METALLOGRAPHY II LABORATORY

LAB MANUAL

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FOREWORD

This lab manual is designed to suit the needs of the students pursuing diploma in metallurgy engineering and acquainting them with the principles of metallography. The metallographic techniques are stressed, as these practices are essential for a metallurgist. In this lab manual 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 manual. It is important that all the information necessary to complete the lab report is obtained before students leave the lab.

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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 during an emergency
2. Know the location of the fire extinguisher, eye wash, and sa know how to use them.
3. Notify your instructor immediately after any injury, fire or explo
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.

ical safety

1. Treat every chemical as if it were hazardous.
2. Make sure all chemicals are clearly and currently labeled 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.)

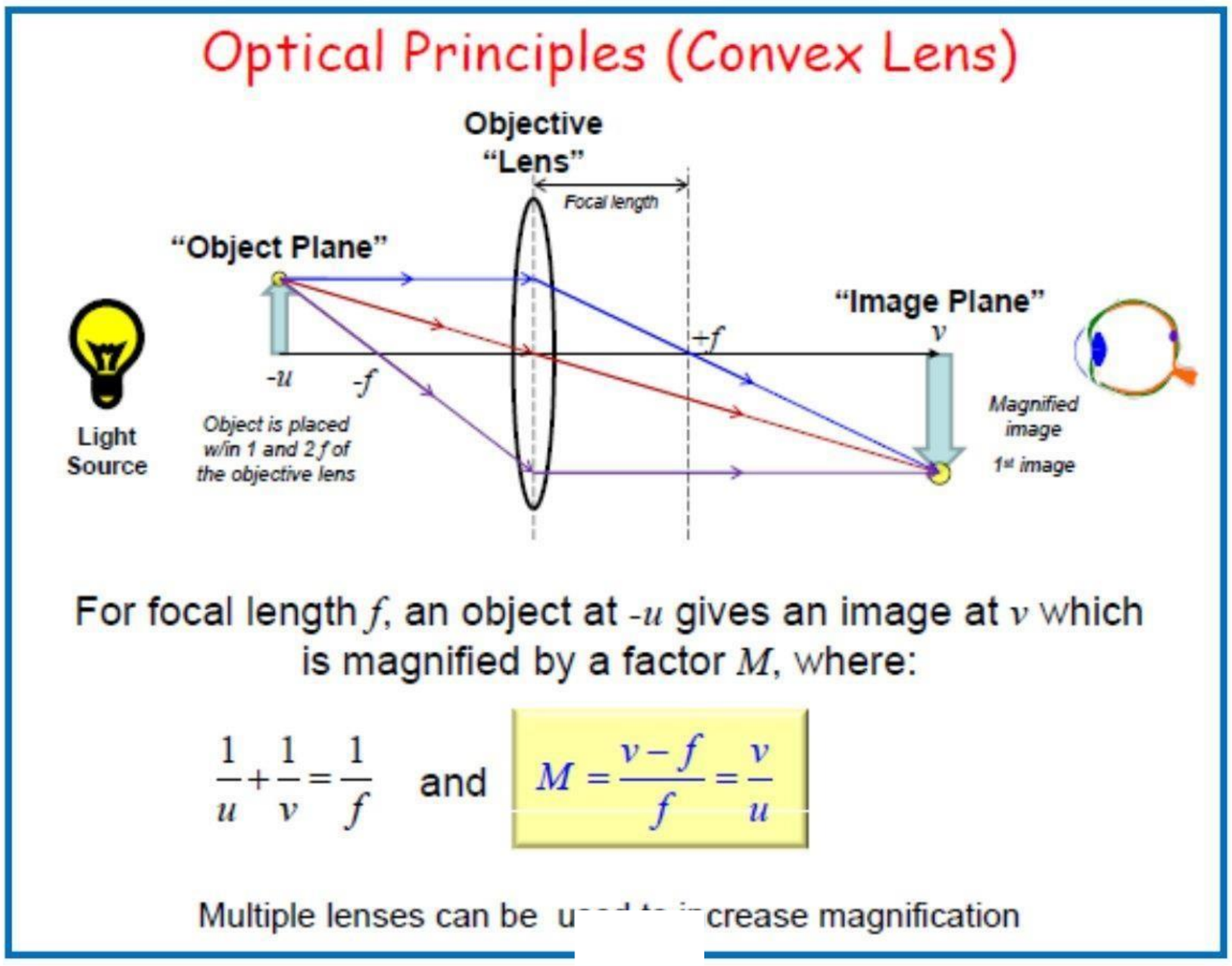
METALLURGICAL MICROSCOPE

Objective: To study the components and functions Metallurgical Microscope.

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.

Upright Metallurgical Microscope

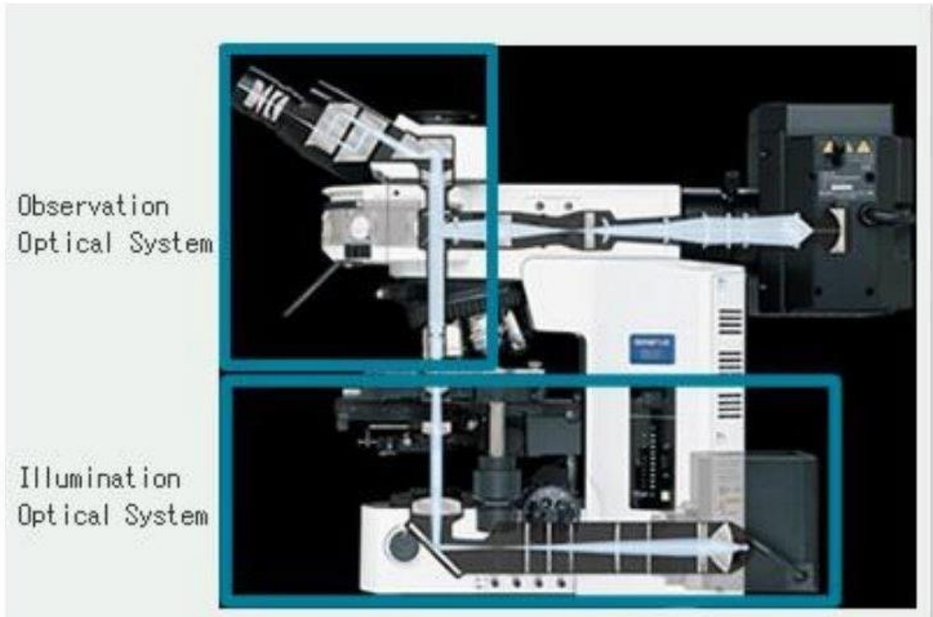
Inverted Metallurgical Microscope



Microscope Basic Functions

An optical microscope consists of the following two major basic functions.

- Creating a Magnified Image of a Specimen
- Illuminating a Specimen



Microscope Parts and Functions

1. Eyepiece: 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. Eyepiece 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

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

Answer the following Questions

1. Briefly describe the path of the light as it travels from the microscope's light source to your eye. What structures does it pass through?

2. What is the resolution of an optical microscope?

3. How is the magnification of a microscope defined?

4. What is the difference between Resolution and Magnification?

5. Define Numerical Aperture of a microscope objective. What are the advantages of a high NA objective? What advantages might a Low-NA objective have?

6. Define depth of field? How, one can improve depth of field?

7. What is the difference between depth of field and depth of focus?

8. How does phase-contrast microscopy differ from bright-field microscopy?

9. If a drawing is 80 mm and the actual size of the object is 20 μ m, what is the drawing magnification? For this drawing magnification, how long would a scale bar have to be to represent a distance of 10 μ m?

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

SPECIMEN PREPARATION FOR METALLOGRAPHIC ANALYSIS

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.

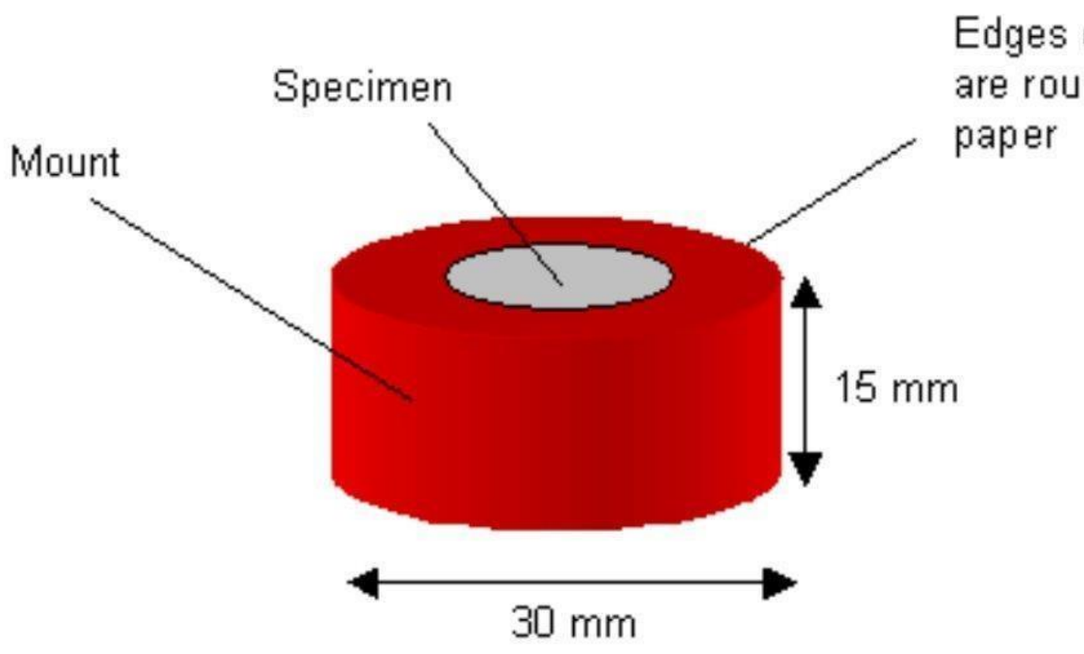
Cutting a specimen

It 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 electropolished (an Electrolytic process) or examined under a Scanning Electron Microscope , then the mounting material should also be electrically conducting.

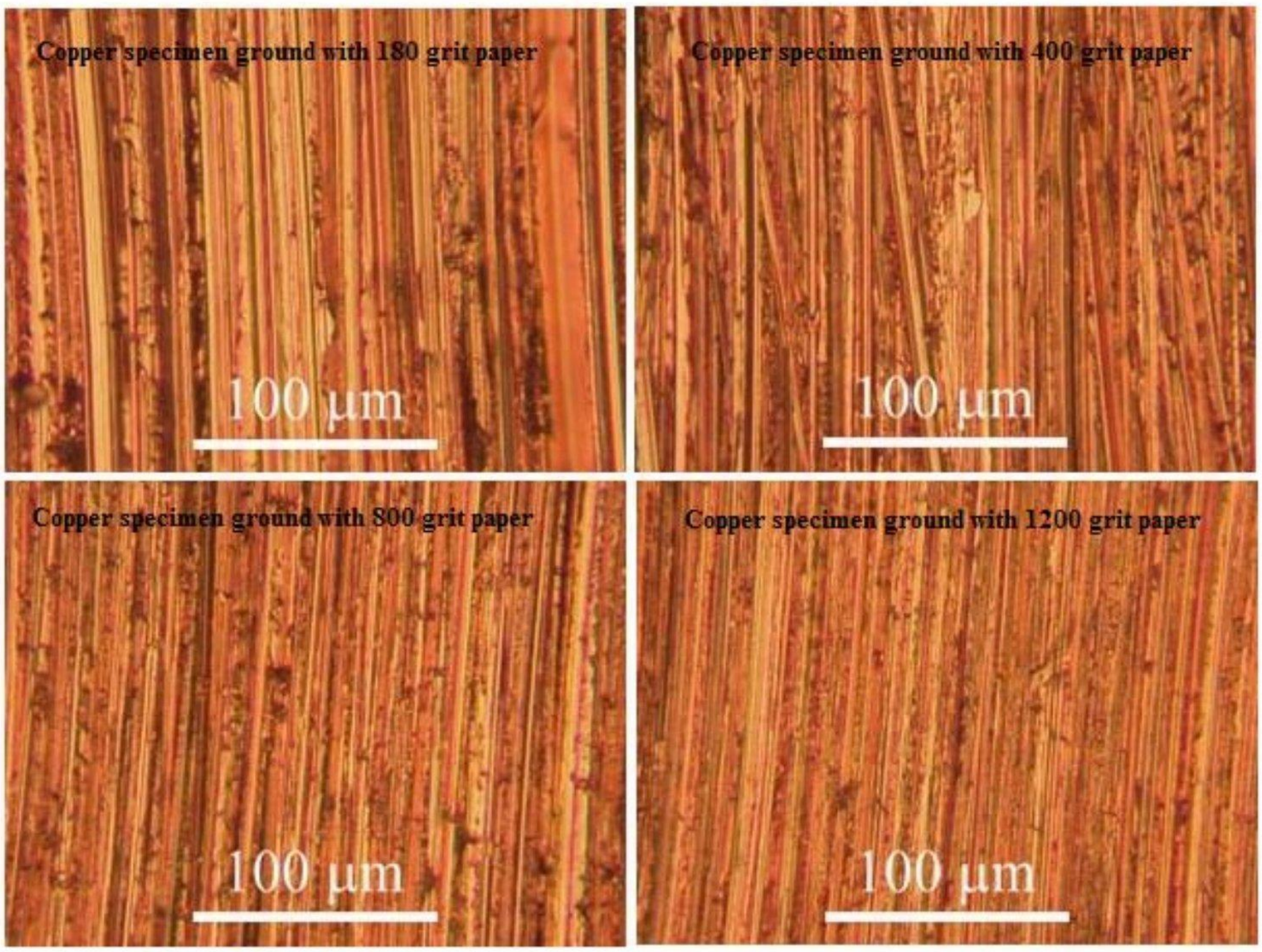


Specimens can be hot mounted (at around 200°C) using a mounting press, either in a thermosetting plastic (*e.g.* phenolic resin), or a thermosoftening plastic (*e.g.* acrylic resin). If hot mounting will alter the structure of the specimen a cold-setting resin can be used, *e.g.* epoxy,

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:

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

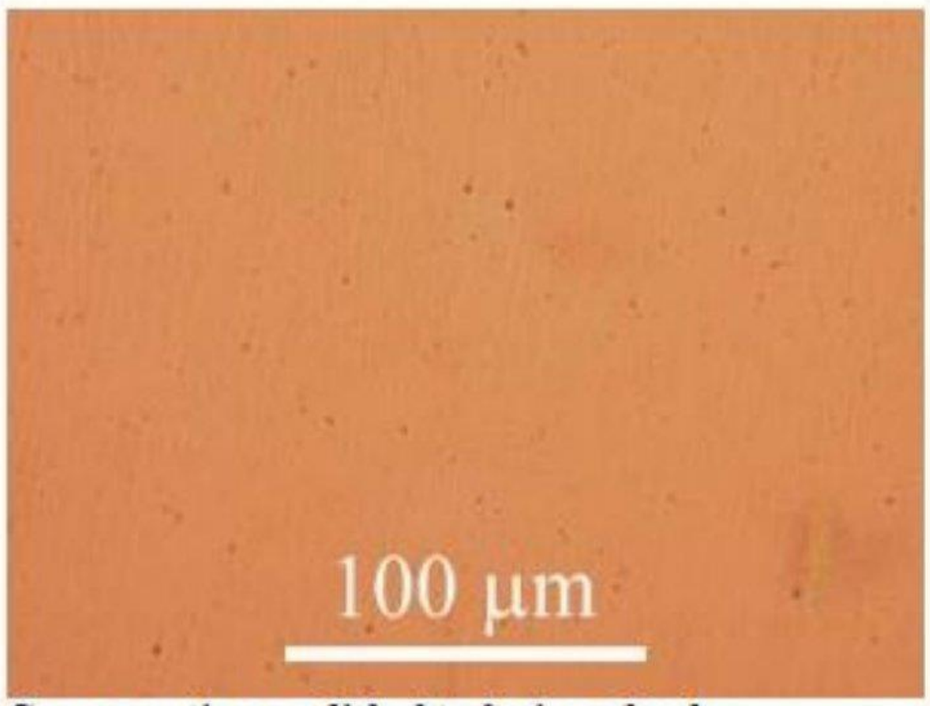
1. This prevents putting more damage into the sample than already exists; the coarsest grades of paper are often not useful.

