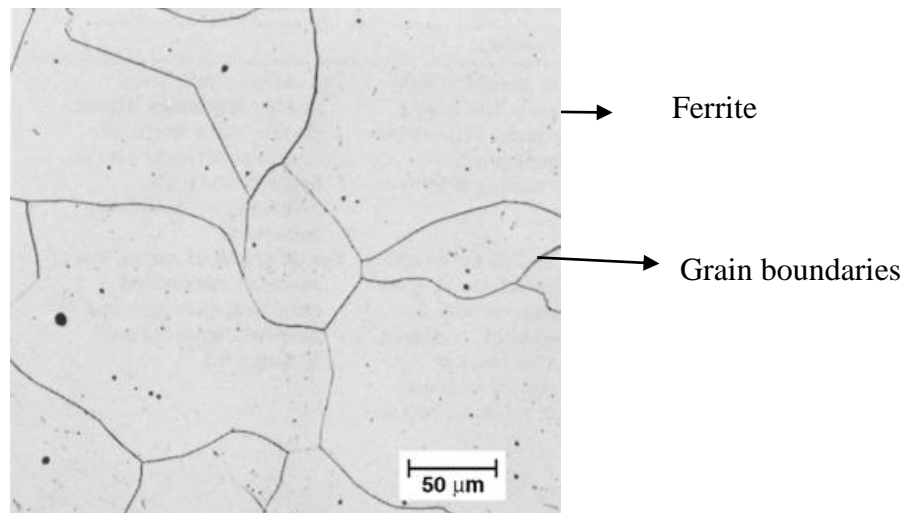
#### 3. Applications

Very-low-carbon sheet steels are used in the electrical appliance and power transmission industry. Electric motors contain hundreds of steel sheets called motor laminations that are stacked and wound with copper wire in the rotor and stator of the motor. Very-low-carbon 3.25% Si sheet steels are used as laminations in electrical power transformers. A thin iron oxide surface layer or organic coating electrically insulates these steel laminations from each other in order to minimize power losses

#### 4. Hypoeutectoid steel

**Microstructure of steel containing <0.02% C**

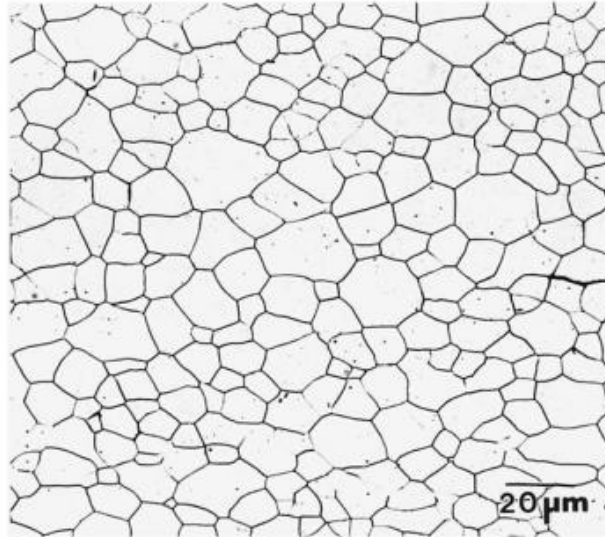
Fig 1 shows the microstructure of low carbon steel etched with 2% nital. The microstructure shows ferrite grains.





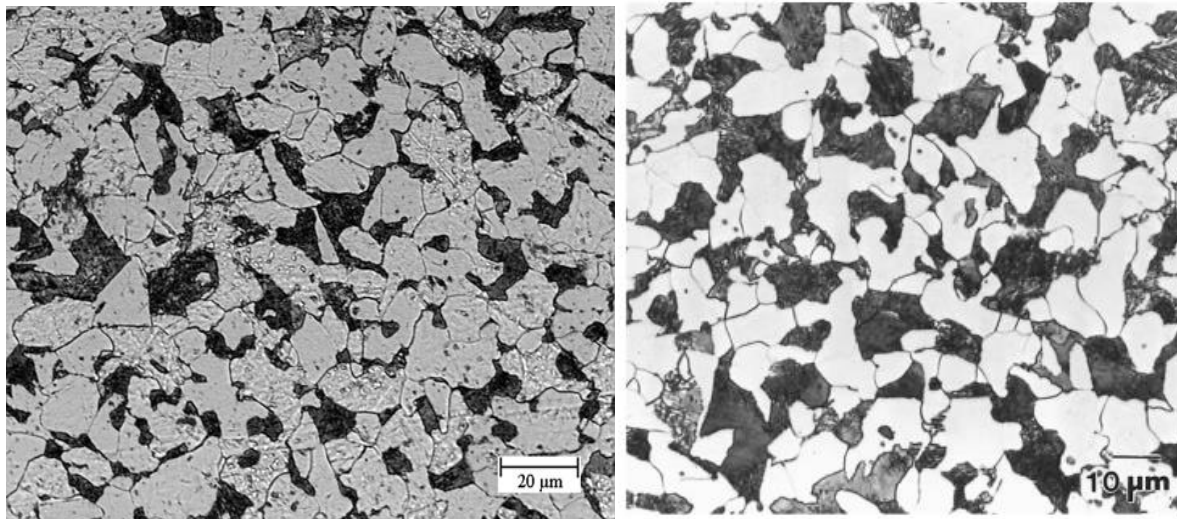
**Fig.1:** Microstructure of low carbon steel etched with nital

Fig.2 shows fine grains of ferrite etched with Marshall's reagent.



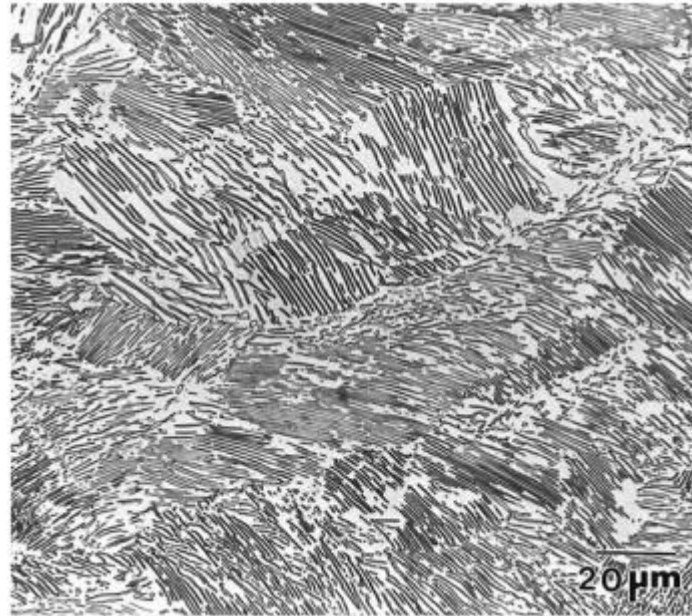
**Fig. 2:** Ferrite grains in a low-carbon (0.02% C) steel. Marshall's reagent.

**Microstructure of steel containing 0.02- 0.4% C**



**Figure 3.** The microstructure of AISI 1040 steel showing ferrite and pearlite. The mean grain diameter is 26.173 μm. Etchant 2% Nital.

