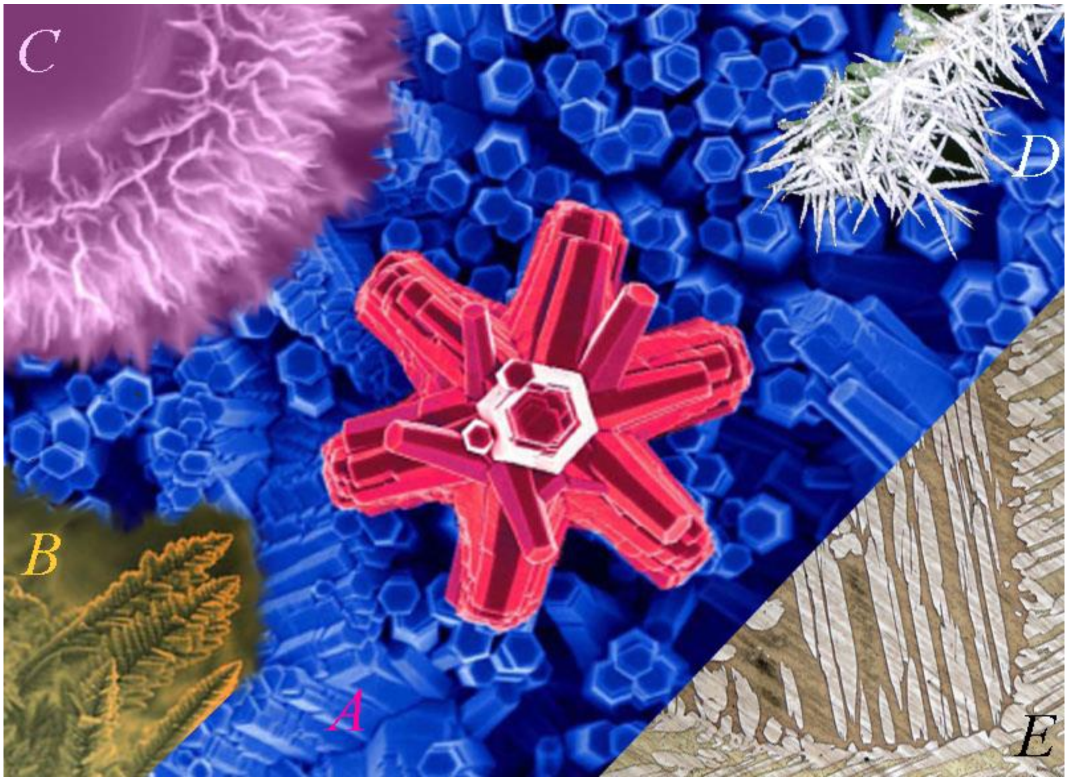
PHYSICAL METALLURGY LABORATORY

OBSERVATION BOOK



Department of Metallurgy & Materials Engineering

VEER SURENDRA SAI UNIVERSITY OF TECHNOLOGY, BURLA

Odisha – 768018

PHYSICAL METALLURGY LABORATORY

OBSERVATION BOOK

Name of the Student: ………………………………………………

Scholar No: ....

LABOROTARY COORDINATOR(S)

A. LAVA KUMAR
Assistant Professor

SUBHADRA SAHOO
Assistant Professor

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FOREWORD

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

Ms. Subhadra Sahoo
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 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.)

4. Comply with fire regulations concerning storage quantities, types of approved containers and cabinets, proper labeling, 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.

____

(Signature with date)

PHYSICAL METALLURGY LAB

Odd semester 2015-16

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 through Ball Models	......./10	
2.	Metallurgical Microscope: Principles and Operations	......./10	
3.	Specimen Preparation techniques for Metallographic Analysis	......./10	
4.	Fe-C Phase Diagram	......./10	
5.	Microstructural Analysis of Carbon Steels	......./10	
6.	Microstructural Analysis of Cast Irons	......./10	
7.	Microstructural Analysis of Non-Ferrous Metals	......./10	
8.	Standard test Methods for Estimation of Grain Size	......./10	
	PROJECT : Colour Metallography	......./20	
	Total Marks obtained		

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 your each experiment. Your instructor will make a assessment and transmit the mark.