2. Cleaning specimens in an ultrasonic bath can also be helpful, but is not essential.

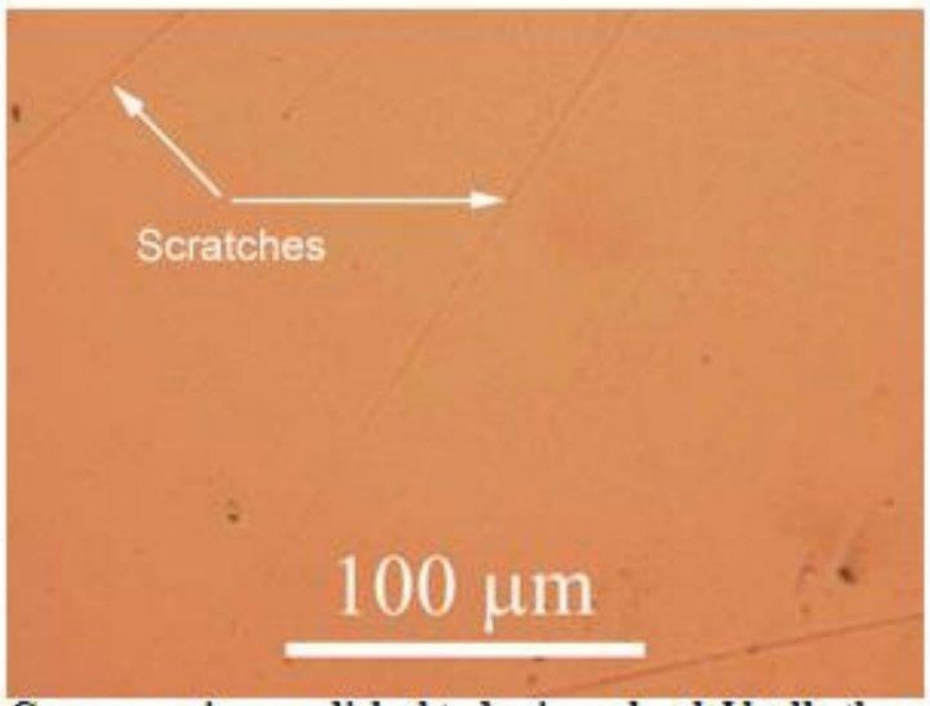
3. The series of photos below shows the progression of the specimen when ground with progressively finer paper.

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

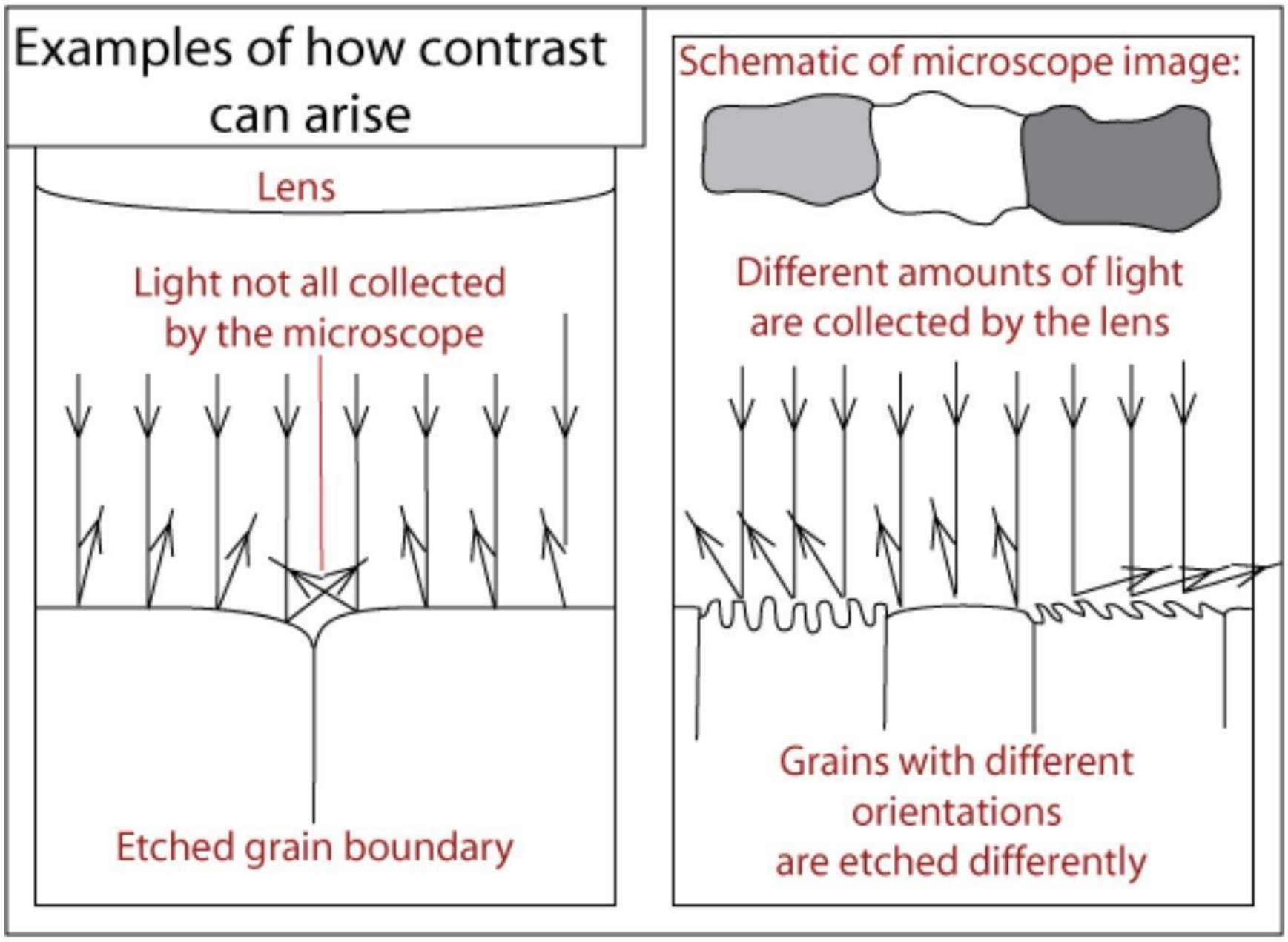
Mechanical polishing will always leave a layer of disturbed material on the surface of the specimen, if the specimen is particularly susceptible to mechanical damage (or excessive force is used in the grinding and polishing stages) debris can become embedded in the surface and plastic deformation may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

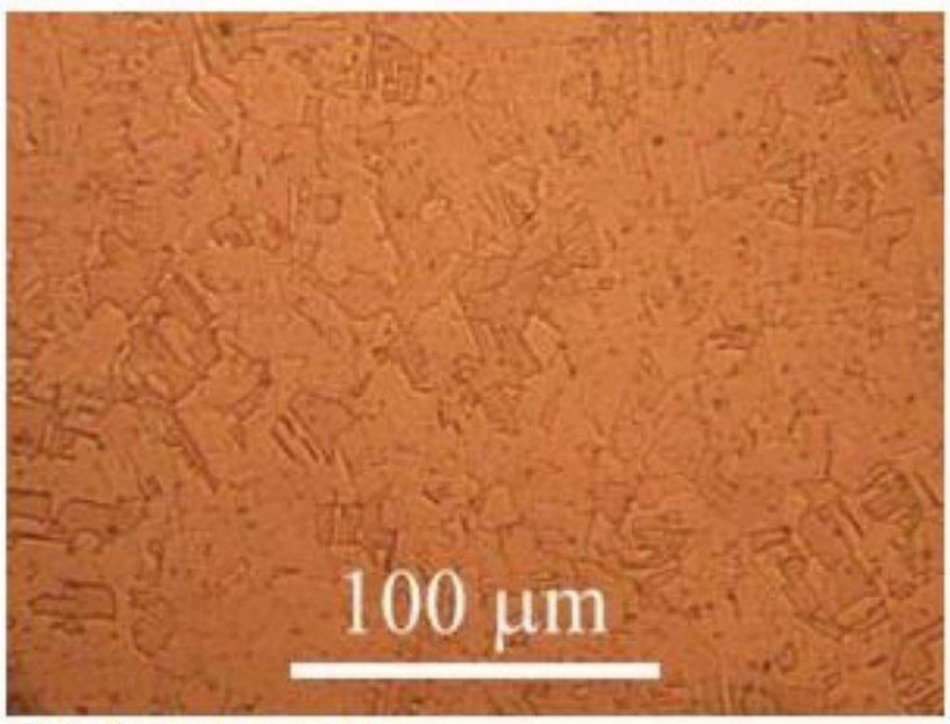
In all samples, etchants will preferentially attack high energy sites, such as boundaries and defects.



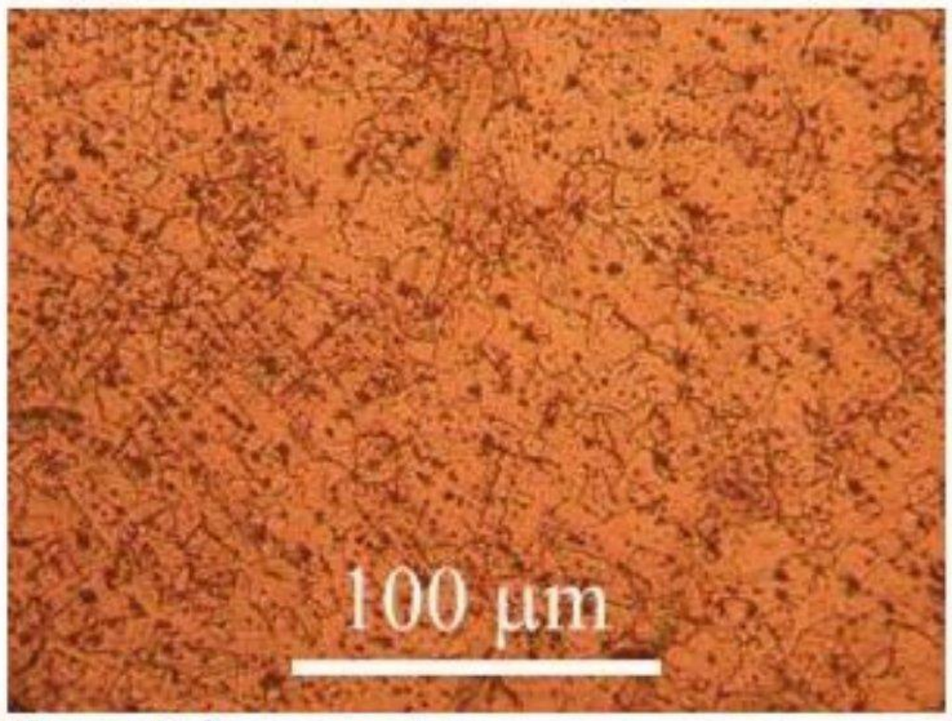
The specimen is etched using a reagent. For example, for etching stainless steel or copper and its alloys, a saturated aqueous solution of ferric chloride, containing a few drops of hydrochloric acid is used. This is applied using a cotton bud wiped over the surface a few times (Care should be taken 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.

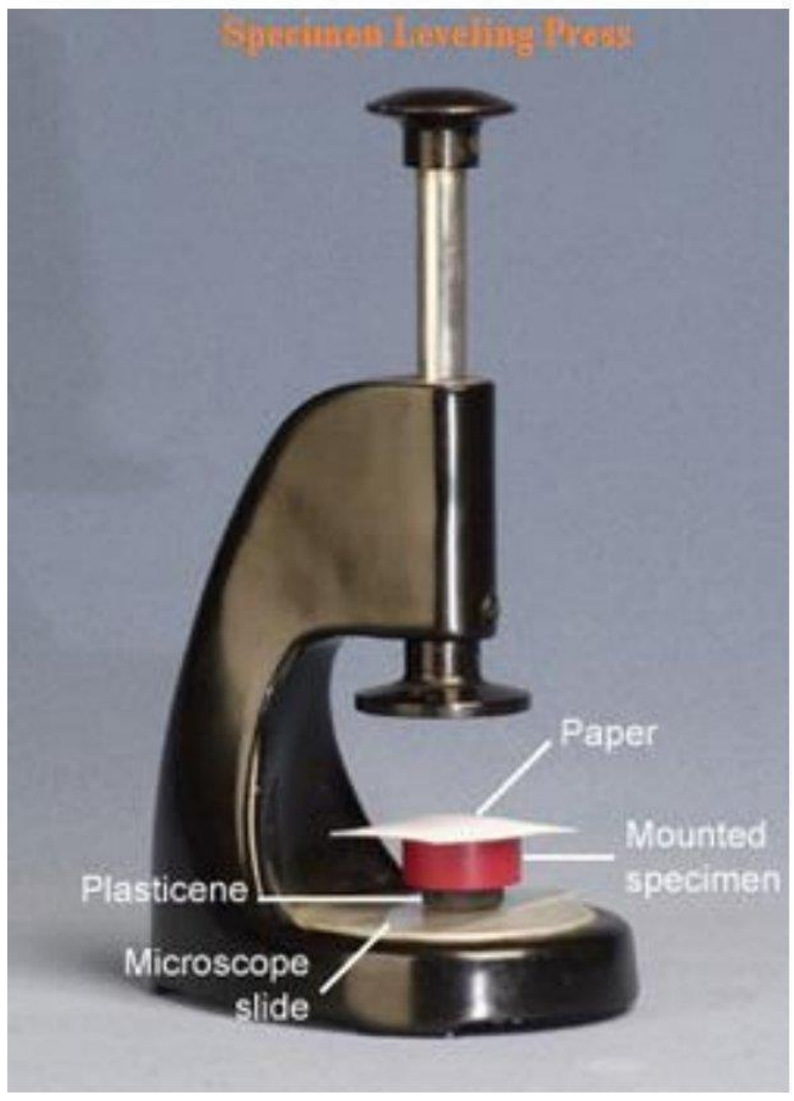


Etched copper specimen



Over etched copper specimen

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
Kalling's No. 1	Distilled water $CuCl_2$ 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.
Kalling's No. 2	$CuCl_2$ 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.
Kellers Etch	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.
Kroll's Reagent	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.
Nital	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.
Marble's Reagent	$CuSO_4$ 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_2SO_4 to increase activity.
Murakami's	$K_3Fe(CN)_6$ KOH Water	10 grams 10 grams 100 ml	Pre-mix KOH and water before adding $K_3Fe(CN)_6$	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.
Picral	Ethanol Picric acid	100 ml 2-4 grams	Seconds to minutes Do not let etchant crystallize or dry –**explosive**	Recommended for microstructures containing ferrite and carbide.
Vilella's Reagent	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

CERAMICS

Thin Sections

To prepare ceramic specimens for transmitted light microscopy, a thin slice, approximately 5 mm thick, is cut using a diamond saw or cutting wheel. One surface is then lapped using liquid suspensions of successively finer silicon carbide powders. Between stages in the process the specimen must be thoroughly cleaned. After final washing and drying the ground surface is bonded to a microscope slide with resin. A cut off saw is used on the exposed face to reduce the thickness to about 0.7 mm. The specimen is then lapped to take it to the required thickness – usually about 30 μm, although some ceramic specimens are thinned to as little as 10 μm, due to their finer grain size. The slide is checked for thickness under the microscope, and then hand finished. The slide is then covered with a protective cover slip.

Lapping

The lapping process is an alternative to grinding, in which the abrasive particles are not firmly fixed to paper. Instead a paste and lubricant is applied to the surface of a disc. Surface roughness from coarser preparation steps is removed by the micro-impact of rolling abrasive particles.

Polished sections

These differ from ordinary thin sections in that the upper surface of the specimen is not covered with a cover slip, but is polished. Care must be taken to prevent the specimen breaking. Sections may be examined using both transmitted and reflected light microscopy, which is particularly useful if some constituents are opaque.

POLYMERS

Thin sections

Thin sections of organic polymers are prepared from solid material by cutting slices using a microtome – a mechanical instrument used for cutting thin sections. They must be cut at a temperature below the glass transition temperature of the polymer. A cut section curls up during cutting and must be unrolled and mounted on a microscope slide and covered with a cover slip. A few drops of mounting adhesive are used and must be compatible with the specimen. As always the mounting temperature must not affect the microstructure of the specimen.

The thickness of cut slices of polymer tends to lie in the range 2 to 30 μm depending on the type of material. Harder polymers can be prepared in the same way as thin ceramic specimens.

Polished sections

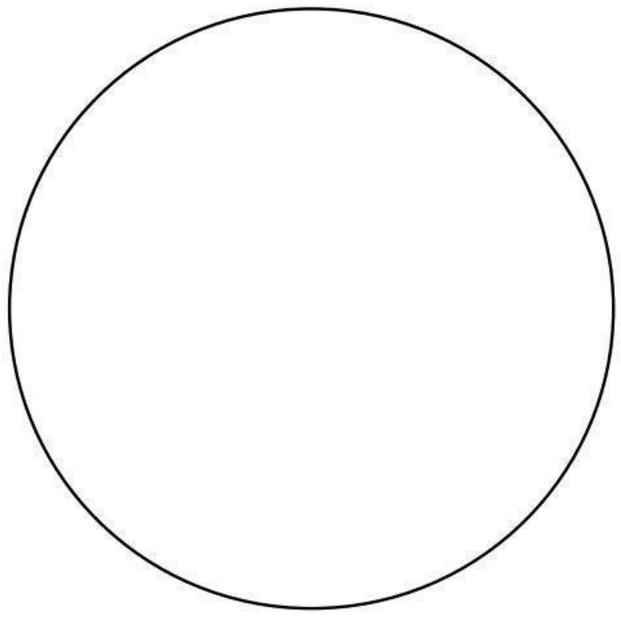
These are prepared in the same way as metallographic specimens. Elastomers are more difficult to polish than thermosetting polymers and require longer polishing times. Lubricants used during polishing must not be absorbed by the specimen.

As crystalline regions are attacked more slowly than amorphous ones, etching of polymer specimens can produce contrast revealing the polymer structure.

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?