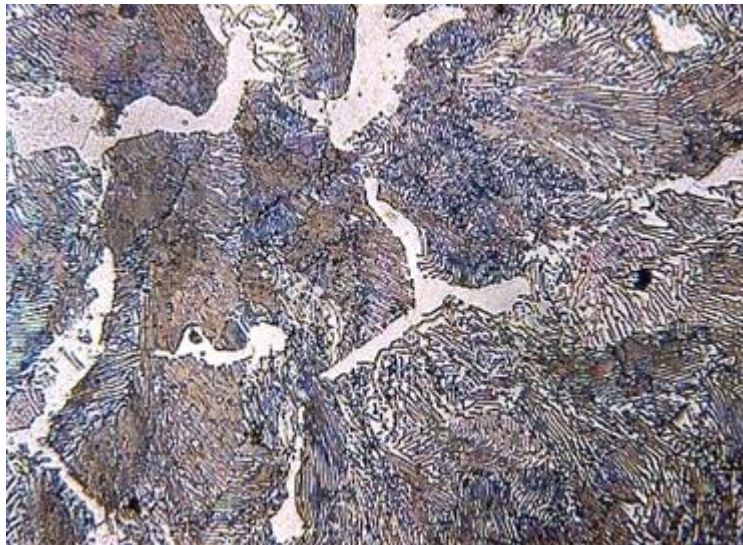
### Microstructure of eutectoid steel containing 0.8% C



**Figure 4.** The microstructure of AISI/SAE 1080 steel showing pearlite etched with 4% Picral.

### Hypereutectoid steel

### Microstructure of High Carbon Steel containing >0.8% C



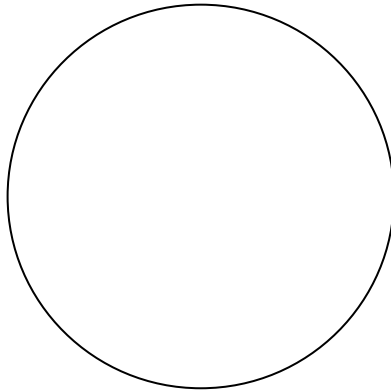
**Figure 5:** The microstructure of high carbon steel showing proeutectoid cementite around pearlite grains.

## OBSERVATIONS

Observe the microstructure and write down the phases present

**Sample No:**

*Draw the microstructure in the circle given below and write down the phases*



Magnification used \_\_\_\_\_

The phases present in the microstructure and the approximate % of major phases are

	Phases Present	Percentage
➤	-----	-----
➤	-----	-----
➤	-----	-----
➤	-----	-----

Etchant used \_\_\_\_\_

From the observation of microstructure, the given sample is .....

.....

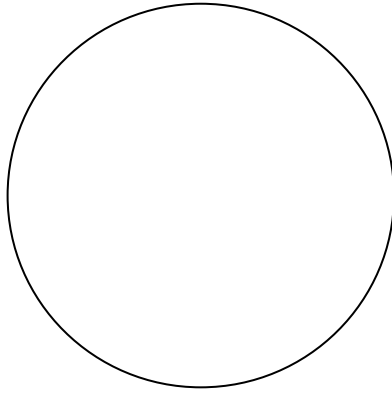
Any other observation .....

.....

.....

**Sample No:**

*Draw the microstructure in the circle given below and write down the phases*



Magnification used \_\_\_\_\_

The phases present in the microstructure and the approximate % of major phases are

	Phases Present	Percentage
➤	-----	-----
➤	-----	-----
➤	-----	-----
➤	-----	-----

Etchant used \_\_\_\_\_

From the observation of microstructure, the given sample is .....

.....

Any other observation .....

.....

.....

## Answer the Following Questions

1. How do you differentiate ferrite and austenite?
2. How do you differentiate ferrite and cementite phases?
3. Define Hypo eutectoid, eutectoid and Hyper eutectoid steels?
4. What is the difference between pro-eutectoid  $\alpha$ , and ferrite  $\alpha$ ?
5. Define equilibrium diagram? Can we call Fe-Fe<sub>3</sub>C is an equilibrium diagram? If yes/no state, the reasons.



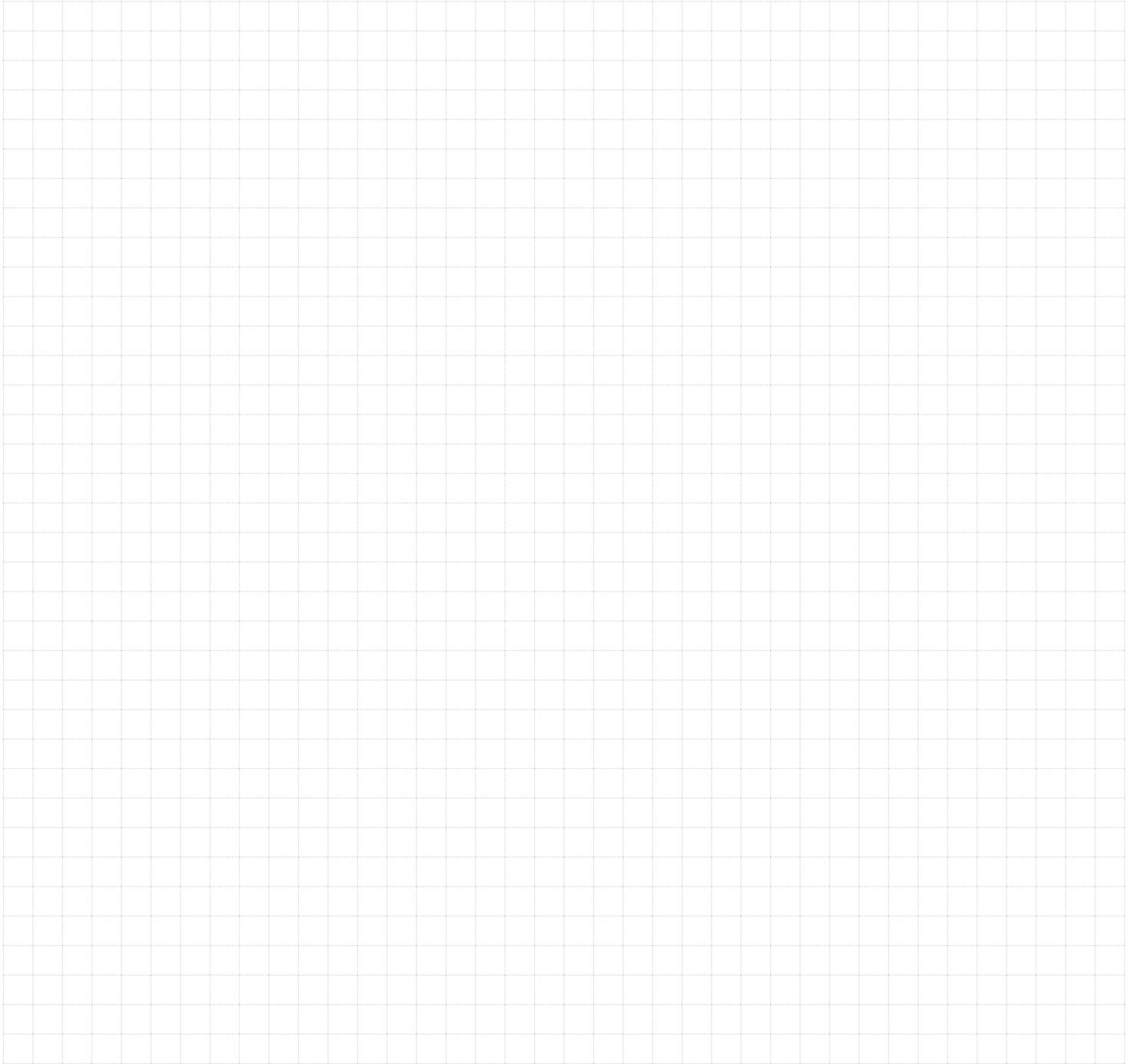
EXPERIMENT 5

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**MICROSTRUCTURAL ANALYSIS OF CAST IRON SAMPLES**

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**1. Draw Fe-C Phase Diagram and Markdown the phases**



## Objective

1. To understand the Iron-carbon equilibrium phase diagram.
2. To be familiar with microscopic observation of phases present in cast iron
3. To find out the type of cast iron

## Theory

**Cast iron** (>2.1% carbon) usually refers to grey iron, but also identifies a large group of ferrous alloys, which solidify with a eutectic. The colour of a fractured surface can be used to identify an alloy. **White cast iron** is named after its white surface when fractured, due to its carbide impurities which allow cracks to pass straight through. **Grey cast iron** is named after its grey fractured surface, which occurs because the graphitic flakes deflect a passing crack and initiate countless new cracks as the material breaks. And other types are **Ductile/Nodular cast iron** and **Malleable cast iron**.