A. LAVA KUMAR
Laboratory Coordinator

SUBHADRA SAHOO
Laboratory Co-coordinator

EXPERIMENT 1

CRYSTAL STRUCTURE

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.

Ideal Crystal
(Considers ***both*** Geometrical Entity AND Physical Property/)

↓

Crystal
(Here we consider ***either*** geometrical entity OR physical property)

↓

Crystal
(Consider ***either*** the Orientational OR the Positional Order)

↓

'Real Crystal'
(Presence of Crystalline defects & Free Surface & Thermal Vibration)

↓

~Microconstituents
(Put in Multiple Crystals *(Phases)* giving rise to interfacial defects)

↓

Microstructure
(Put in multiple ~microconstituents and add additional residual stress[✝])

↓

Material
(Put in many microstructures)

↓

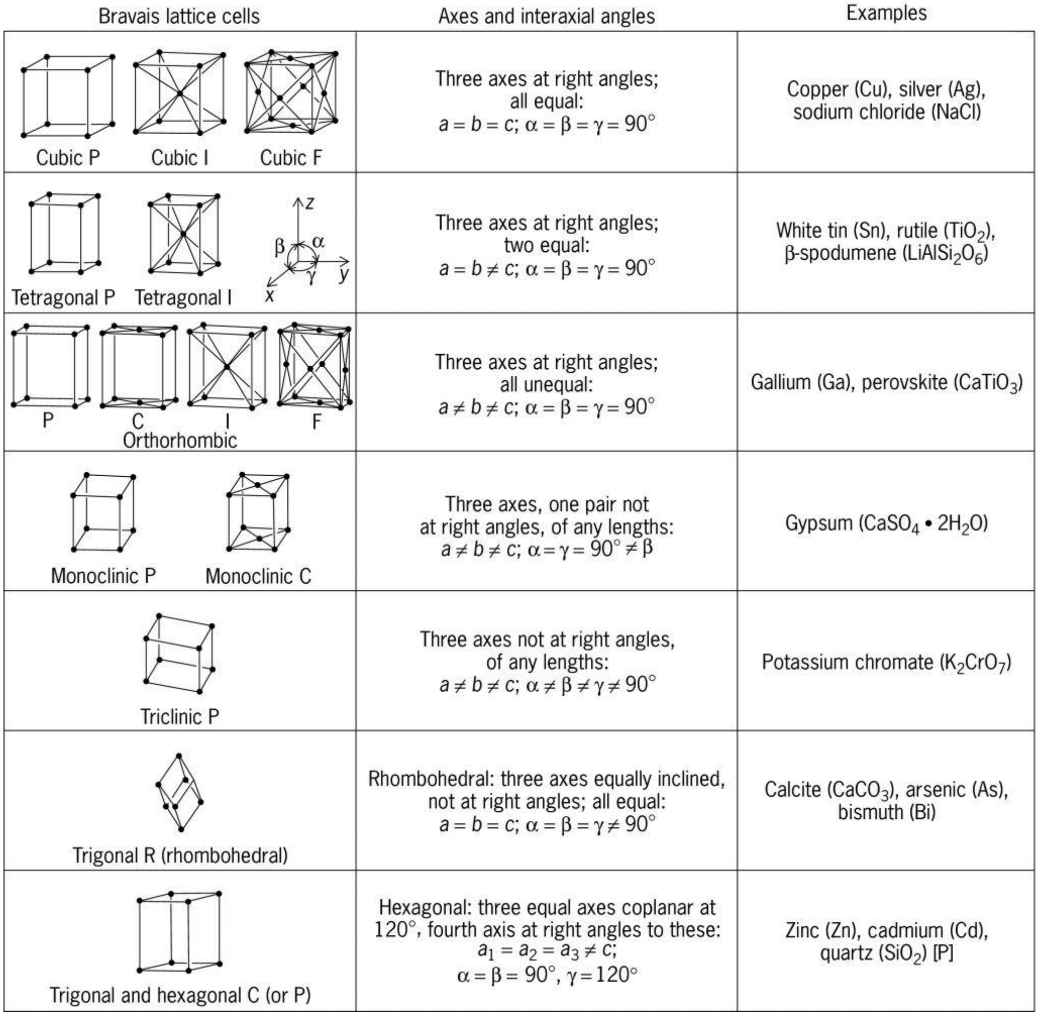
Component
(Put in material/s and/or material treatment i.e. temperature/pressure)

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.