6. Why metallographic samples are sometimes mounted in plastic?

7. Why microstructures are different for different metals?

8. What is emery/grit paper? What is the significance of 80,200,240,320...etc

9. State the different type of polishing clothes? On which basis can we select the polishing clothes?

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

48XX	Nickel-moly steels [illegible]
50XX	Low chromium steels
51XX	Medium chromium steels
52XX	Carbon-chromium steels
60XX	Chromium-vandium steels
70XX	Heat Resisting Casting Alloy
80XX	Ni-Cr-Moly steels
90XX	Silicon-Manganese Spring steels

Thus, AISI/SAE 1040 steel is plain carbon steel with a nominal carbon content of 0.40% C.

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. Etching reagents for plain carbon steels

Etchant	**Ingredients**	**Applications/remarks**
2% natal	2 ml nitric acid (conc.) 99 ml ethyl alcohol	Used to reveal ferrite grain boundaries
Marshall's reagent	Part A: 5 ml sulfuric acid (conc.) 8 gms oxalic acid 100 ml water Part B: 30% solution hydrogen Peroxide Part A can be mixed and stored, but do not store the mixture of parts A and B. Part B must be used fresh. Mix equal parts (A and B) just before using.	For ferrite grain boundaries (more uniform than nital). Colors cementite tan. Reveals prior-austenite grain boundaries in martensitic low-carbon steels

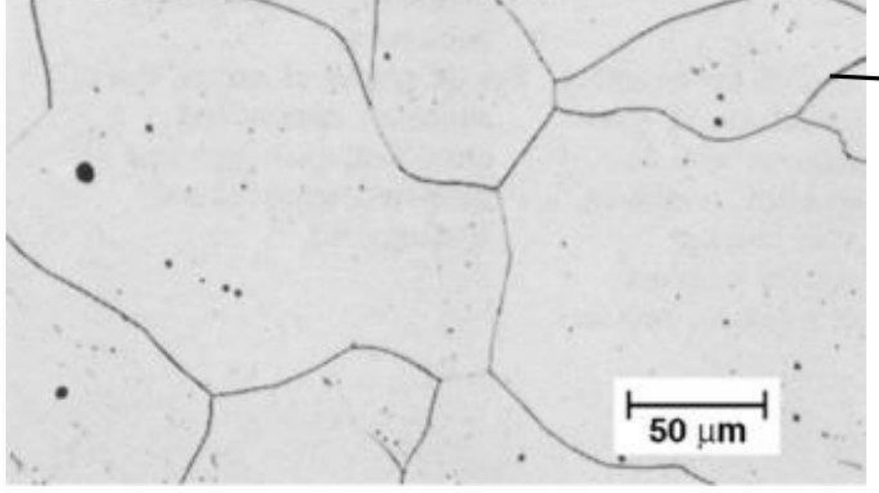


Fig.1: Microstructure of low carbon steel etched with nital

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

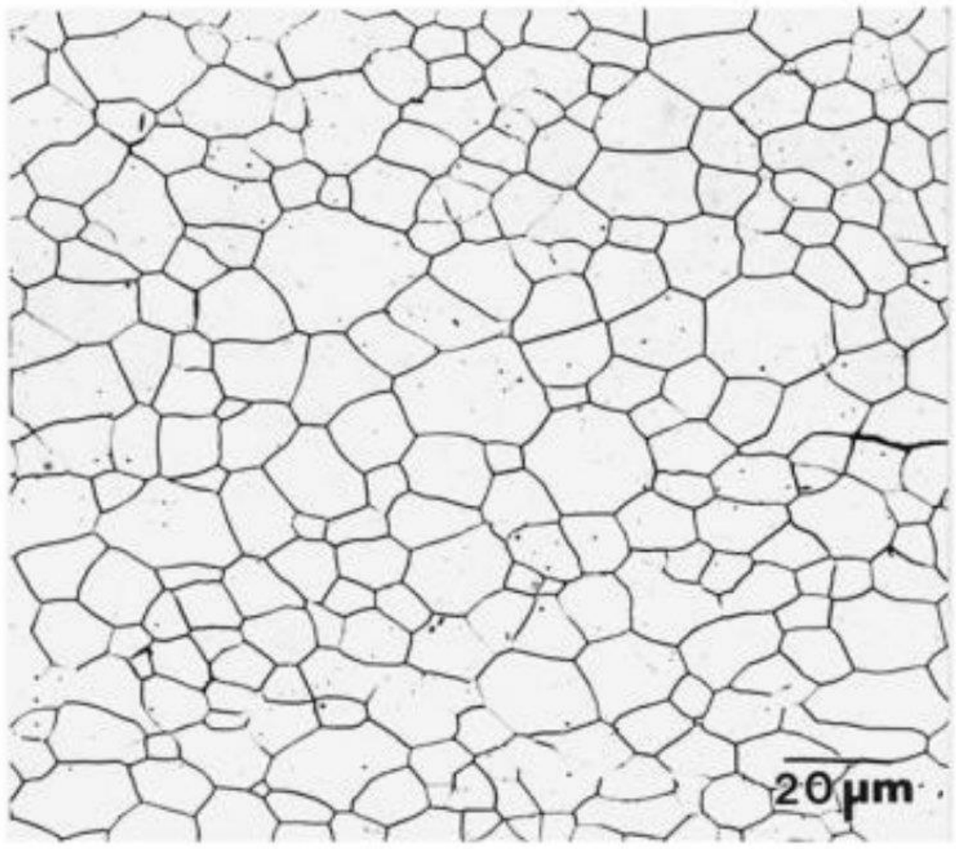


Fig. 2: Ferrite grains in a low-carbon (0.02% C) steel. Marshall's reagent.

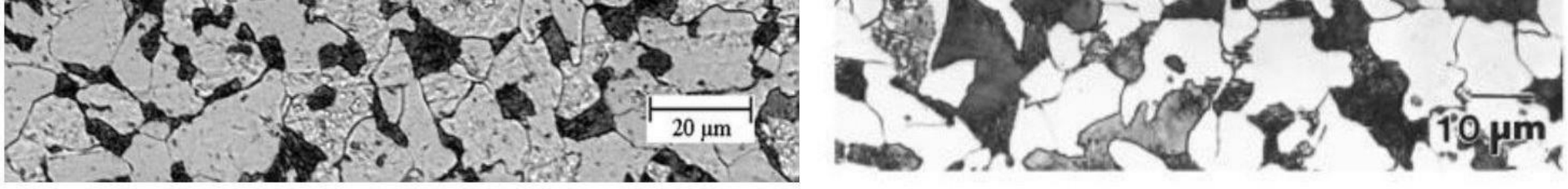


Figure 3. The microstructure of AISI 1040 steel showing ferrite and pearlite. The mean grain diameter is 26.173 µm. Etchant 2% Nital.

Microstructure of eutectoid steel containing 0.8% C

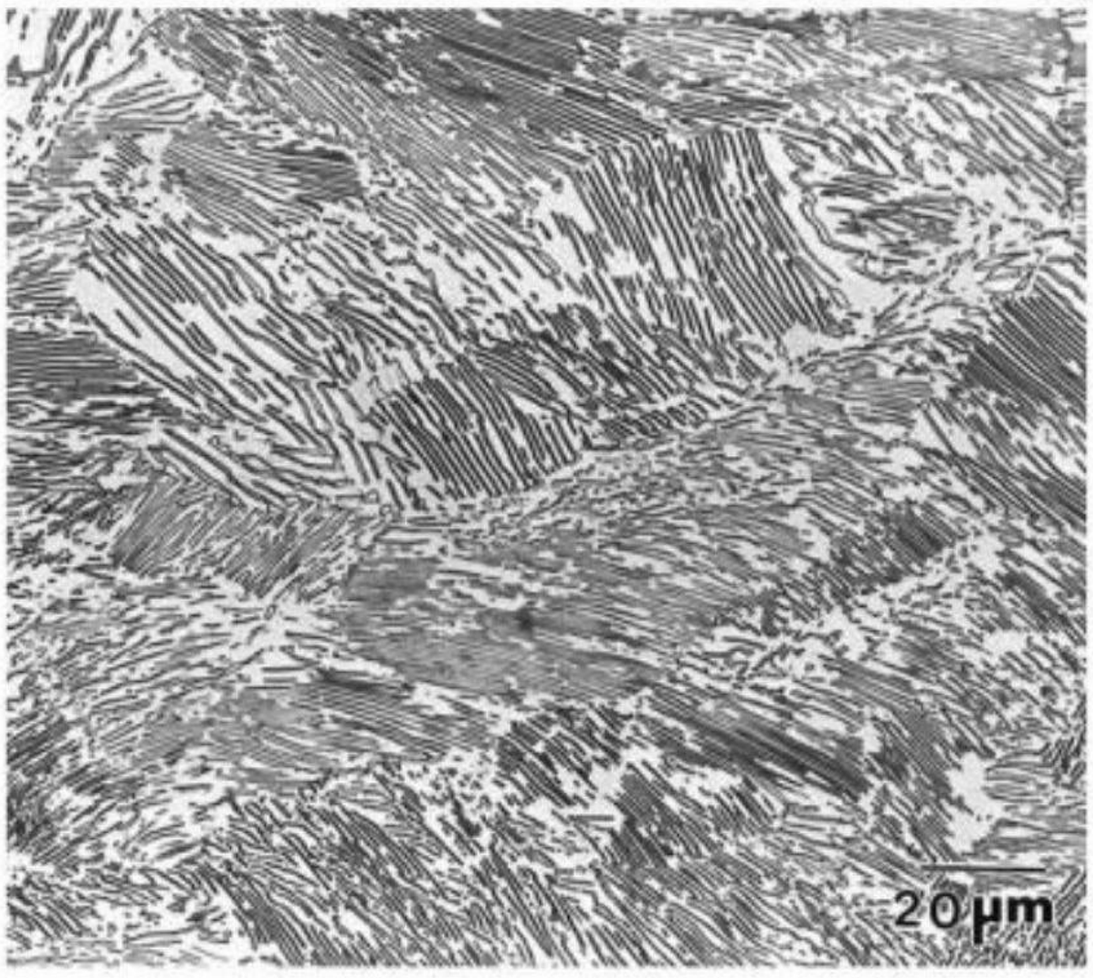


Figure 4. The microstructure of AISI/SAE 1080 steel showing pearlite etched with 4% Picral.

Hypereutectoid steel

Microstructure of High Carbon Steel containing >0.8% C

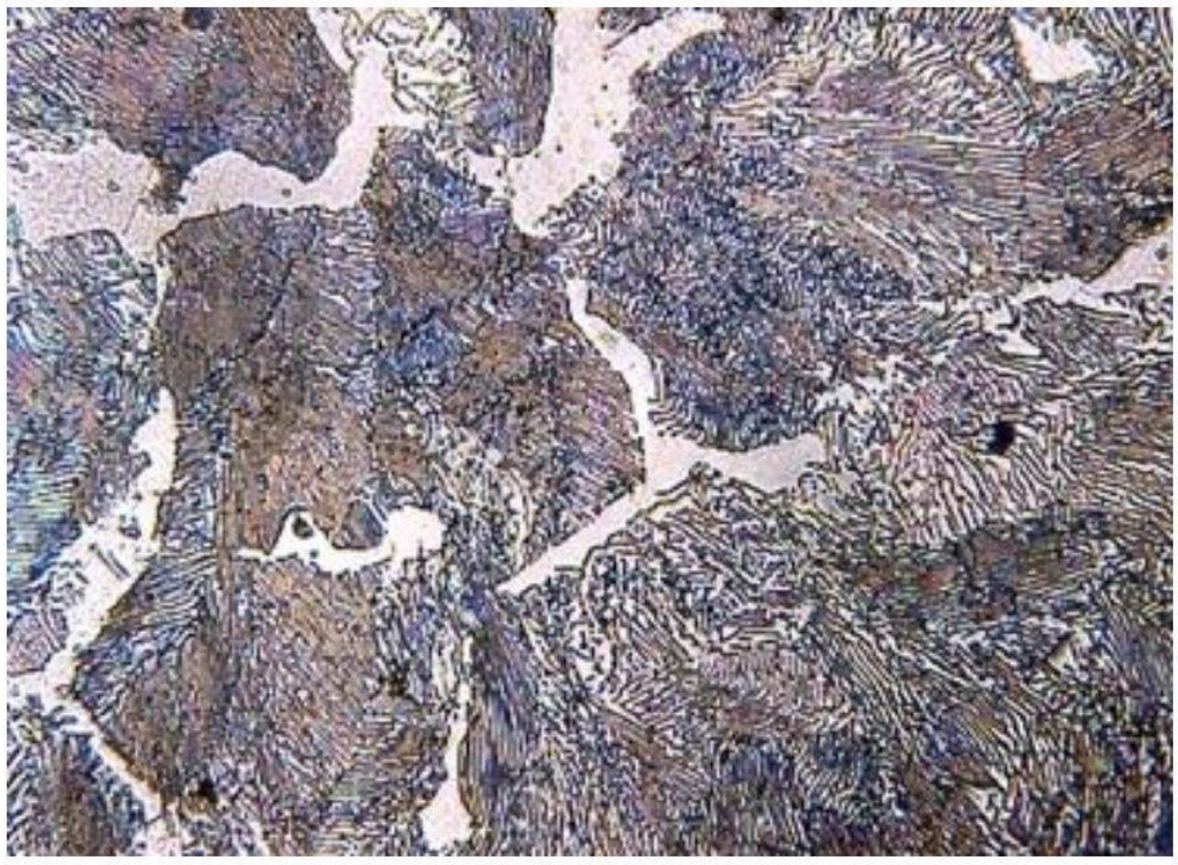


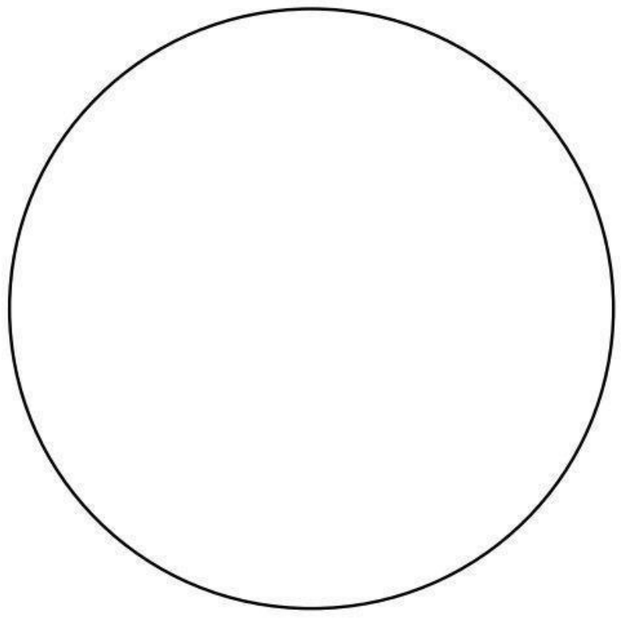
Figure 5: The microstructure of high carbon steel showing proeutectoid cementite around pearlite grains.

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 ……

……..

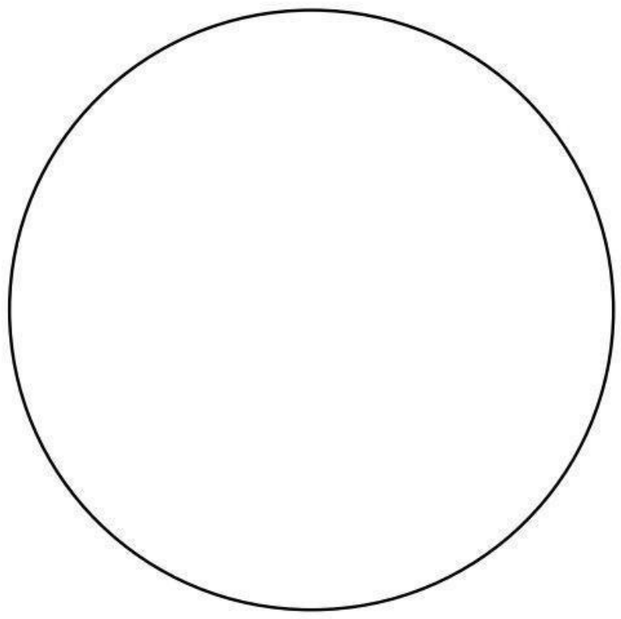
Any other observation ……

……....

.....