Carbon (C) and silicon (Si) are the main alloying elements, with the amount ranging from 2.1 to 4 wt.% and 1 to 3 wt.%, respectively. While this technically makes these base alloys ternary Fe-C-Si alloys, the principle of cast iron solidification is understood from the binary iron-carbon phase diagram. Since the compositions of most cast irons are around the eutectic point of the iron-carbon system, the melting temperatures closely correlate, usually ranging from 1,150 to 1,200 °C which is about 300 °C lower than the melting point of pure iron.

Cast iron tends to be brittle, except for malleable cast irons. With its relatively low melting point, good fluidity, castability, excellent machinability, resistance to deformation and wear resistance, cast irons have become an material with a wide range of applications and are used in pipes, machines and automotive industry parts, such as cylinder heads (declining usage), cylinder blocks and gearbox cases (declining usage). It is resistant to destruction and weakening by oxidisation (rust).

## Types of cast iron

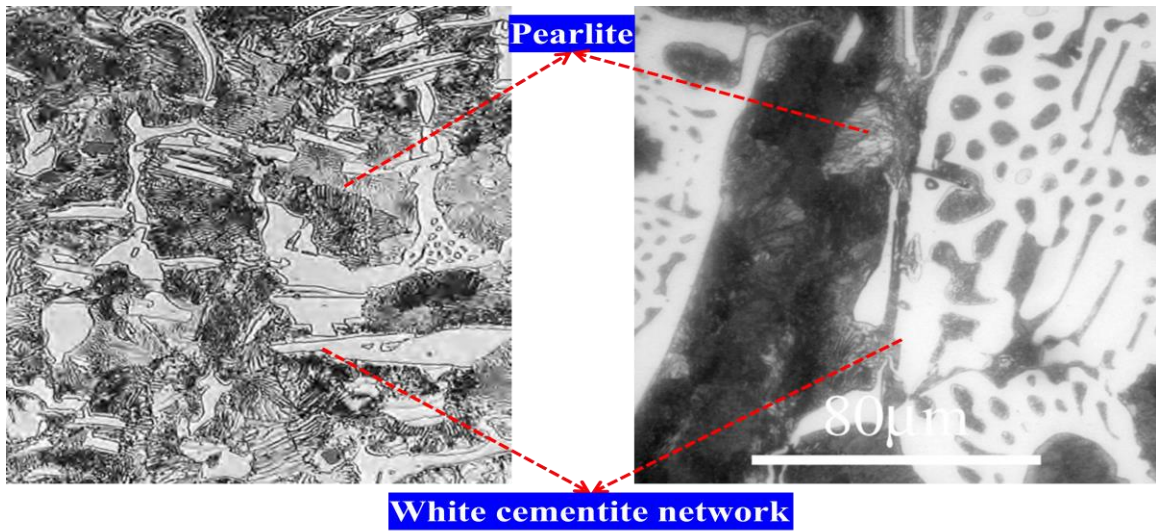
1. Grey cast iron
2. Ductile iron/Spheroidal Graphite iron/Nodular Iron (SG Iron)
3. Compacted Graphite Iron
4. Malleable Iron
5. White cast iron



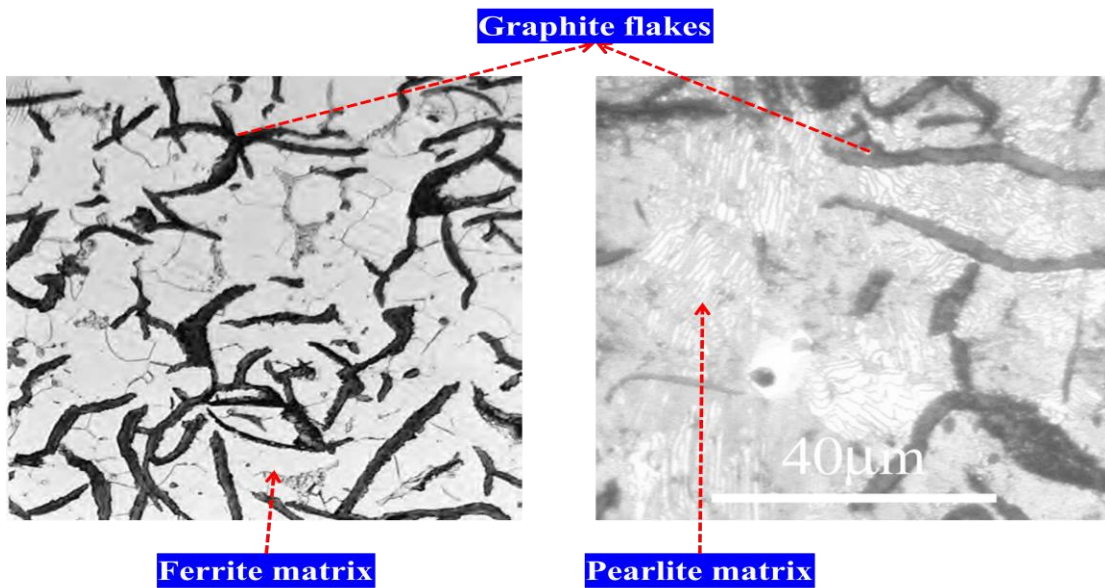
**Table 1. Range of compositions for typical unalloyed Cast Irons**

Type of Iron	Carbon	Silicon	Manganese	Sulfur	Phosphorus
Gray	2.5-4.0	1.0-3.0	0.2-1.0	0.02-0.25	0.02-1.0
Ductile/Nodular	3.0-4.0	1.8-2.8	0.1-1.0	0.01-0.03	0.01-0.1
Compacted Graphite Iron	2.5-4.0	1.0-3.0	0.2-1.0	0.01-0.03	0.01-0.1
Malleable	2. -2.9	0.9-1.9	0.15-1.2	0.02-0.2	0.02-0.2
White	1.8-3.6	0.5-1.9	0.25-0.8	0.06-0.2	0.06-0.2