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

Table 1: The 14 Bravais lattices



Bravais lattice cells	Axes and interaxial angles	Examples
Cubic P Cubic I Cubic F	Three axes at right angles; all equal: $a = b = c$; $\alpha = \beta = \gamma = 90°$	Copper (Cu), silver (Ag), sodium chloride (NaCl)
Tetragonal P Tetragonal I	Three axes at right angles; two equal: $a = b \neq c$; $\alpha = \beta = \gamma = 90°$	White tin (Sn), rutile (TiO_2), β-spodumene ($LiAlSi_2O_6$)
P C I F Orthorhombic	Three axes at right angles; all unequal: $a \neq b \neq c$; $\alpha = \beta = \gamma = 90°$	Gallium (Ga), perovskite ($CaTiO_3$)
Monoclinic P Monoclinic C	Three axes, one pair not at right angles, of any lengths: $a \neq b \neq c$; $\alpha = \gamma = 90° \neq \beta$	Gypsum ($CaSO_4 \bullet 2H_2O$)
Triclinic P	Three axes not at right angles, of any lengths: $a \neq b \neq c$; $\alpha \neq \beta \neq \gamma \neq 90°$	Potassium chromate (K_2CrO_7)
Trigonal R (rhombohedral)	Rhombohedral: three axes equally inclined, not at right angles; all equal: $a = b = c$; $\alpha = \beta = \gamma \neq 90°$	Calcite ($CaCO_3$), arsenic (As), bismuth (Bi)
Trigonal and hexagonal C (or P)	Hexagonal: three equal axes coplanar at 120°, fourth axis at right angles to these: $a_1 = a_2 = a_3 \neq c$; $\alpha = \beta = 90°$, $\gamma = 120°$	Zinc (Zn), cadmium (Cd), quartz (SiO_2) [P]

OBSERVATIONS

1. **Show the photographs of crystal structures what you made with the help of different materials.**

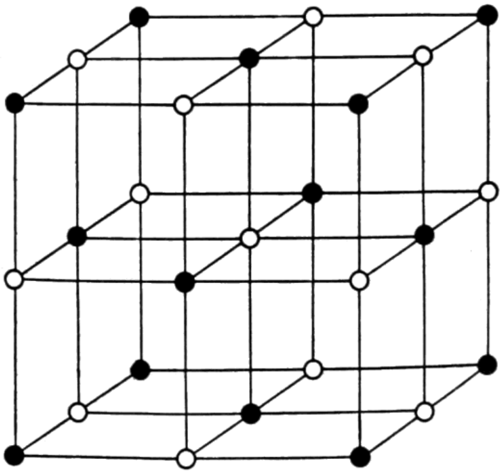
2. ***Draw the possible void structures in B.C.C and F.C.C.***

Crystal structure of metallic element: Generally, the metallic elements crystallize mainly in cubic structures.

Body centred cubic structure (B.C.C.): Atoms are arranged at the corners of the cube with another atom at the cube centre. *Face centred cubic structure (F.C.C):* Atoms are arranged at the corners and centre of each faces of the cubic cell.

Draw the BCC structure in the square given below and show the tetrahedral interstitial and octahedral interstitial site.	*Draw the FCC structure in the square given below and show the tetrahedral interstitial and octahedral interstitial site.*
Calculations No. of tetrahedral sites ________ No. of octahedral sites ________	*Calculations* No. of tetrahedral sites ________ No. of octahedral sites ________

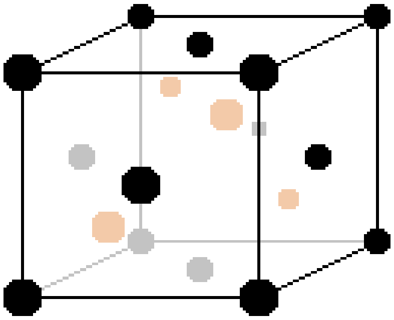
3. Identify the crystal structures and possible number of voids?