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 ……

……

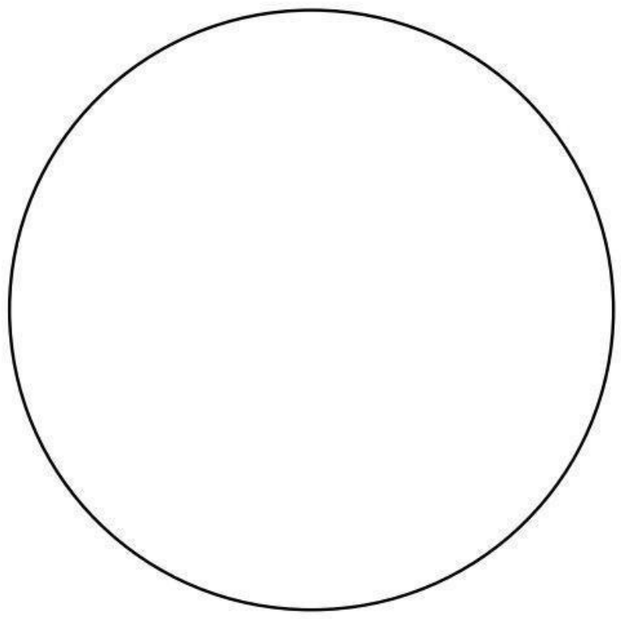
Any other observation ……

……

……

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 ……

…… ……

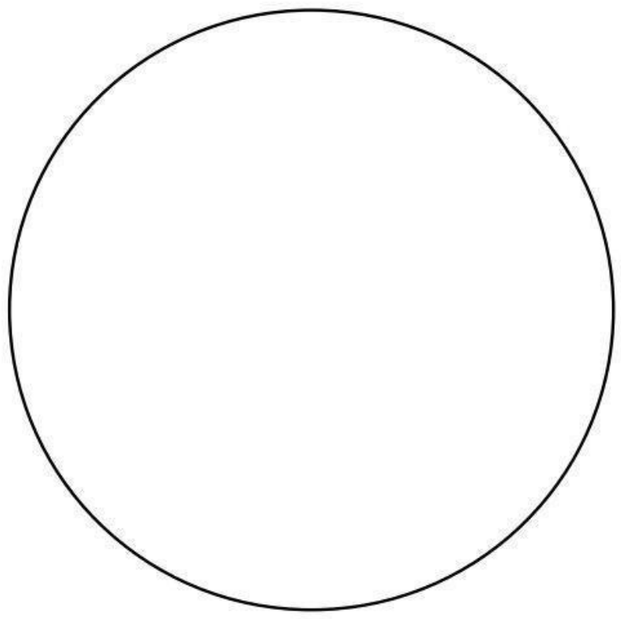
Any other observation ……

……..

……

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 ……

……

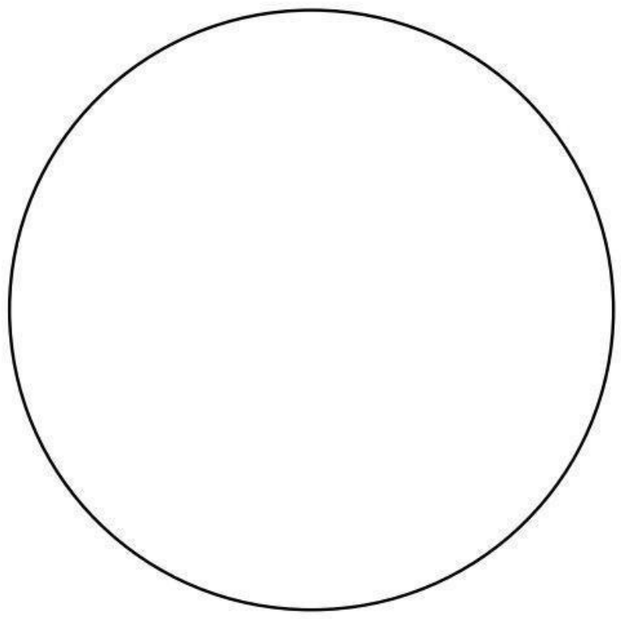
Any other observation ……

……

……

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 α, and ferrite α?

5. Define equilibrium diagram? Can we call Fe-Fe_3C is a equilibrium diagram? If yes/no state the reasons.

6. Cite three variables that determine the microstructure of the alloy?

7. What thermodynamic condition must be met for a state of equilibrium to exist?

8. How to differentiate between pearlite and graphite phases?

9. Which was the hardest and softest phase in Ferrite, Austenite, Cementite, Pearlite, ledeburite, graphite? Why?

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

MICROSTRUCTURE OF CAST IRON SAMPLES

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 engineering 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 gear box cases (declining usage). It is resistant to destruction and weakening by oxidisation (rust).

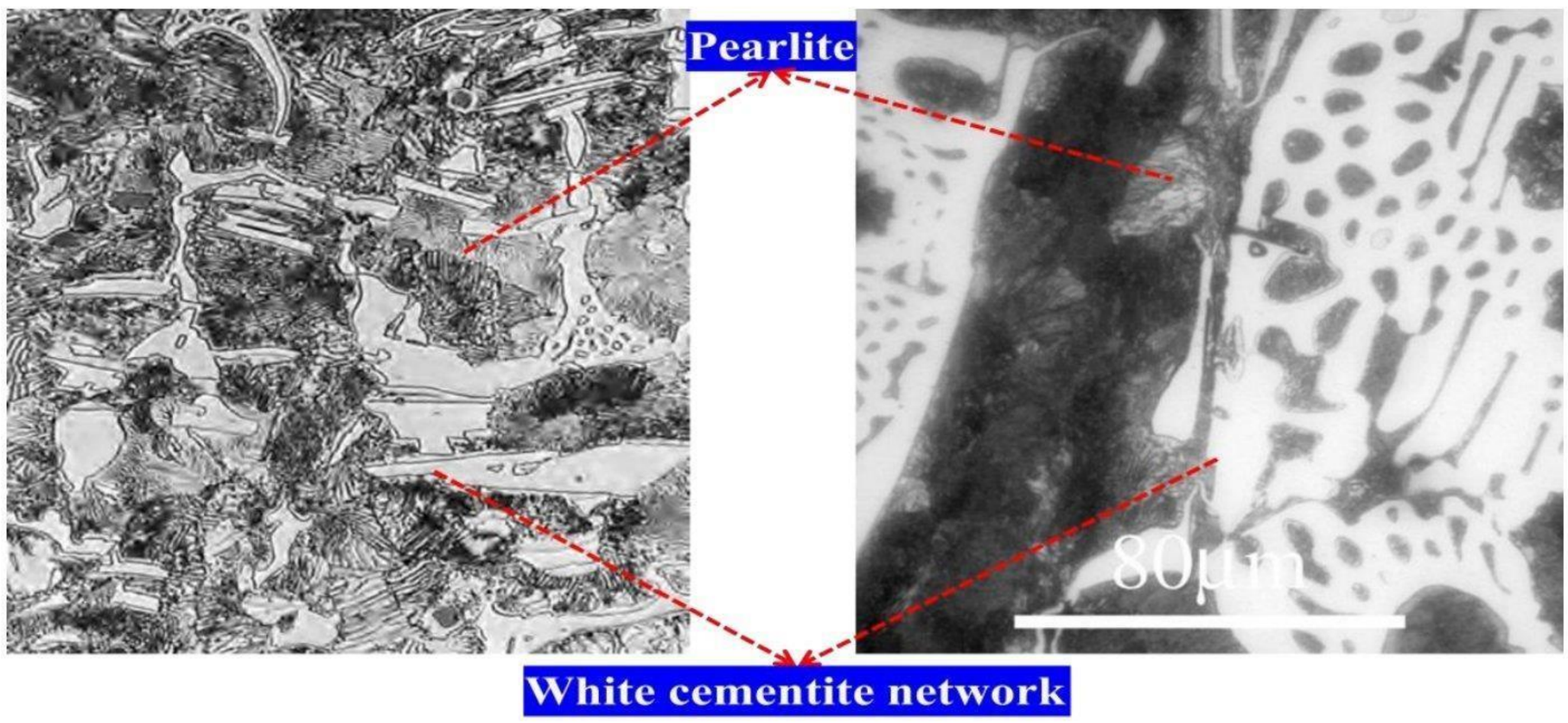
Types of cast iron

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

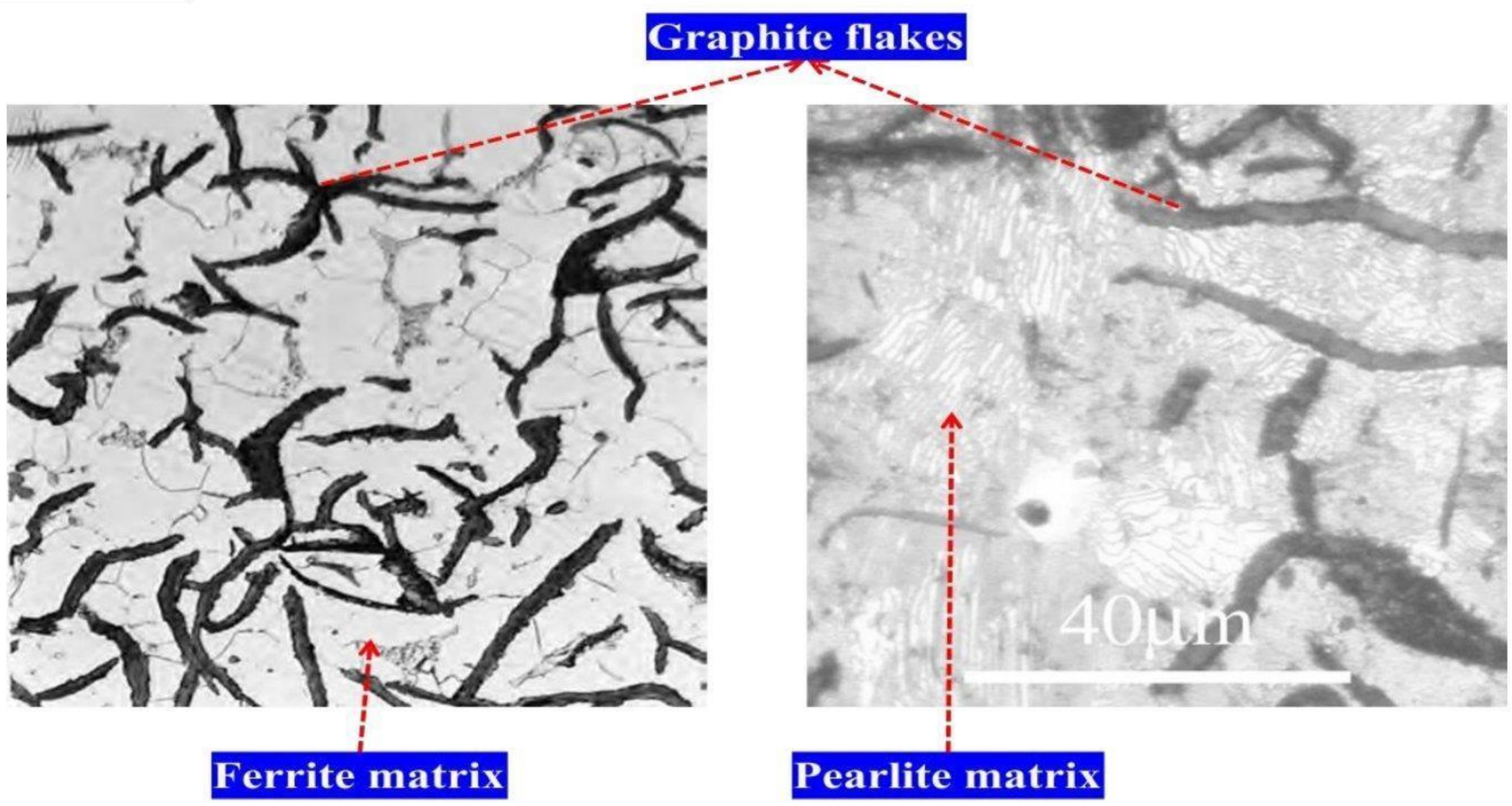
Table 1. Range of compositions for typical unalloyed Cast Irons

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

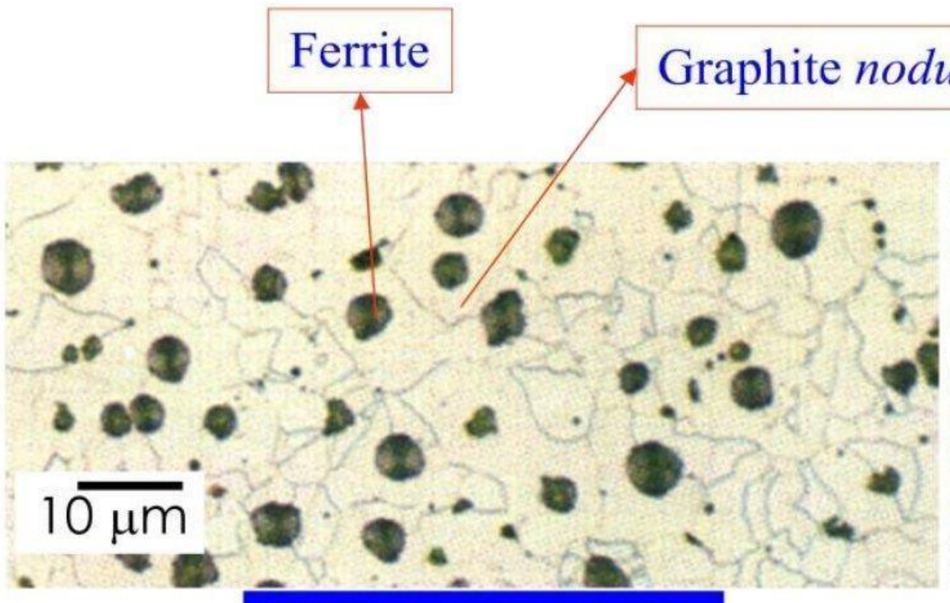
White Cast Iron



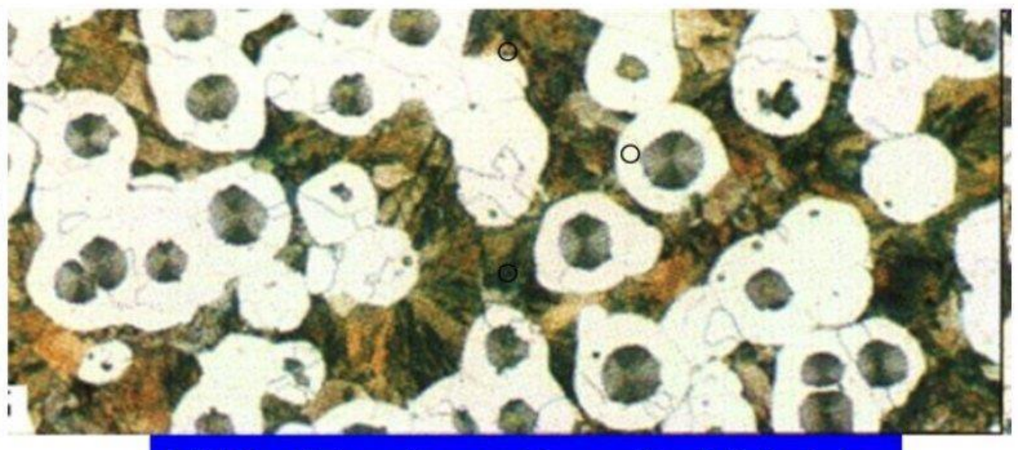
Grey Cast Iron



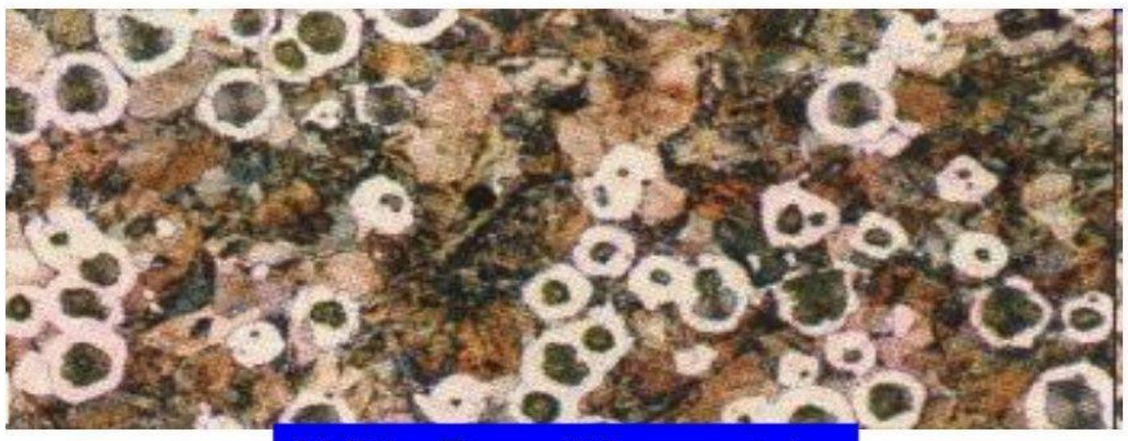
Ductile / Nodular cast iron (or) Spheroidal graphite (SG) iron