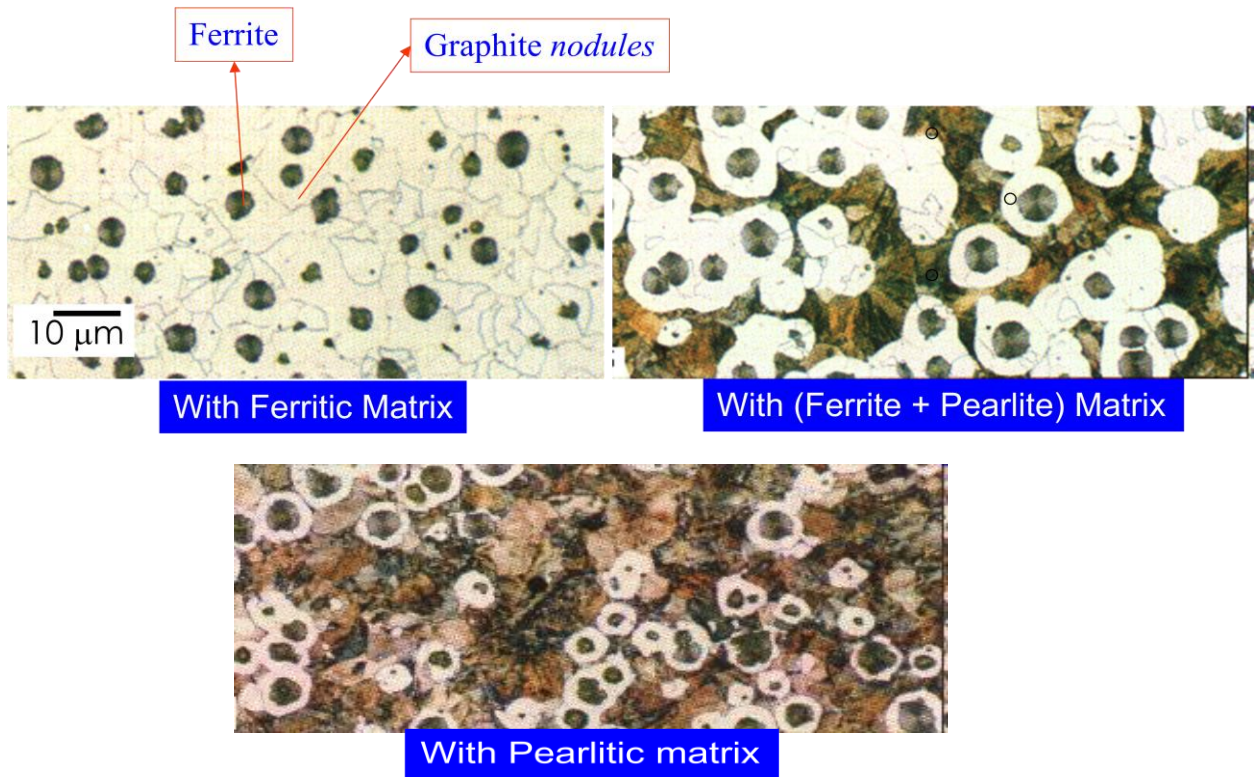
**White Cast Iron**



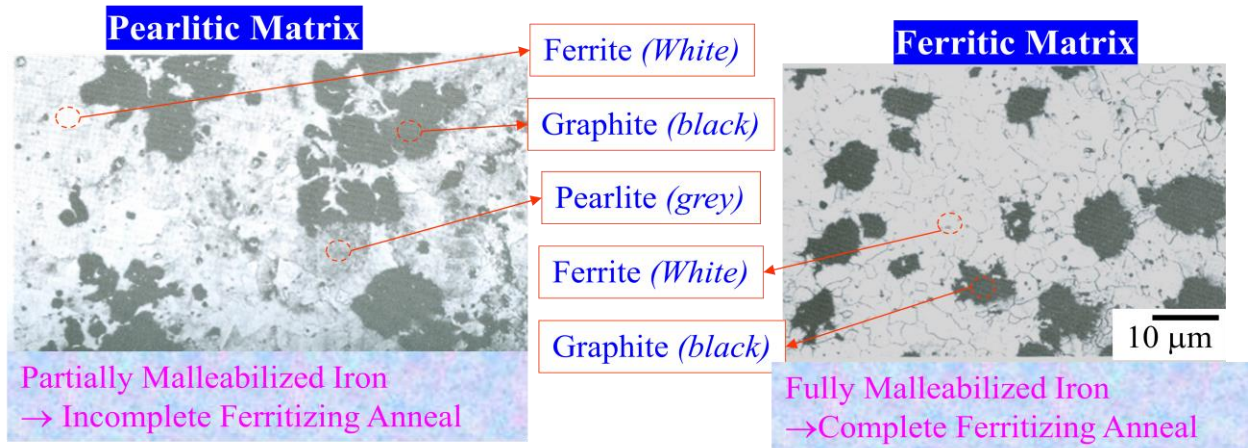
**Grey Cast Iron**



**Ductile / Nodular cast iron (or) Spheroidal graphite (SG) iron**

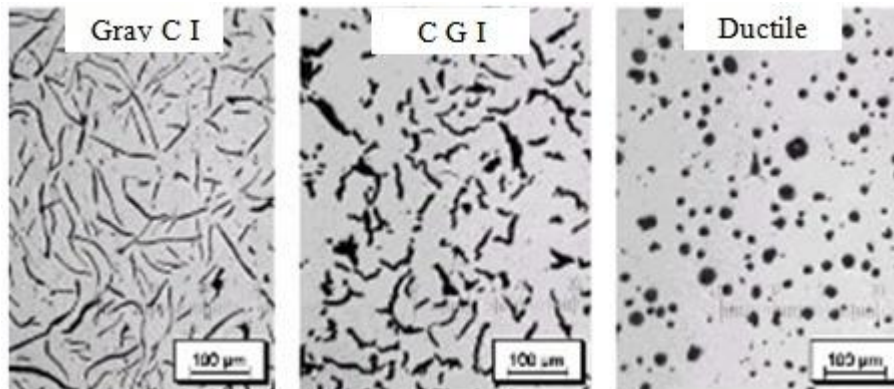
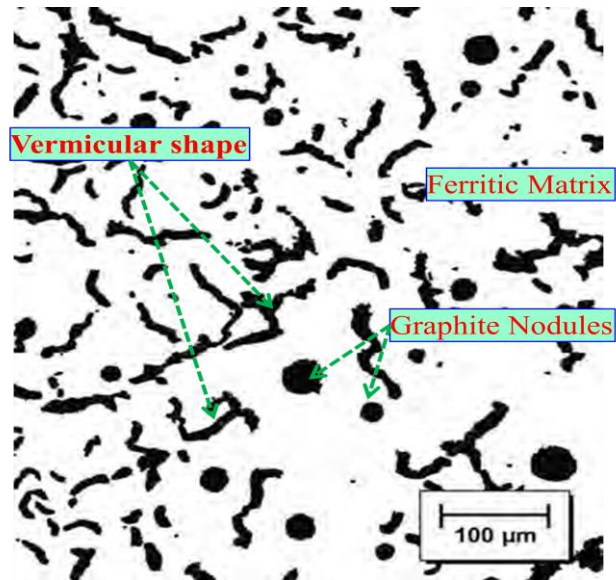