Crystal structure: ____________________________

No. of tetrahedral sites: ______________________

No. of octahedral sites: ________________________



Crystal Structure ____________________

No. of tetrahedral site ___________________

No. of octahedral site ____________________

Answer the following Questions

1. Define Crystal, Lattice and Motif.

2. What is symmetry? What is the relation between symmetry and crystal systems?

3. For a point at (xyz) show a translation, a reflection and an inversion operation.

4. What is a unit cell? What is lattice parameter?

5. What is Bravais lattice? How are the Bravais lattices obtained from the primitive cell? How many types of Bravais lattices are there?

6. How many atoms are present in unit cell of BCC, FCC, HCP and Diamond Cubic?

7. Define atomic packing factor. Obtain its expression for FCC?

8. Why FCC metals are more ductile compared to BCC and HCP metals?

9. Define linear density. and Planner density?

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

EXPERIMENT 2

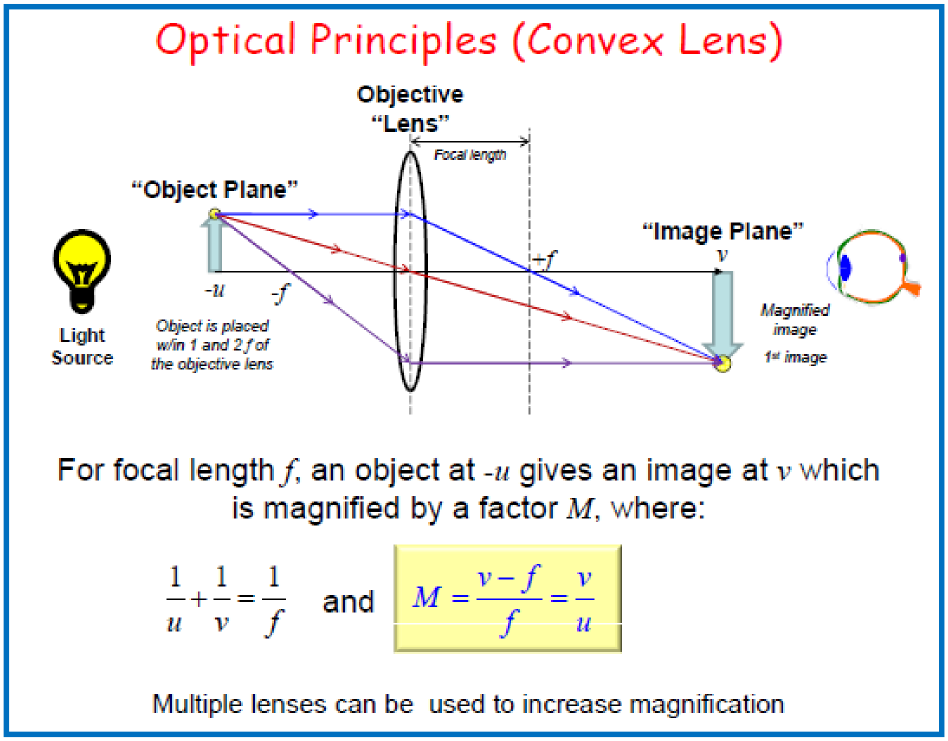
METALLURGICAL MICROSCOPE

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



Optical Principles (Convex Lens)

For focal length f, an object at $-u$ gives an image at v which is magnified by a factor M, where:

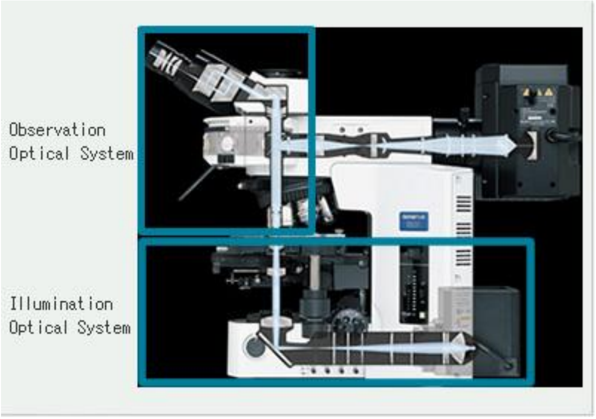
$$\frac{1}{u} + \frac{1}{v} = \frac{1}{f} \quad \text{and} \quad \boxed{M = \frac{v-f}{f} = \frac{v}{u}}$$

Multiple lenses can be used to increase magnification

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?