With Ferritic Matrix



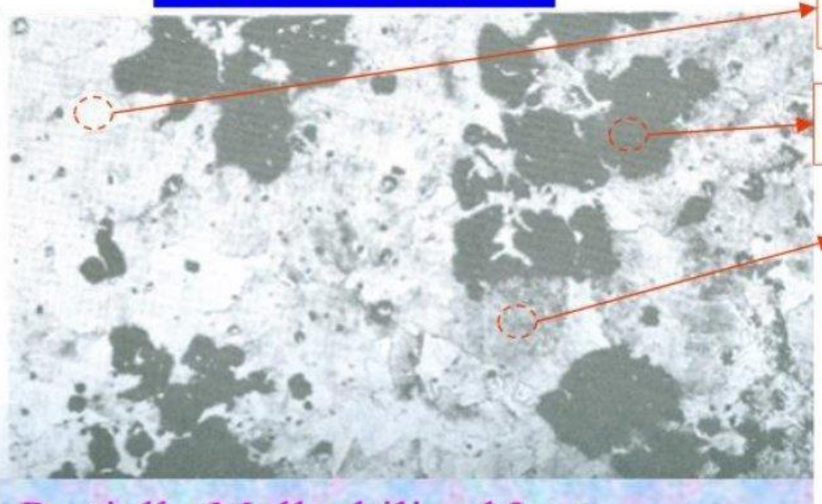
With (Ferrite + Pearlite) Matrix



With Pearlitic matrix

Malleable cast iron

Pearlitic Matrix



Ferrite *(White)*

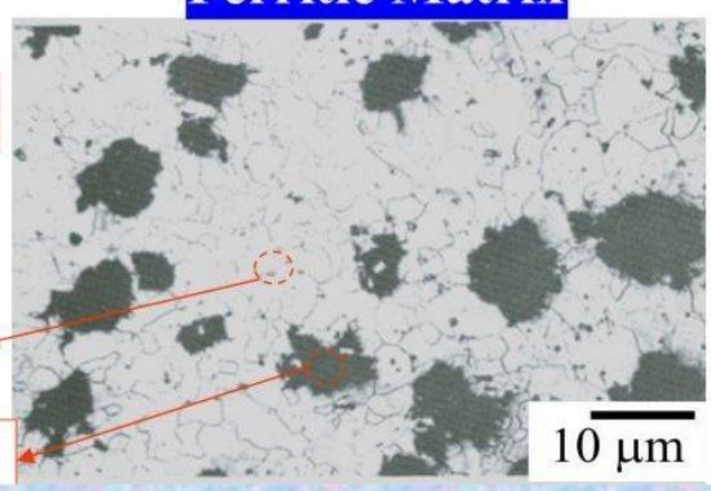
Graphite *(black)*

Pearlite *(grey)*

Ferrite *(White)*

Graphite *(black)*

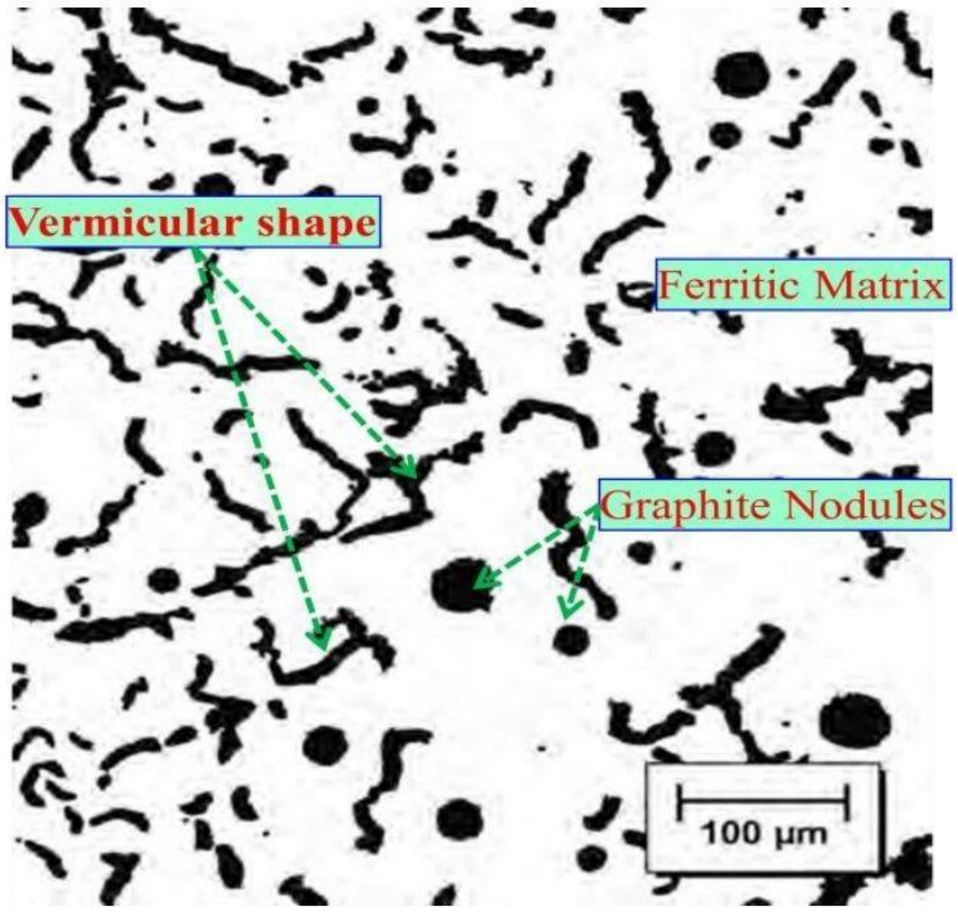
Ferritic Matrix

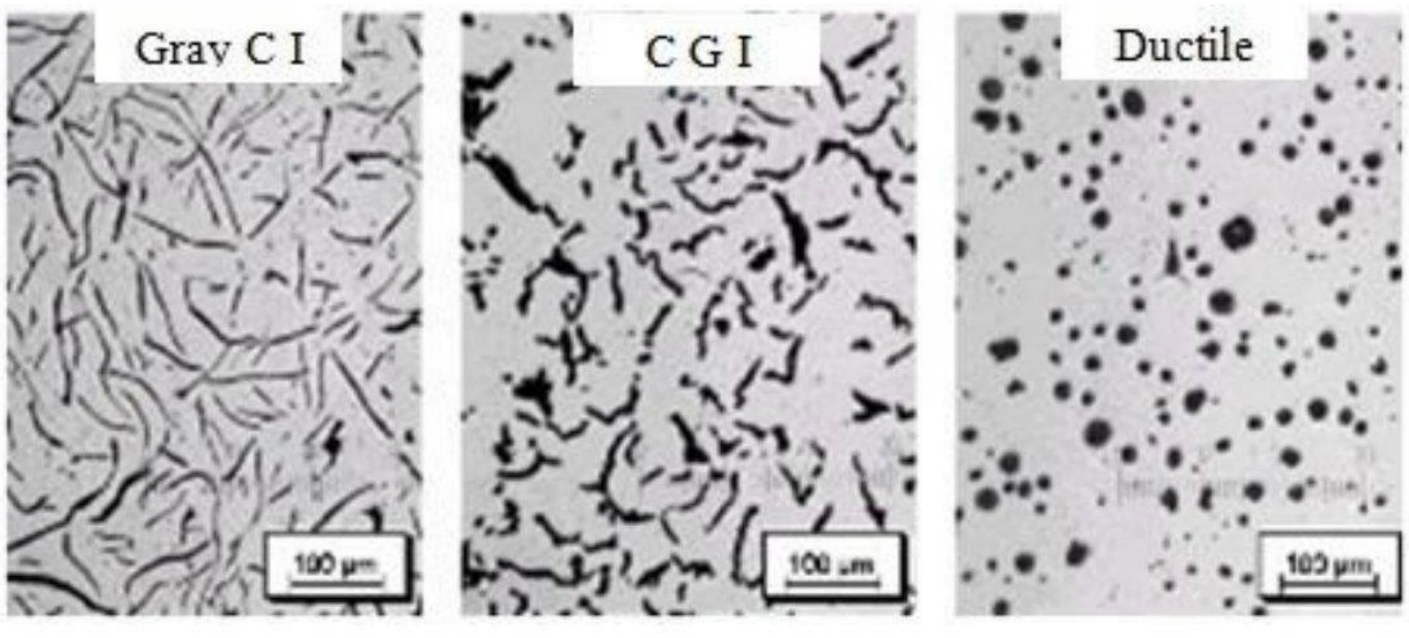


Partially Malleabilized Iron
→ Incomplete Ferritizing Anneal

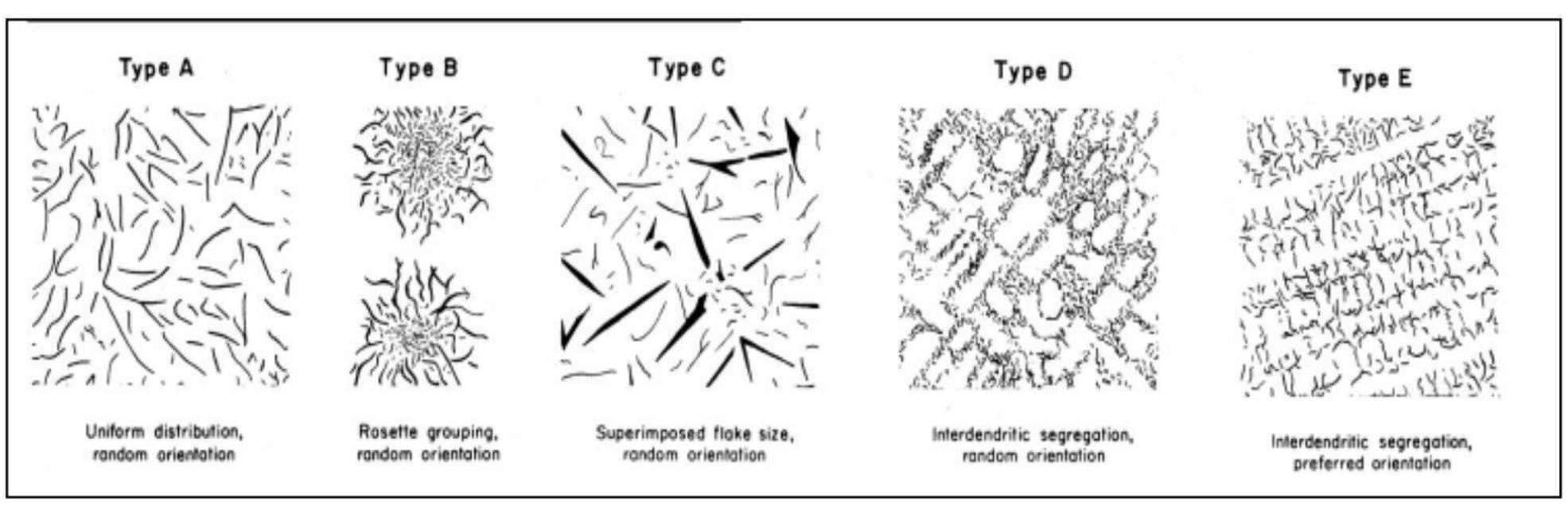
Fully Malleabilized Iron
→Complete Ferritizing Anneal

Compacted graphite cast iron

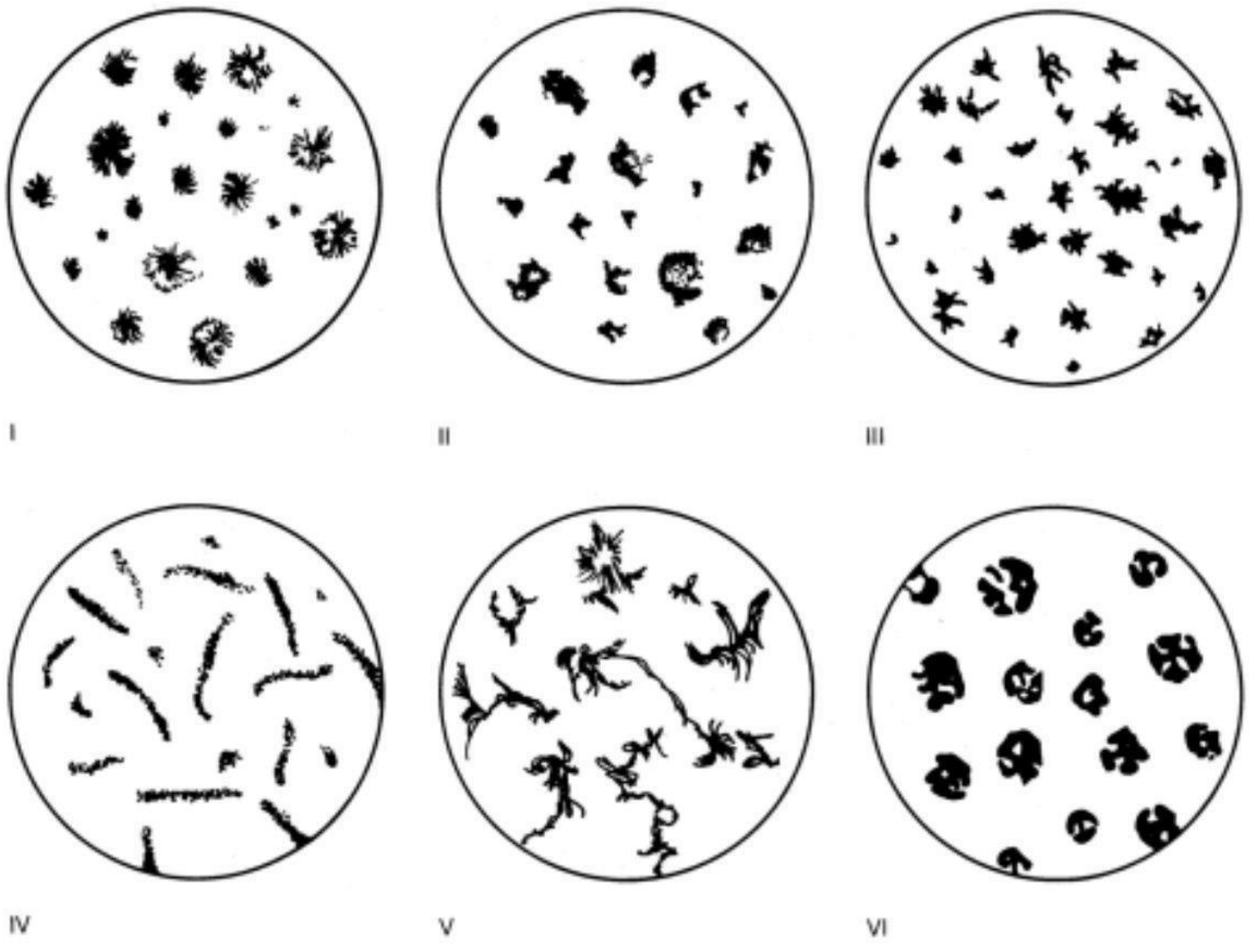




Types of Graphite flakes in gray iron



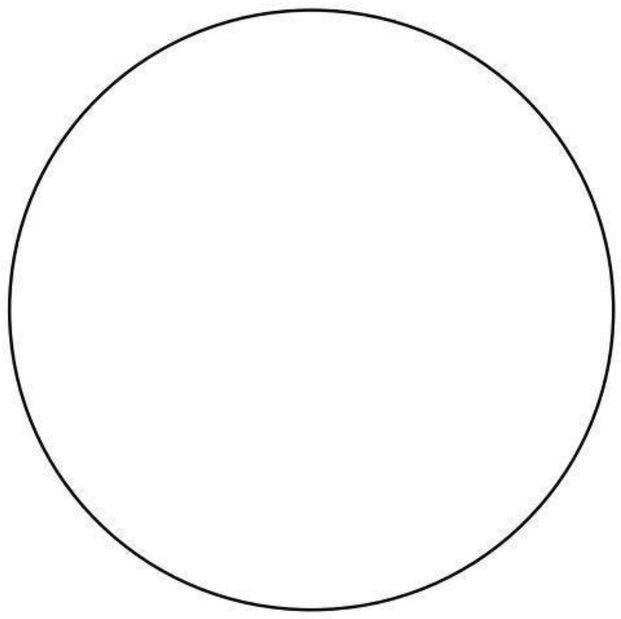
Typical Graphite shapes in cast iron



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 ……

……

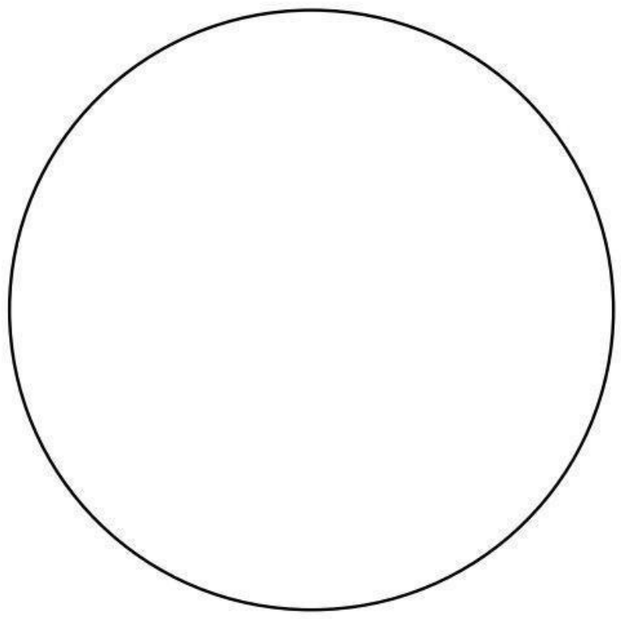
Any other observation ……

……

……

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 ……

……

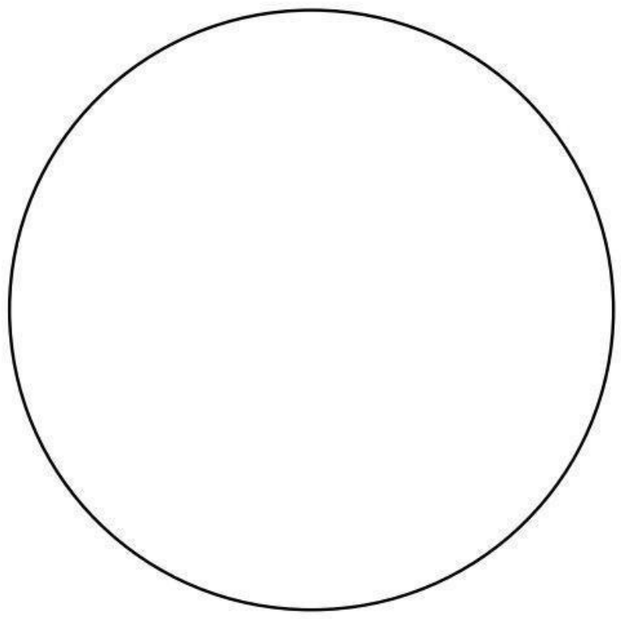
Any other observation ……

……

……

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 ……

……

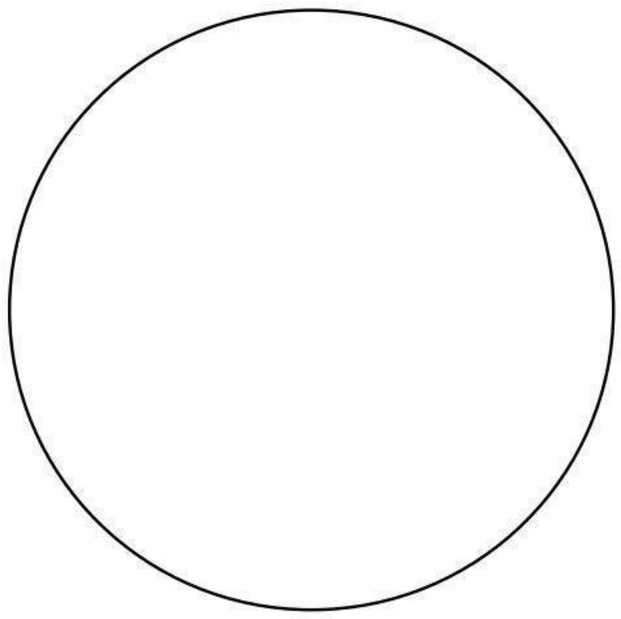
Any other observation ……

……

……

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_3C and Fe-C phase diagram?