**Malleable cast iron**

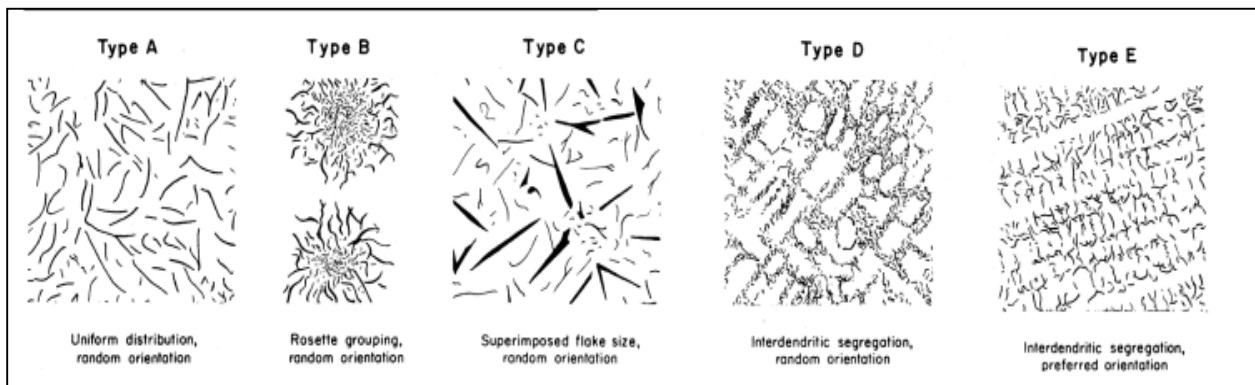




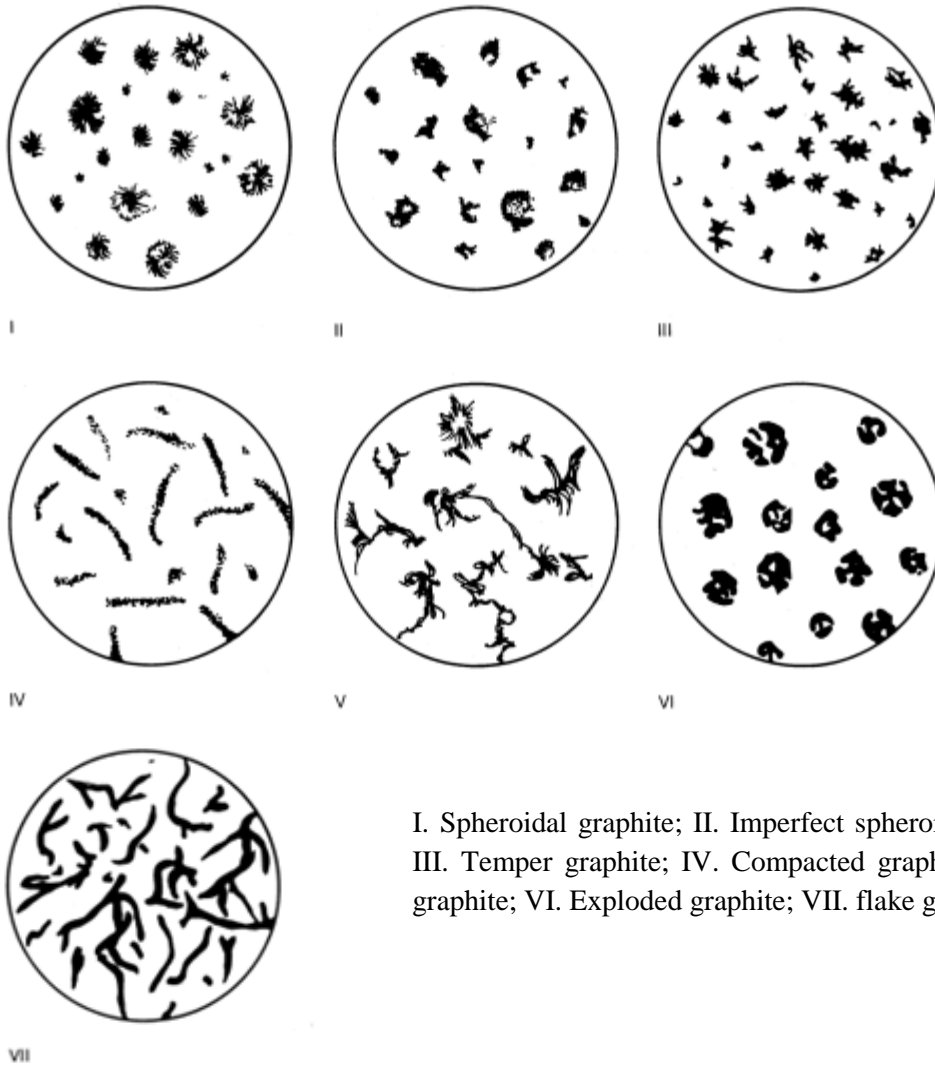
## Compacted graphite cast iron



## Types of Graphite flakes in gray iron



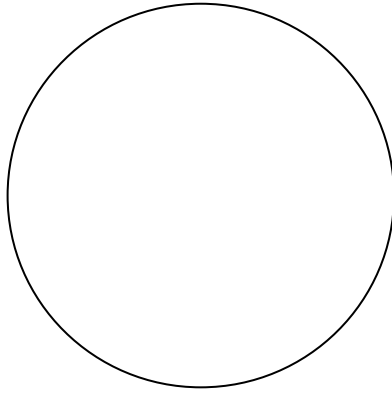
## Typical Graphite shapes in cast iron



I. Spheroidal graphite; II. Imperfect spheroidal graphite; III. Temper graphite; IV. Compacted graphite; V. Crab graphite; VI. Exploded graphite; VII. flake graphite

**Sample No:**

*Draw the microstructure in the circle given below and write down the phases*



Magnification used \_\_\_\_\_

The phases present in the microstructure and the approximate % of major phases are

	Phases Present	Percentage
➤	-----	-----
➤	-----	-----
➤	-----	-----
➤	-----	-----

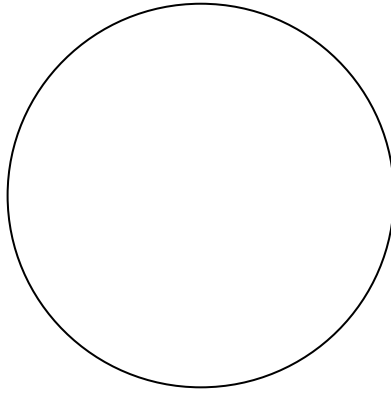
Etchant used \_\_\_\_\_

From the observation of microstructure, the given sample is .....

Any other observation .....

**Sample No:**

*Draw the microstructure in the circle given below and write down the phases*



Magnification used \_\_\_\_\_

The phases present in the microstructure and the approximate % of major phases are

	Phases Present	Percentage
➤	-----	-----
➤	-----	-----
➤	-----	-----
➤	-----	-----

Etchant used \_\_\_\_\_

From the observation of microstructure, the given sample is .....

Any other observation .....

## Answer the Following Questions

1. What would be the differences between Fe-Fe<sub>3</sub>C and Fe-C phase diagram?
2. Compared to Fe-Fe<sub>3</sub>C phase diagram, why does eutectic and eutectoid composition will shift left side in Fe-C phase diagram?
3. Which type of cast iron is ductile in comparison to others? Why?
4. On the basis of microstructure, explain why grey iron is brittle and weak in tension?





8. Compare grey and malleable cast irons with respect to (a) microstructure and (b) mechanical characteristics.

9. Why do intermetallic compounds and pure metals melt at fixed temperature?

10. What is the significance of this experiment? How is it related to your course of study?

## EXPERIMENT 6

### MICROSTRUCTURAL ANALYSIS OF ALUMINUM ALLOYS

#### Objective

1. To be familiar with metallographic preparation techniques of Aluminium alloys
2. To be familiar with microscopic observation of phases present in Aluminium alloys.
3. To interpret the microstructure with phase diagram

#### Theory

Aluminum is a light metal ( $\rho = 2.7 \text{ g/cc}$ ); is easily machinable; has wide variety of surface finishes; good electrical and thermal conductivities; highly reflective to heat and light.

- Versatile metal - can be cast, rolled, stamped, drawn, spun, roll-formed, hammered, extruded and forged into many shapes.
- Aluminum can be riveted, welded, brazed, or resin bonded.
- Corrosion resistant - no protective coating needed, however it is often anodized to improve surface finish, appearance.
- Al and its alloys - high strength-to-weight ratio (high specific strength) owing to low density.
- Such materials are widely used in aerospace and automotive applications where weight savings are needed for better fuel efficiency and performance.
- Al-Li alloys are lightest among all Al alloys and find wide applications in the aerospace industry.