EXPERIMENT 3

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.

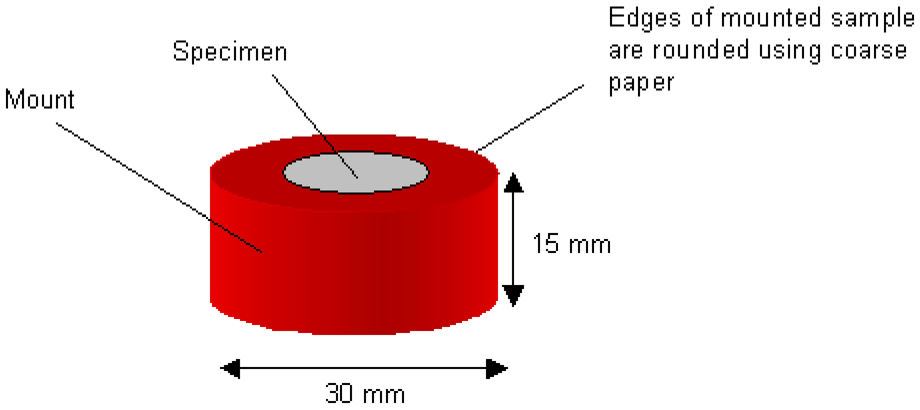
Cutting a specimen

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

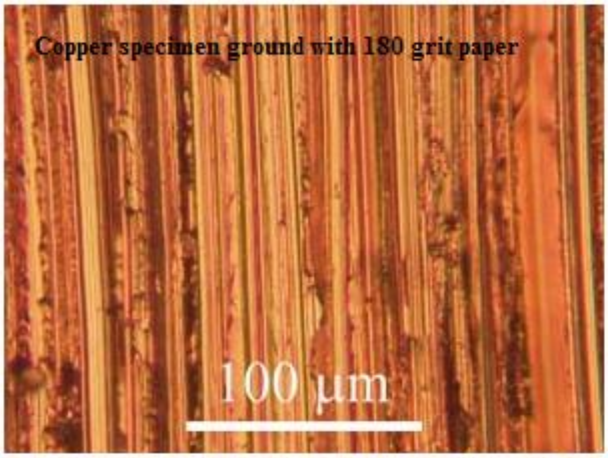
Mounting of specimens is usually necessary to allow them to be handled easily. It also minimises the amount of damage likely to be caused to the specimen itself. The mounting material used should not influence the specimen as a result of chemical reaction or mechanical stresses. It should adhere well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