2. Compared to Fe-Fe_3C phase diagram, why does eutectic and eutectoid composition will shifts 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 gray iron is brittle and weak in tension?

5. How to test grey iron, compacted iron or white cast iron in foundry practice. [other than metallographic technique]

6. Is it possible to produce malleable cast iron in pieces having large cross-sectional dimensions? Why or why not?

7. Compute the volume percent of graphite V_{Gr} in a 2.5 wt %C cast iron, assuming that all the carbon exists as the graphite phase. Assume densities of 7.9 and 2.3 g/cm^3 for ferrite and graphite, respectively.

MICROSTRUCTURAL ANALYSIS OF NON-FERROUS METALS

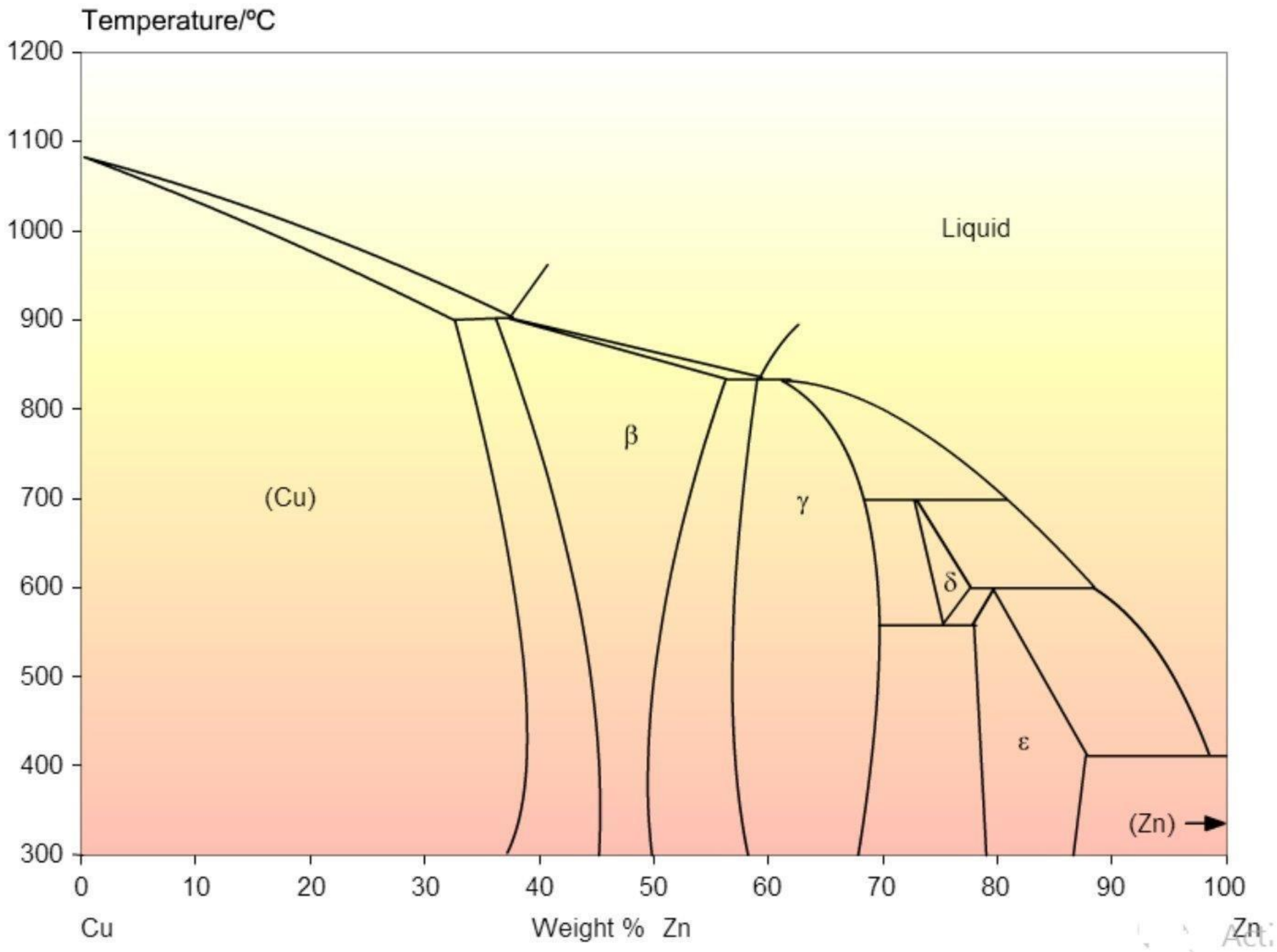
Objective

1. To be familiar with metallographic preparation techniques of non-ferrous metals
2. To be familiar with microscopic observation of phases present in Brass & Bronze and Aluminium alloy.
3. To interpret the microstructure with phase diagram

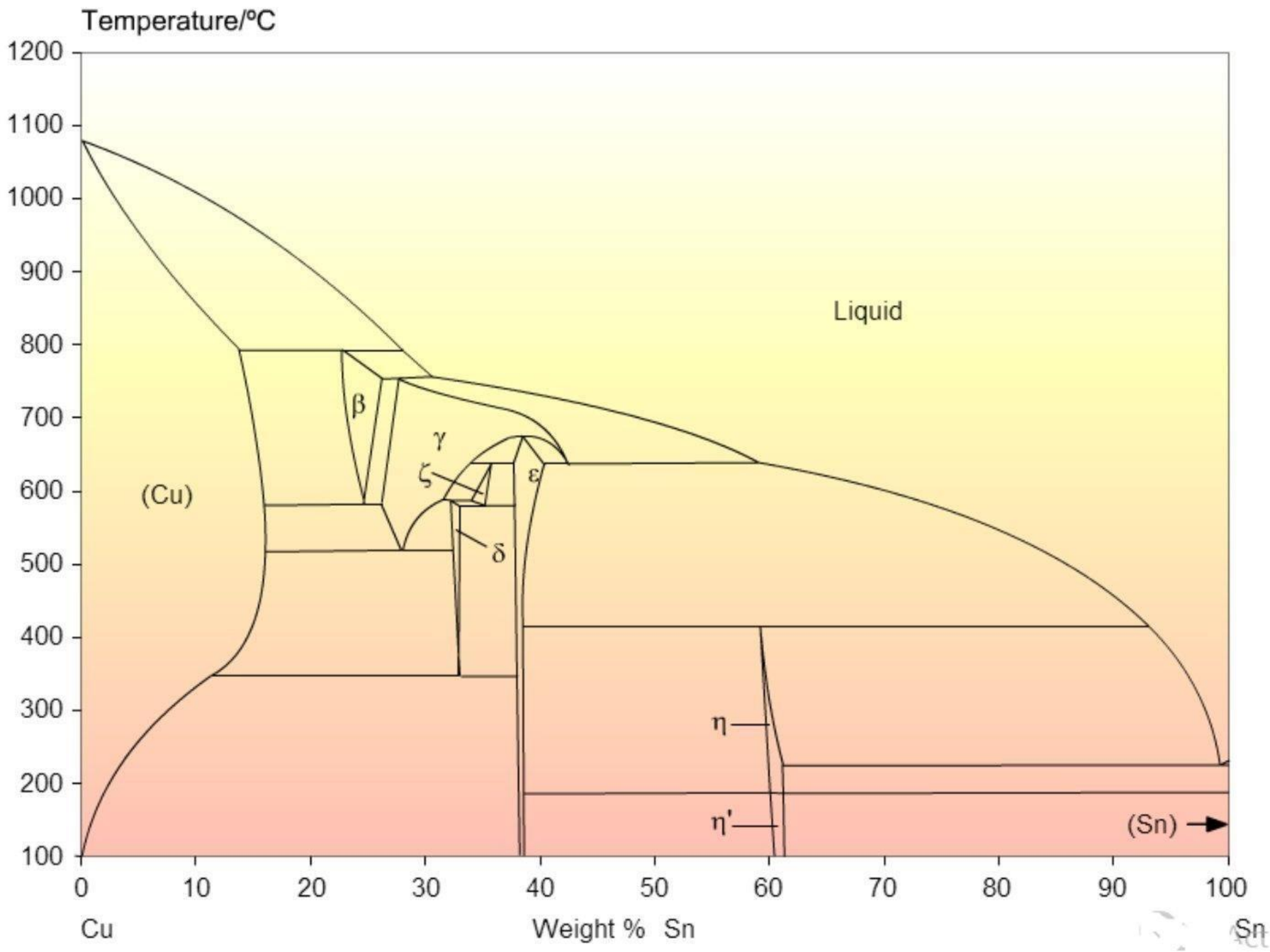
Theory

Brass is a copper-zinc alloy, whereas bronze is a copper-tin alloy. Brass is stronger than copper and has a higher malleability then either copper and zinc. Brass is also a good conductor of heat, has excellent acoustic properties and is generally resistant to corrosion in salt water. Brass is commonly rolled and extruded; however, these processes also work hardens and can be quanitified by metallographic analysis

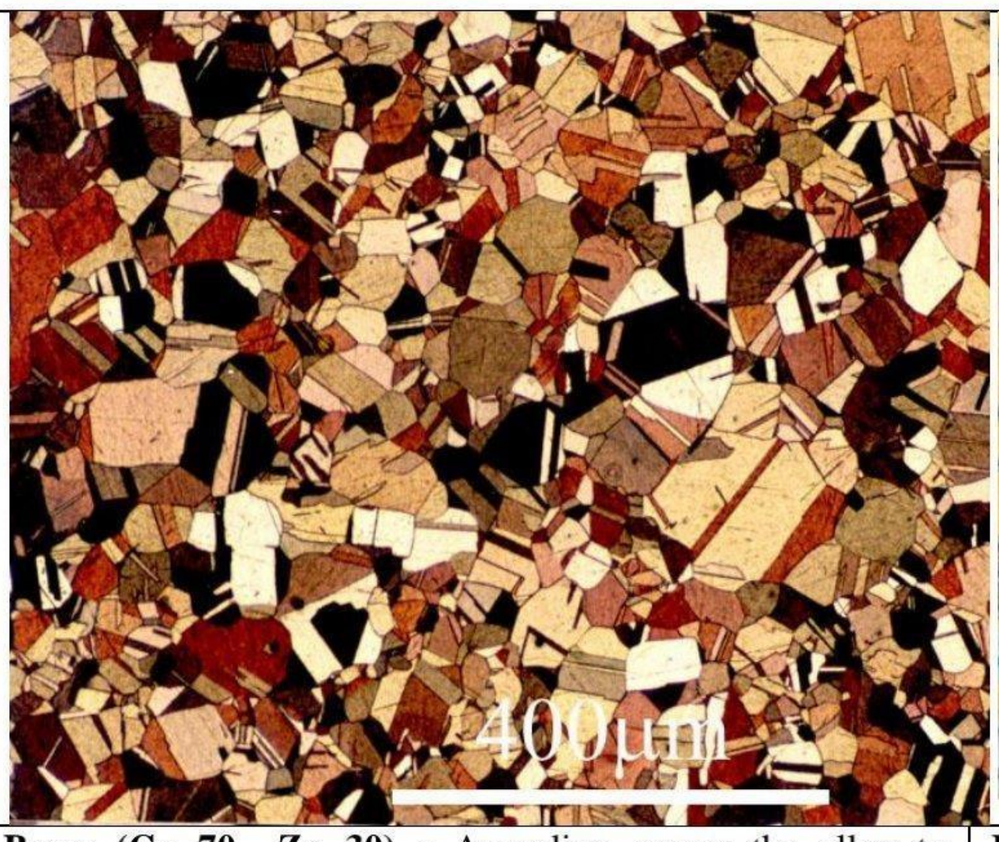
Observations



Identify the Phases, Temperatures & sitions of Cu-Zn phase diagram alloy

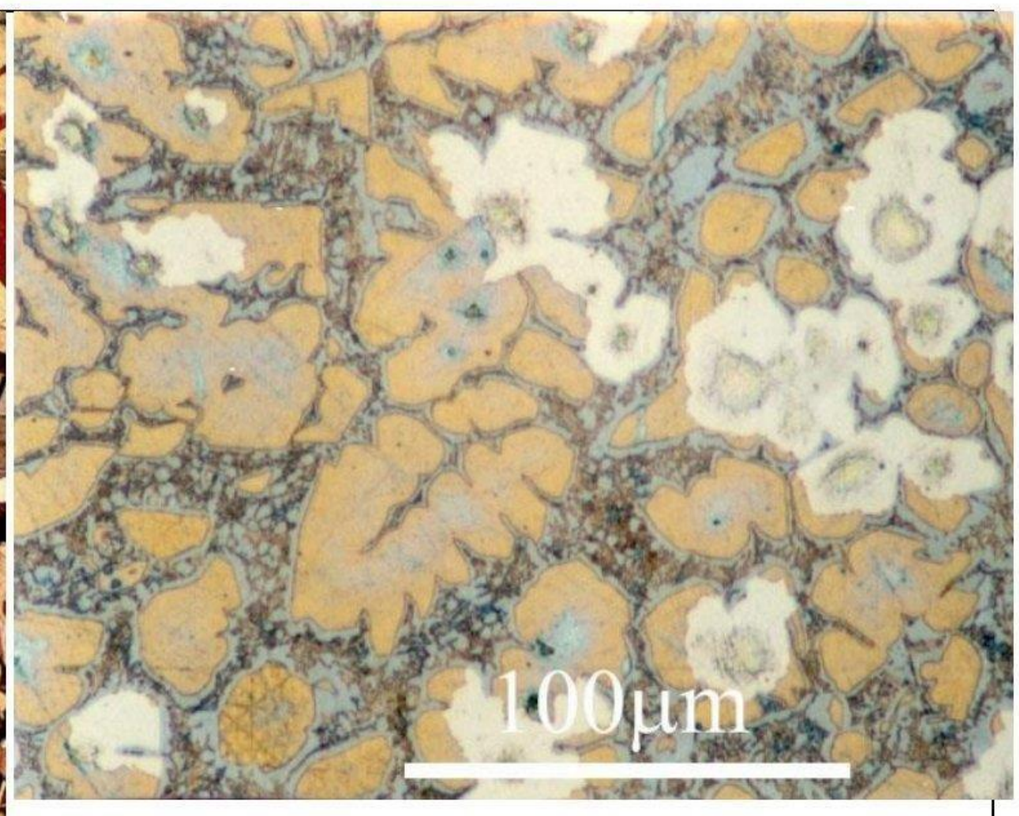


Identify the Phases, Temperatures & compositions of Cu-Sn phase diagram alloy



Brass (Cu 70 –Zn 30) : Annealing causes the alloy to recrystallise and grow. This results in a single a phase containing annealing twins (cast Annealed)

Etchant : Ferric chloride

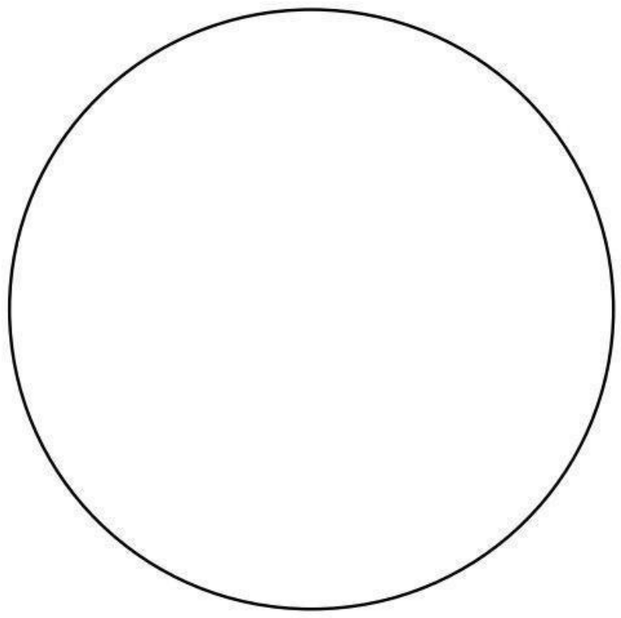


Bronze (Cu 80-Sn 20) : As cast copper rich bronze showing copper dendrites in a matrix of α (copper) and β phases

Etchant : Ferric Chloride

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 ……

……

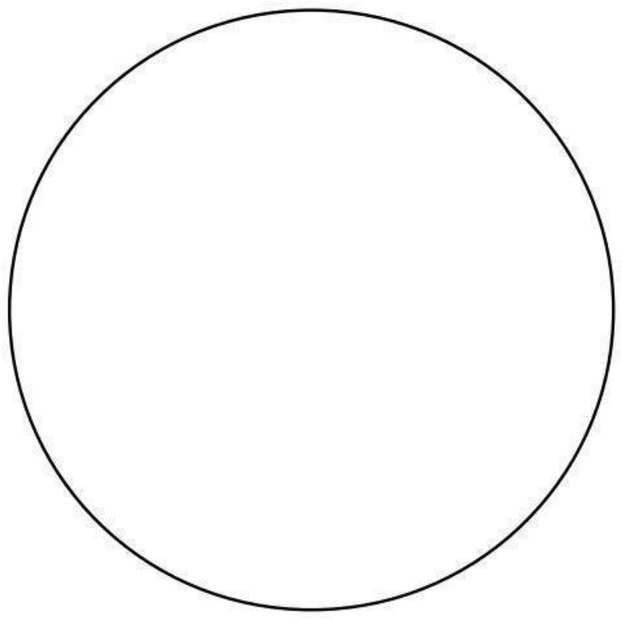
Any other observation ……

……

……

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 is the most important property of copper?