Aluminum alloys are classified into two categories – Cast and Wrought alloys. Wrought alloys can be either heat-treatable or non-heat treatable. Alloys are designated by a 4-digit number. Wrought – the 1<sup>st</sup> digit indicates the major alloying element. Cast – The last digit after the decimal indicates product from (casting - 0 or ingot -1)

#### Wrought

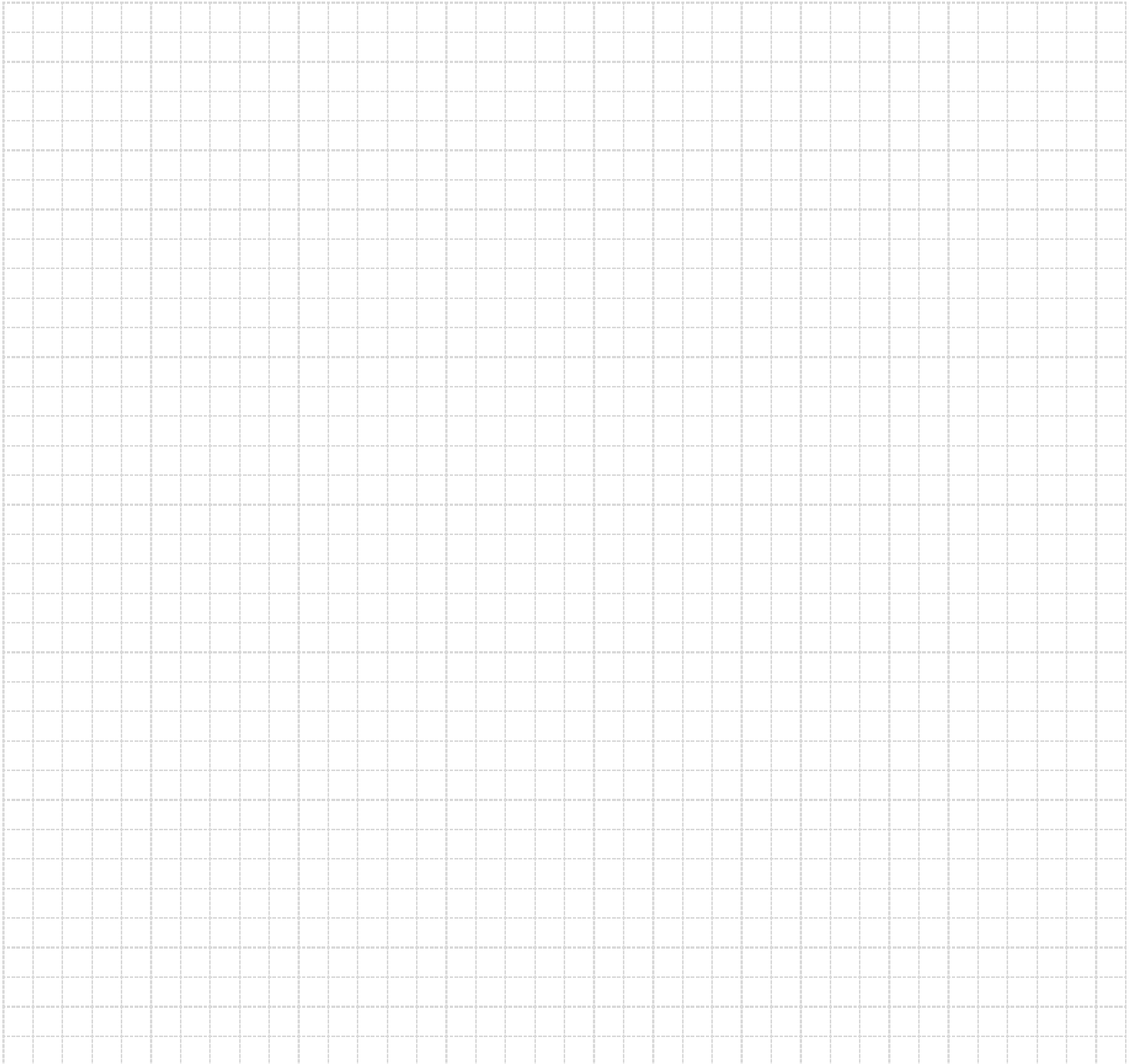
<b>Alloy Series</b>	<b>Principal Alloying Element</b>
1xxx	Minimum 99.00% Aluminum
2xxx	Copper
3xxx	Manganese
4xxx	Silicon
5xxx	Magnesium
6xxx	Magnesium and Silicon
7xxx	Zinc
8xxx	Other Elements

#### As Cast

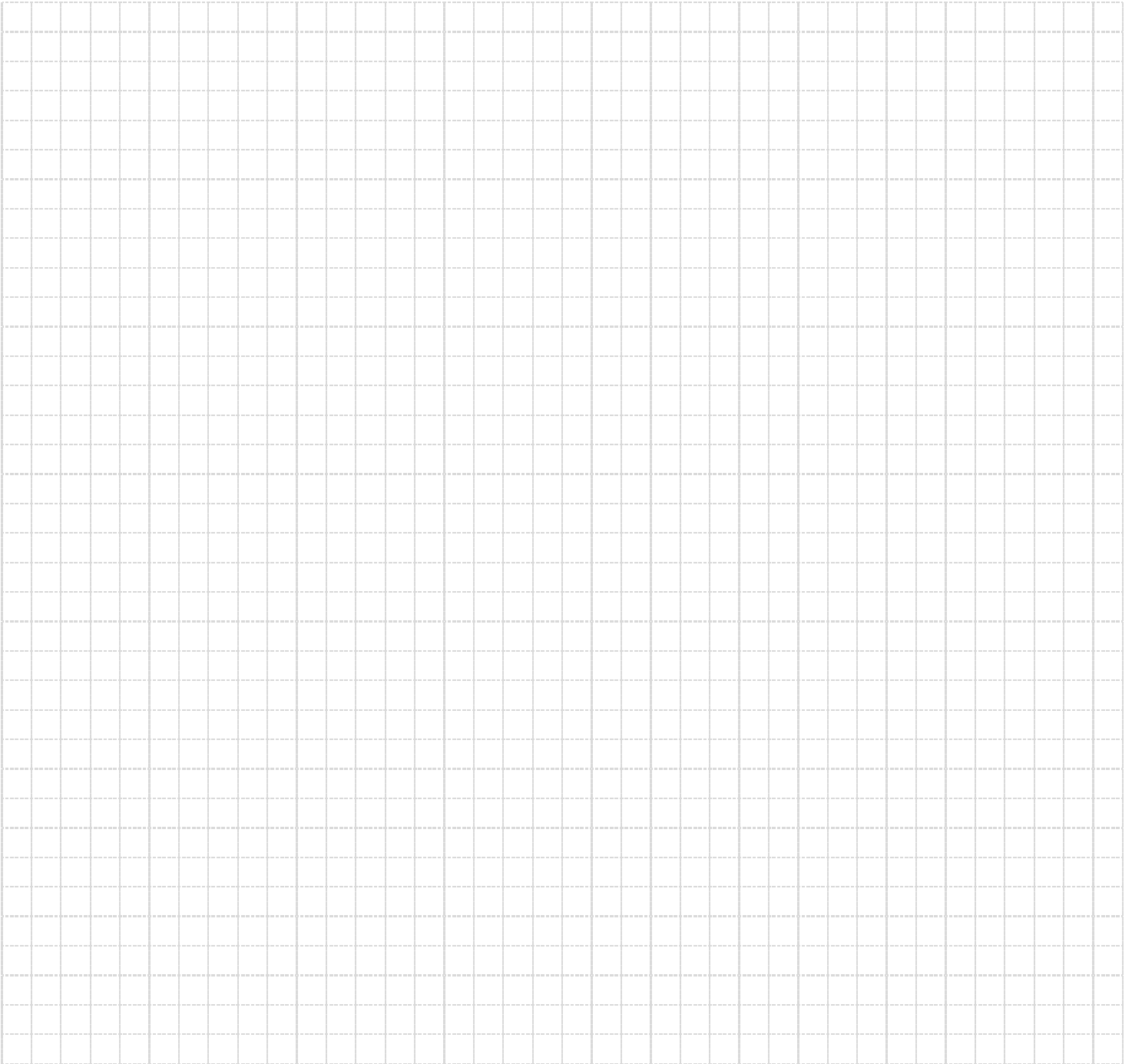
<b>Alloy Series</b>	<b>Principal Alloying Element</b>
1xx.x	Aluminum, 99.00% or greater
2xx.x	Copper
3xx.x	Silicon with Copper and/or Magnesium
4xx.x	Silicon
5xx.x	Magnesium
6xx.x	Unused Series
7xx.x	Zinc
8xx.x	Tin
9xx.x	Other Elements

## Observations

### 1. Draw Al-Si Phase Diagram and identify the phases

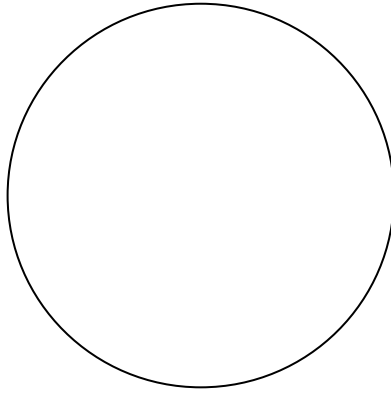


**2. Draw Al-Cu Phase Diagram and identify the phases**



**Sample No:**

*Draw the microstructure in the circle given below and write down the phases*



Magnification used \_\_\_\_\_

The phases present in the microstructure and the approximate % of major phases are

	Phases Present	Percentage
➤	-----	-----
➤	-----	-----
➤	-----	-----
➤	-----	-----

Etchant used \_\_\_\_\_

From the observation of microstructure, the given sample is .....

Any other observation .....

## **Answer the Following Questions**

1. What are the outstanding properties of aluminium?
2. Explain the meaning of the digits in the following aluminium specifications:  
2107-T4, 7075-T6, 6061-0
3. Why do aluminium alloy 2017 rivets have to be refrigerated until used?
4. What properties do Al-Si alloys have? Give some typical applications
5. What properties do Al-Cu alloys have? Give some typical applications



## EXPERIMENT 7

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### ASTM GRAIN SIZE ANALYSIS

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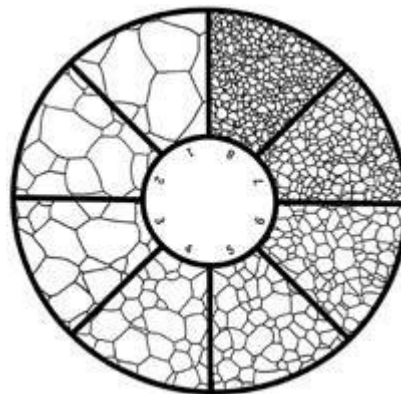
#### Objective

To find out the grain size in single and multi-phase alloy systems.

#### Theory

For the grain size determination, the commonly used methods are:

- i. Comparison Method
- ii. Grain Counting Method
- iii. Intercept Method



#### Grain Size Determination

The microstructural quantity known as the ASTM Micro Grain Size Number,  $n$ , is defined by the following relationship

$$N = 2^{n-1}$$

Where  $N$  is the number of grains per square inch, measured at a magnification of 100x. The most common methods for estimating the value of  $n$  are:

- **Comparison methods:** The overall appearance is simply compared with a standard set of micrographs or “plates” for which the ASTM Grain Size Number has been determined.
- **Grain counting methods:** The number of grains per unit area is counted directly. The ASTM grain size number is then determined according to the definition.
- **Intercept methods:** The number of grain boundary intercepts per unit test is measured. This is a measure of grain boundary area per unit volume and is, therefore, related to the grain size.

The methods for grain size measurement are described in great detail in the ASTM Standard, E112, “Standard Test Methods for Determining Average Grain Size.” The information below is intended to provide a cursory explanation of the three categories listed above.

#### 1. Comparison Method

This is the simplest, yet least quantitative, method, and is described in section 8 of ASTM E112. Because the comparison of grain structures may be influenced by the overall type of microstructure, four standard categories of grain size plates are used for comparison.

-Plate 1: Untwined grains, flat-etch at 100x.



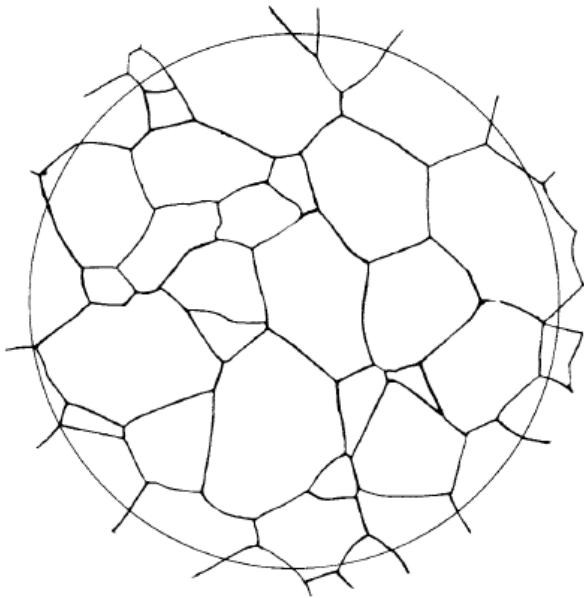


FIG. 1 Example of Untwinned Grains (Flat Etch) from Plate I.  
Grain Size No. 3 at 100X

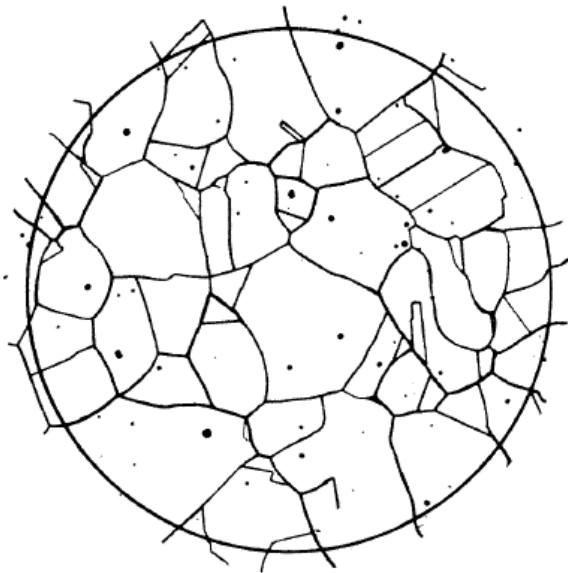


FIG. 2 Example of Twin Grains (Flat Etch) from Plate II. Grain  
Size No. 3 at 100X



FIG. 3 Example of Twin Grains (Contrast Etch) from Plate III.  
Grain Size 0.090 mm at 75X

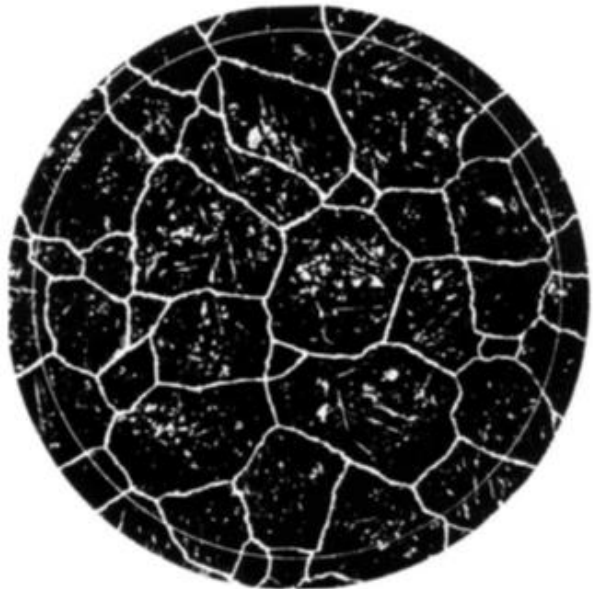


FIG.4 Example of Austenite Grains in Steel from Plate IV.  
Grain Size No. 3 at 100X

Include grain size numbers 00, 0, 0.5, 1, 1.5, 2, 2.5, 3, 3.5, 4, 4.5, 5, 5.5, 6, 6.5, 7, 7.5, 8, 8.5, 9, 9.5, and 10.