2. Write down the possible invariant reactions in Cu-Zn phase diagram?

3. What are the different types of brasses available in market, explain with utility.

4. What is dezincification? How can it be minimized?

5. Write down the possible invariant reactions in Cu-Sn phase diagram?

6. Differentiate between the terms brass and bronze?

7. Classify the different types of bronzes based on alloying elements

8. How is the core structure homogenised?

9. What are the major applications of non ferrous metals like Al, Ti and Superalloys.

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

STUDY OF SPECIMEN MOUNTING PRESS AND PREPARATION OF MOUNTED SPECIMEN

Objective: Preparation of a Specimen for metallographic examination.

FOR METALS

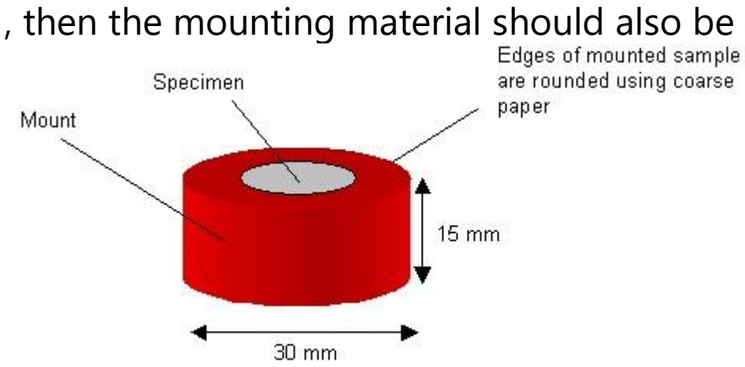
When preparing samples for microscopy, it is important to produce something that is representative of the whole specimen. It is not always possible to achieve this with a single sample. Indeed, it is always good practice to mount samples from a material under study in more than one orientation. The variation 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 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

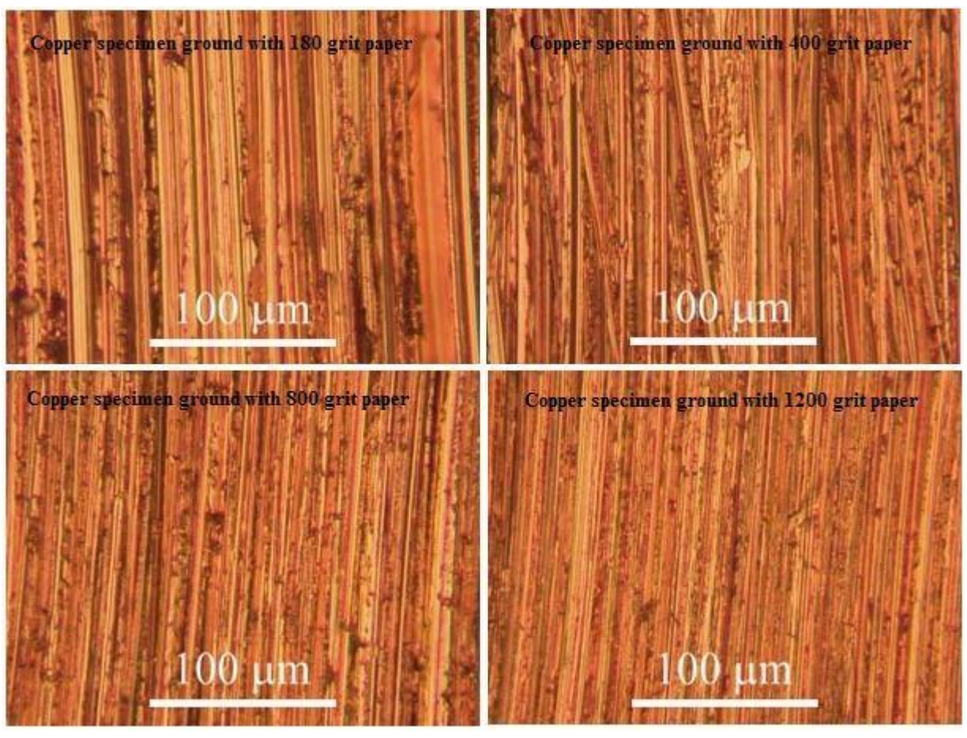
Mounting of specimens is usually necessary to allow them to be handled easily. It also minimises the amount of damage likely to be caused to the specimen itself. The mounting material used should not influence the specimen as a result of chemical reaction or mechanical stresses. It should adhere well to the specimen and, if the specimen is to be electropolished (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 thermosoftening 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:

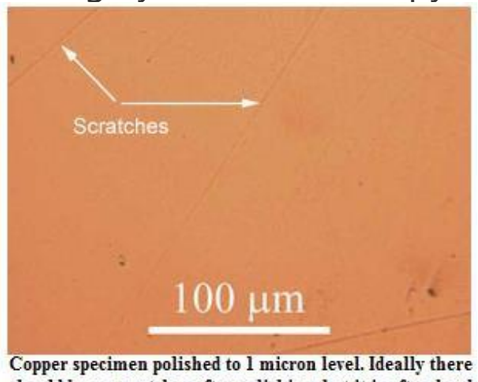
$$\text{Paper grit size} = \frac{16000}{\text{Width of largest scratch (in microns)}}$$

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

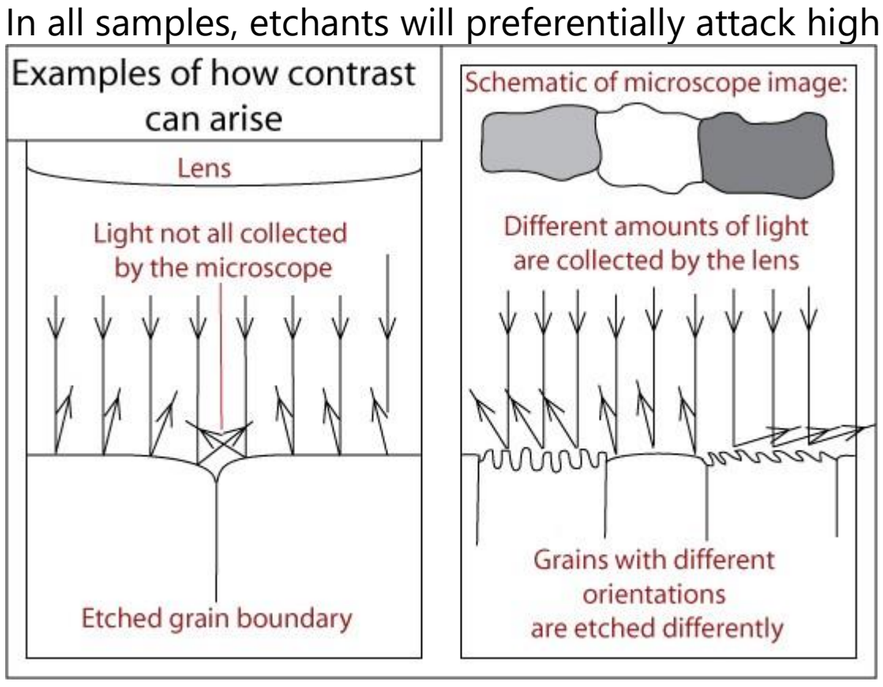
Mechanical polishing will always leave a layer of disturbed material on the surface of the specimen, if the specimen is particularly susceptible to mechanical damage (or excessive force is used in the grinding and polishing stages) debris can become embedded in the surface and plastic deformation may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

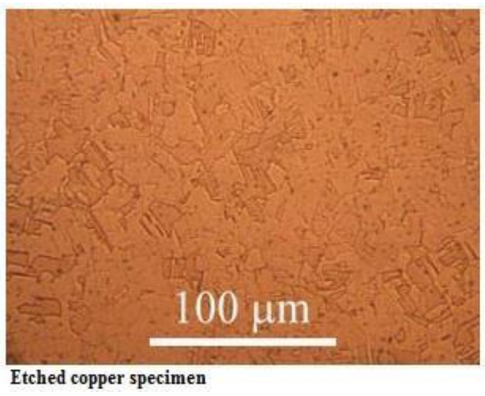
In all samples, etchants will preferentially attack high energy sites, such as boundaries and defects.



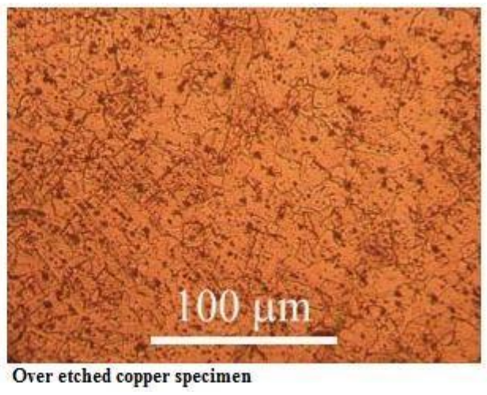
The specimen is etched using a reagent. For example, for etching stainless steel or copper and its alloys, a saturated aqueous solution of ferric chloride, containing a few drops of hydrochloric acid is used. This is applied using a cotton bud wiped over the surface a few times (Care should be taken 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.

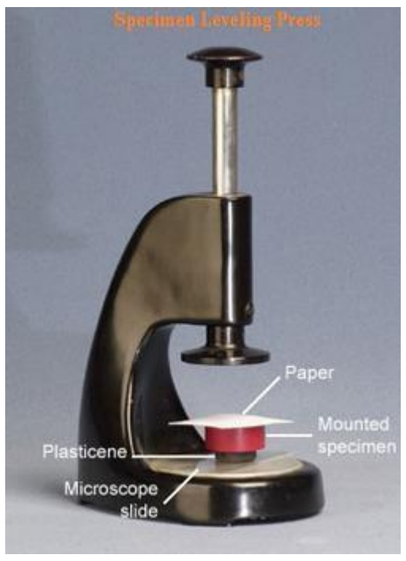


Etched copper specimen



Over etched copper specimen

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
Kalling's No. 1	Distilled water $CuCl_2$ 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.
Kalling's No. 2	$CuCl_2$ Hydrochloric acid Ethanol	5 grams 100 ml 100 ml	Immersion etching at 20 degrees Celcius	For etching duplex stainless steels and NiCu alloys and superalloys.
Kellers Etch	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.
Kroll's Reagent	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.
Nital	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.

Marble's Reagent	$CuSO_4$ 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_2SO_4 to increase activity.
Murakami's	$K_3Fe(CN)_6$ KOH Water	10 grams 10 grams 100 ml	Pre-mix KOH and water before adding $K_3Fe(CN)_6$	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.
Picral	Ethanol Picric acid	100 ml 2-4 grams	Seconds to minutes Do not let etchant crystallize or dry – **explosive**	Recommended for microstructures containing ferrite and carbide.
Vilella's Reagent	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

CERAMICS

Thin Sections

To prepare ceramic specimens for transmitted light microscopy, a thin slice, approximately 5 mm thick, is cut using a diamond saw or cutting wheel. One surface is then lapped using liquid suspensions of successively finer silicon carbide powders. Between stages in the process the specimen must be thoroughly cleaned. After final washing and drying the ground surface is bonded to a microscope slide with resin. A cut off saw is used on the exposed face to reduce the thickness to about 0.7 mm. The specimen is then lapped to take it to the required thickness – usually about 30 μm, although some ceramic specimens are thinned to as little as 10 μm, due to their finer grain size. The slide is checked for thickness under the microscope, and then hand finished. The slide is then covered with a protective cover slip. **Lapping**

The lapping process is an alternative to grinding, in which the abrasive particles are not firmly fixed to paper. Instead a paste and lubricant is applied to the surface of a disc. Surface roughness from coarser preparation steps is removed by the micro-impact of rolling abrasive particles.

Polished sections

These differ from ordinary thin sections in that the upper surface of the specimen is not covered with a cover slip, but is polished. Care must be taken to prevent the specimen breaking. Sections may be examined using both transmitted and reflected light microscopy, which is particularly useful if some constituents are opaque.

POLYMERS

Thin sections

Thin sections of organic polymers are prepared from solid material by cutting slices using a microtome – a mechanical instrument used for cutting thin sections. They must be cut at a temperature below the glass transition temperature of the polymer. A cut section curls up during cutting and must be unrolled and mounted on a microscope slide and covered with a cover slip. A few drops of mounting adhesive are used and must be compatible with the specimen. As always the mounting temperature must not affect the microstructure of the specimen.

The thickness of cut slices of polymer tends to lie in the range 2 to 30 μm depending on the type of material. Harder polymers can be prepared in the same way as thin ceramic specimens.

Polished sections

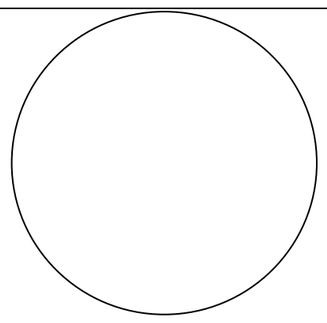
These are prepared in the same way as metallographic specimens. Elastomers are more difficult to polish than thermosetting polymers and require longer polishing times. Lubricants used during polishing must not be absorbed by the specimen.

As crystalline regions are attacked more slowly than amorphous ones, etching of polymer specimens can produce contrast revealing the polymer structure.

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?

6. Why metallographic samples are sometimes mounted in plastic?

7. Why microstructures are different for different metals?

8. What is emery/grit paper? What is the significance of 80,200,240,320...etc

9. State the different type of polishing clothes? On which basis can we select the polishing clothes?

ASTM GRAIN SIZE ANALYSIS

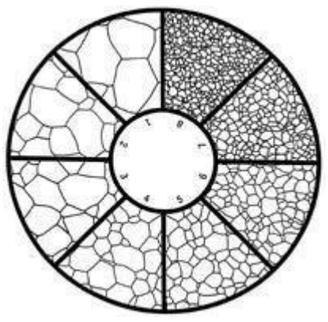
Objective

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. ASTM Method
ii. Heyn's intercept method
iii. Jeffries planimetric method



Grain Size Determination

i.ASTM Method: American Society for Testing Materials (ASTM) has developed a method to specify the grain size in a specimen by a number G called grain size number. The grain size number. The grain size number is related to the number of grains per square mm at a magnification of 1X (linear). The measurement is made directly under the microscope or on a photomicrograph of the specimen.

The observed grain structure can also be compared with the ASTM chart carrying grain size numbers. By proper matching the grain size number of the microstructure under examination is known from which the grain size diameter can be found out. In ASTM method the standard number of the grain per mm^2 is related to the grain size number at magnification of 01X by the following relation:

$$G = -2.9542 + 1.4472 \ln n$$

$$\ln n = \frac{G + 2.9542}{1.4472}$$

$$n = e^{(G+2.9542)/1.4472}$$

Note: The ASTM grain size number, N, is related to the number of grains per square inch at 100X magnification, n, by the relationship,$N=2^{n-1}$Where, N is the ASTM grain size number, and n is the number of grains per square inch at 100X.

The micrographs are always taken at magnifications other than 01X of the area not exactly 1 mm^2. Conversion to 1X magnification and to 1 mm^2 area should be made to find out the grain size number. For example, a photomicrograph of a microstructure shows 30 grains in an area of 30 X 40 mm^2 at 250X.

The ASTM data for the grain size number, number of grain/mm^2 and average grain size diameter in mm is given in Table 1.

ASTM no	Grains/mm^2	Grains/mm^3	Ave. Grain Dia. (mm)
-1	3.9	6.1	0.5
0	7.8	17.3	0.36
1	15.5	49.6	0.26
2	31.0	138.0	0.18
3	62.0	391.0	0.125
4	124.0	1105.0	0.090
5	248.0	3126.0	0.065
6	496	8842.0	0.045
7	992	25010.0	0.032
8	1986.0	70706.0	0.022
9	3976.0	200,000.0	0.016
10	7940.0	566,000.0	0.011

11	15870.0	1600,000.0	0.008
12	31700.0	4527000.0	0.006

The number of grains per mm^2 at 1X and the grain size number can be found out as given below:

Area of the photomicrograph = 1200 mm^2
At 250X (linear), the actual area will be = $\frac{1200\ mm^2}{250\times250} = 0.0192\ mm^2$

Equivalent number of grains at 1X can be obtained by the following relationship.

$$\frac{n}{1\ mm^2} = \frac{30}{0.0192\ (mm^2)}$$

$$n = \frac{30}{0.0192} = \frac{30\times10^4}{192} = 1.5\times10^4 = 1500$$

The ASTM chart (Table 1) gives the grain size number 'G' as 11 for this value of n. The value of G can also be calculated by using equation "G = -2.9542 + 1.4472 *ln* n"

$$G = -2.9542 + 1.4427\ \ln 15000$$

$$G = -2.9542 + 1.421\times9.6158 = 10.76 \approx 11$$

The same value of G is found from the ASTM chart (Table 1) for 15000 grains/mm^2 From Table 1, the diameter of the grain is 0.008 mm.
Let us compare it with the value obtained from direct observation. From the direct measurement, the n = 15000/mm^2
No of grains in 1mm (linear) = $15000^{1/2}$ =122.47 ~ 123
Diameter of the grain = $\frac{1}{123} = 0.008\ mm$(same as ASTM Table) **ii.Heyn's intercept Method :**

This method is easier than ASTM method. The number of grain boundaries intercepting a test line passing through the grains in any direction is counted. An eyepiece calibrated with a micrometer scale is required to take the measurement while viewing the microstructure. The number of boundaries intercepted per unit length of the test line, N_L, can be computed if the number of boundaries intercepted by L mm length of the test line be n at a magnification of M.

No of intercepted boundaries per unit length (actual mm), $N_L = \frac{n}{L/M} = \frac{n.M}{L}$

Average, intercepted grain diameter, $\bar{l} = \frac{1}{N_L} = \frac{L}{n\times M}\ mm$

Other parameters related to grain boundaries like N_V , the number of grains per unit volume; S_V the surface areaof the grain per unit voulume; $\bar{A}$, the mean planar area per grain, are related to each other and are given in Table 2. These relationships have been experimentally found to be correct, $\bar{l}$and $\bar{A}$ are mostly used to characterize the grain size.

Table 2: Interrelationship of Grain Size Parameter

N_L	N_A	N_V	l	A	V
$S_V = \frac{6N_L^2}{3}$	$\frac{7N_A^2}{3}$	$\frac{8^{N_{V1/3}}}{3}$	-	-	-
$N_V = 0.422N_L^3$	$\frac{2^{N_{A2/3}}}{3}$	-	-	-	$\frac{1}{V}$
$N_A = 0.735N_L^2$	-	-	-	$\frac{1}{A}$	-
N_L =	-	-	$\frac{1}{l}$	-	-

iii.Jeffery's Planimetric Method

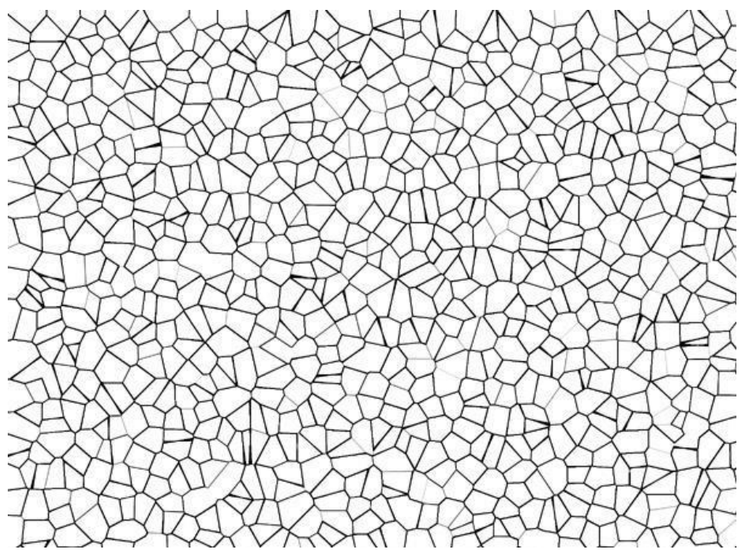
In this method a rectangle or a circle of known area commonly 5000 mm^2 is engraved or drawn, as the case may be, on the eye piece of the microscope or on the photomicrograph. The magnification which gives about 50 grains inside the circle or rectangle is used for the study. All the grains which intersect the boundary of the circle or rectangle are summed up and reduced by half and are added into the number of grains, which fall well inside the boundary line to get the number of equivalent grains. This number multiplied by jeffery's multiplier gives the number of grains per mm^2. Jeffery's multiplier gives the number of grains per mm^2.
Jeffery's multiplier $k = \frac{M^2}{5000} = \frac{100\times100}{5000} = 2.0$; No. of grains per mm^2 =
(4.2 * 2.0) = 84.0 ; Grain size = 84.0 grains / mm^2

Relationship between magnification used and Jeffries Multiplier, k, for an area of 5000mm^2 (a circle of 79.8 mm diameter) (f=0.0002M^2)

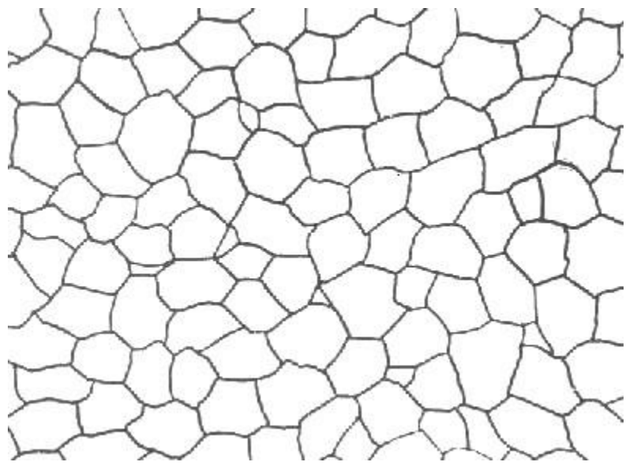
Magnification Used, X	Jeffries Multiplier, k, to obtain Grains/mm^2
1	0.0002
10	0.02
25	0.125
50	0.5
75	1.125
100	2.0
150	4.5
200	8.0
250	12.5
300	18.0
500	50.0
750	112.5
1000	200.0

OBSERVATIONS

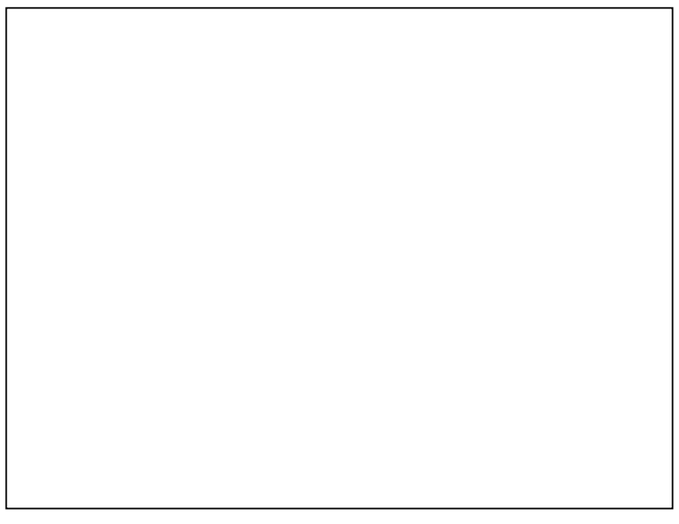
1. The microstructure obtained at 200X magnification is given below. Determine the ASTM grain size of by grain counting method.



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



3. Draw the microstructure obtained at _____X magnification is given below. Determine the ASTM grain size of by grain counting method and intercept method.



Answer the Following Questions

1. Which of the following alloys have one phase and two phases? (Brass, Stainless steel, Sn – 10%Pb), low C –steel, pure aluminium)

2. Will the yield strength decreases or increase with the increase in the grain size?

3. Why are grain boundaries visible in polished and etched samples?

4. Does the grain size number increase or decrease with decreasing grain size? Why?

5. Determine the ASTM grain size number of a metal specimen if 45 grains per square inch are measured at a magnification of 100X

6. For an ASTM grain size of 6, approximately how many grains would there be per square inch at **(a)** a magnification of 100, and **(b)** without any magnification?

7. Determine the ASTM grain size number if 30 grains per square inch are measured at a magnification of 250X

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

IMAGE ANALYSIS OF VARIOUS FERROUS AND NON-FERROUS ALLOYS

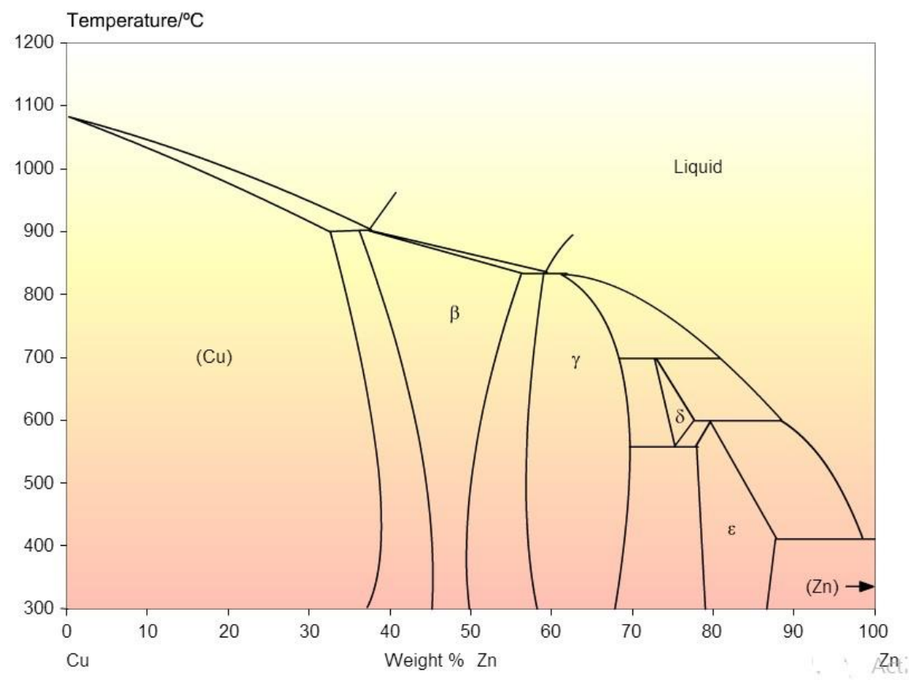
Objective

1. To be familiar with metallographic preparation techniques of non-ferrous metals
2. To be familiar with microscopic observation of phases present in Brass & Bronze and Aluminium alloy.
3. To interpret the microstructure with phase diagram

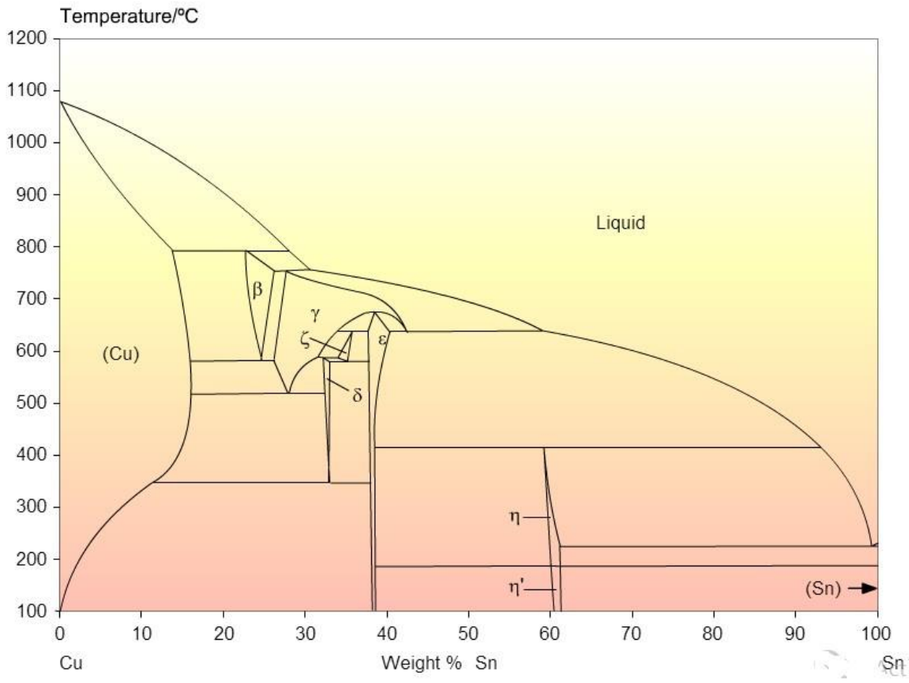
Theory

Brass is a copper-zinc alloy, whereas bronze is a copper-tin alloy. Brass is stronger than copper and has a higher malleability then either copper and zinc. Brass is also a good conductor of heat, has excellent acoustic properties and is generally resistant to corrosion in salt water. Brass is commonly rolled and extruded; however, these processes also work hardens and can be quanitified by metallographic analysis

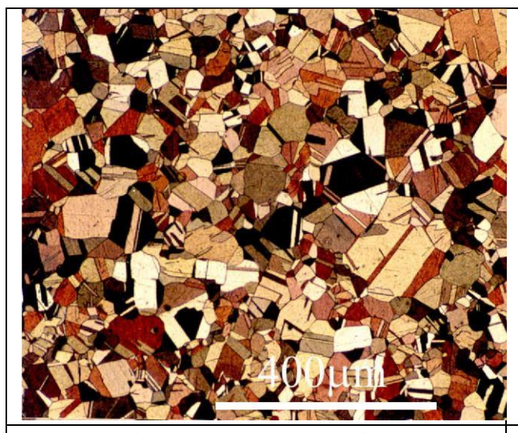
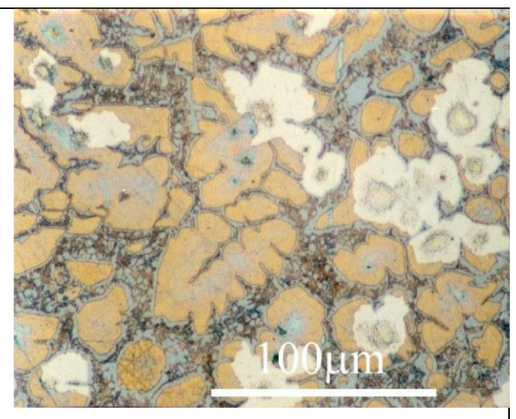
Observations



Identify the Phases, Temperatures & compositions of Cu-Zn phase diagram alloy



Identify the Phases, Temperatures & compositions of Cu-Sn phase diagram alloy

	
Brass (Cu 70 –Zn 30) : Annealing causes the alloy to recrystallise and grow. This results in a single a phase containing annealing twins (cast Annealed) **Etchant :** Ferric chloride	**Bronze (Cu 80-Sn 20) :** As cast copper rich bronze showing copper dendrites in a matrix of α (copper) and β phases **Etchant :** Ferric Chloride

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

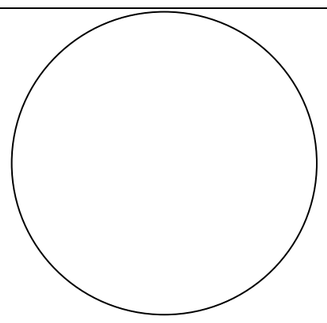
……

……

.....

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

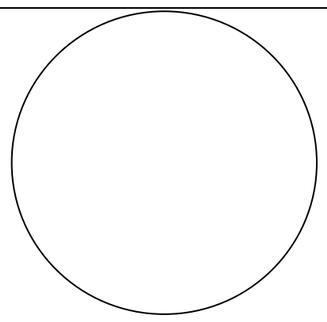
……

……

……

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 is the most important property of copper?

2. Write down the possible invariant reactions in Cu-Zn phase diagram?

3. What are the different types of brasses available in market, explain with utility.

4. What is dezincification? How can it be minimized?

5. Write down the possible invariant reactions in Cu-Sn phase diagram?

6. Differentiate between the terms brass and bronze?

7. Classify the different types of bronzes based on alloying elements

8. How is the core structure homogenised?

9. What are the major applications of non ferrous metals like Al, Ti and Superalloys.

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