Plate 2: Twinned grains, flat-etch at 100x.

Includes grain size numbers 1, 2, 3, 4, 5, 6, 7, and 8.

Plate 3: Twinned grains, contrast-etch at 75x.

Includes nominal grain diameters of 0.2, 0.15, 0.12, 0.09, 0.07, 0.06, 0.05, 0.045, 0.035, 0.025, 0.020, 0.015, 0.010, and 0.005mm.

Plate 4: Austenite grains in steel at 100x.

Includes grain size numbers 1, 2, 3, 4, 5, 6, 7, and 8.

These four plates are shown in the attached Figs.1-4, taken from ASTM E112. For any of these categories, a collection of plates, showing microstructures of the listed grain size numbers would be used for comparison.

## 2. Grain Counting Methods

An example of a grain counting method is the Plan Metric Procedure, also known as Jeffries Method. This method is described in Section 9 of ASTM E112. The basic steps of the procedure are as follows:

- (a) Inscribe a circle (or other shape) of known area,  $A$ , on an image of magnification,  $M$ .
- (b) Count the number of grains that are completely within area.
- (c) Count the number of grains that are partially within the area.
- (d) Divide the result from (c) by 2.
- (e) Add the results from (d) to the result from (b).
- (f) Divide the result from (e) by  $A$ .
- (g) Convert the result from (f) to grain/in<sup>2</sup> @ 100x.
- (h) Use the definition of ASTM grain size number to determine  $n$ .

The method can be used in every case. The only step that may be confusing is (g), where the magnification must be accounted for. The result from (f) will be given in grains per unit area, measured on the image of magnification,  $M$ . To convert this to the 100x equivalent, multiply the result by  $(M/100)^2$ .

ASTM E112 provides some tools for convenience. The standard contains a table that relates grains/in<sup>2</sup> @ 100x and grains/mm<sup>2</sup> @ 1x to ASTM grain size number. To further assist the user, the standard suggests that an area of 5000 mm<sup>2</sup> be used for the grain count, so long as this area encompasses at least 50 grains. The standard also includes a table that provides a “Jeffries’ Multiplier” corresponding to several magnifications. If  $A=5000$  mm<sup>2</sup>, the multiplier indicated by this table can be multiplied by the result from step (f) to yield the grain count in terms of grains/mm<sup>2</sup> @ 1x. Table 2 can then be used to determine the ASTM grain size.

Magnification Used, X	Jeffries Multiplier, k, to obtain Grains/mm <sup>2</sup>
1	0.0002
10	0.02
25	0.125
50	0.5
75	1.125
100	2.0
150	4.5
200	8.0
250	12.5
300	18.0
500	50.0
750	112.5
1000	200.0

### 3. Intercept Methods

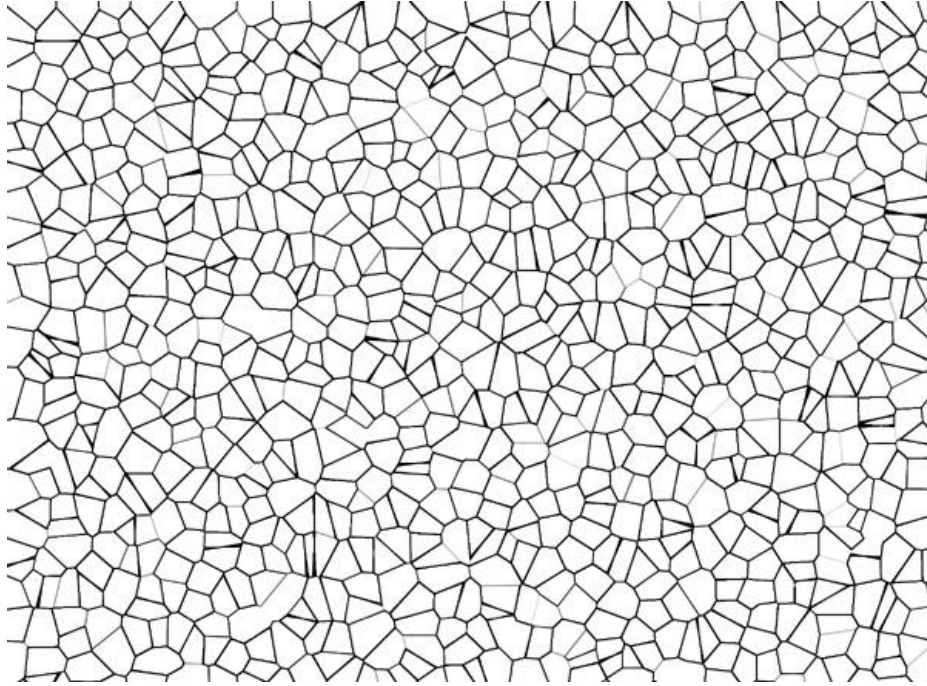
An example of an intercept method in the Lineal Intercept Procedure, also known as the Heyn Method. This is described in detail in Section 11 of the ASTM E112 Standard. The basic steps to this procedure are given here:

- (a) On a single field of view, randomly place one or more straight test lines of known combined total length, L.
- (b) Count the total number of intercepts, P, between the test lines(s) and the grain boundaries. Triple junctions count as 1.5. if P<50, use additional lines.
- (c) Divide the number of intercepts, P, obtained from (b) by the total length, L.
- (d) Repeat (a-c) for 2-4 additional fields of view.
- (e) Obtain  $P_L$  as the average of the result from (c) for all fields of view.
- (f) The ASTM Grain Size Number is given as

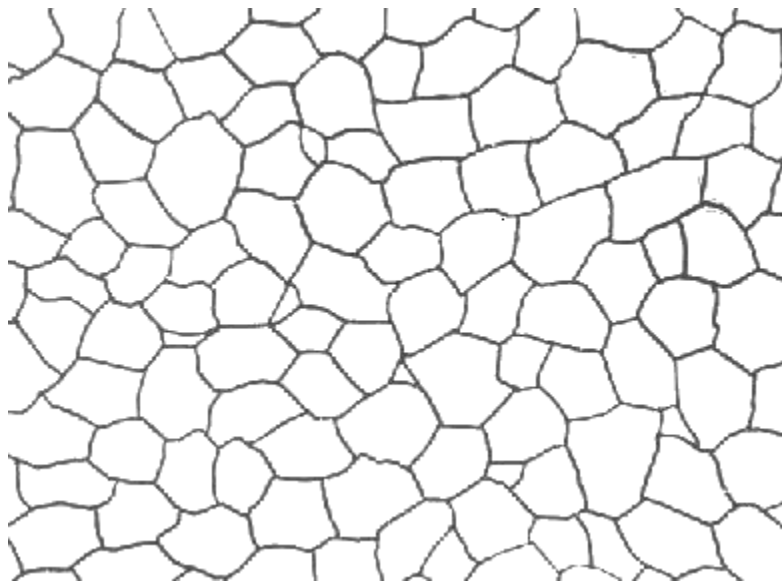
$$n = -3.3 + 6.65 \log_{10}(P_L) \quad \text{where } P_L \text{ is given in } \text{mm}^{-1}.$$

## OBSERVATIONS

1. The microstructure obtained at 200X magnification is given below. Determine the ASTM grain size of by grain counting method.



2. The microstructure obtained at 200X magnification is given below. Determine the ASTM grain size of by Heyn intercept method.







8. Calculate the average area of a grain if the steel has ASTM grain size number 7?  
Calculate in  $\text{inch}^2$  as well as in  $\text{mm}^2$ .

9. Are grain boundaries desirable for high temperature structural application? Give reasons for your answer.

10. What is the significance of this experiment? How is it related to your course of study?