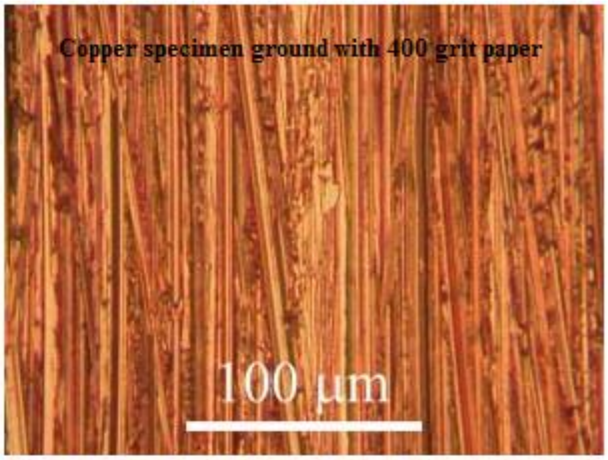


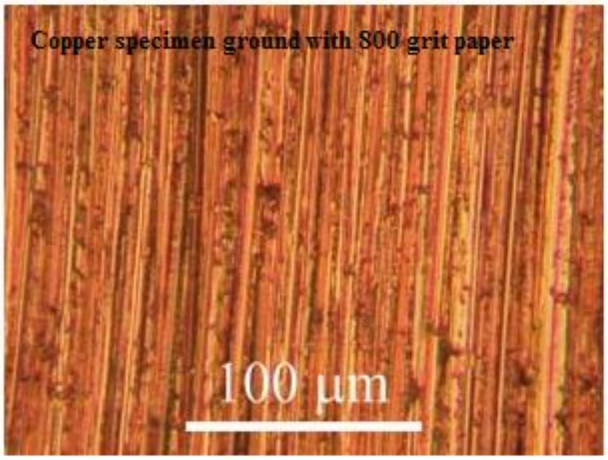
Specimens can be hot mounted (at around 200°C) using a mounting press, either in a thermosetting plastic (*e.g.* phenolic resin), or a thermosoftening plastic (*e.g.* acrylic resin). If hot mounting will alter the structure of the specimen a cold-setting resin can be used, *e.g.* epoxy, acrylic or polyester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

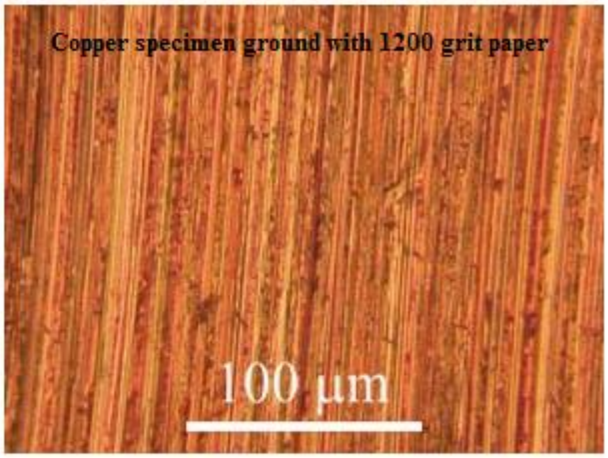
Grinding

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.


Copper specimen ground with 180 grit paper


Copper specimen ground with 400 grit paper


Copper specimen ground with 800 grit paper


Copper specimen ground with 1200 grit paper

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:

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

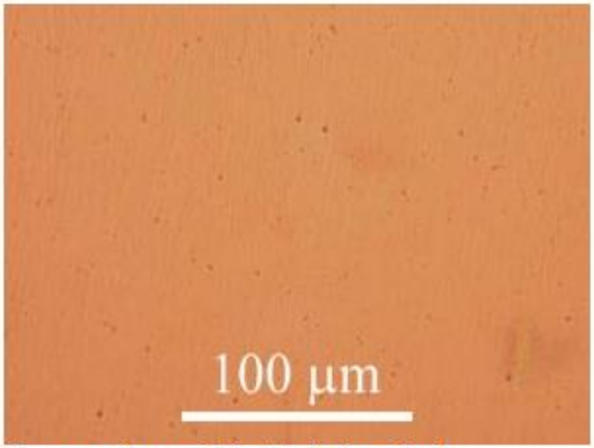
1. This prevents putting more damage into the sample than already exists; the coarsest grades of paper are often not useful.

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

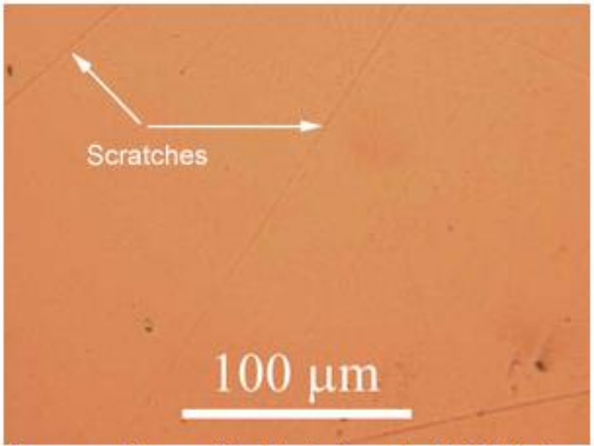
3. The series of photos below shows the progression of the specimen when ground with progressively finer paper.

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

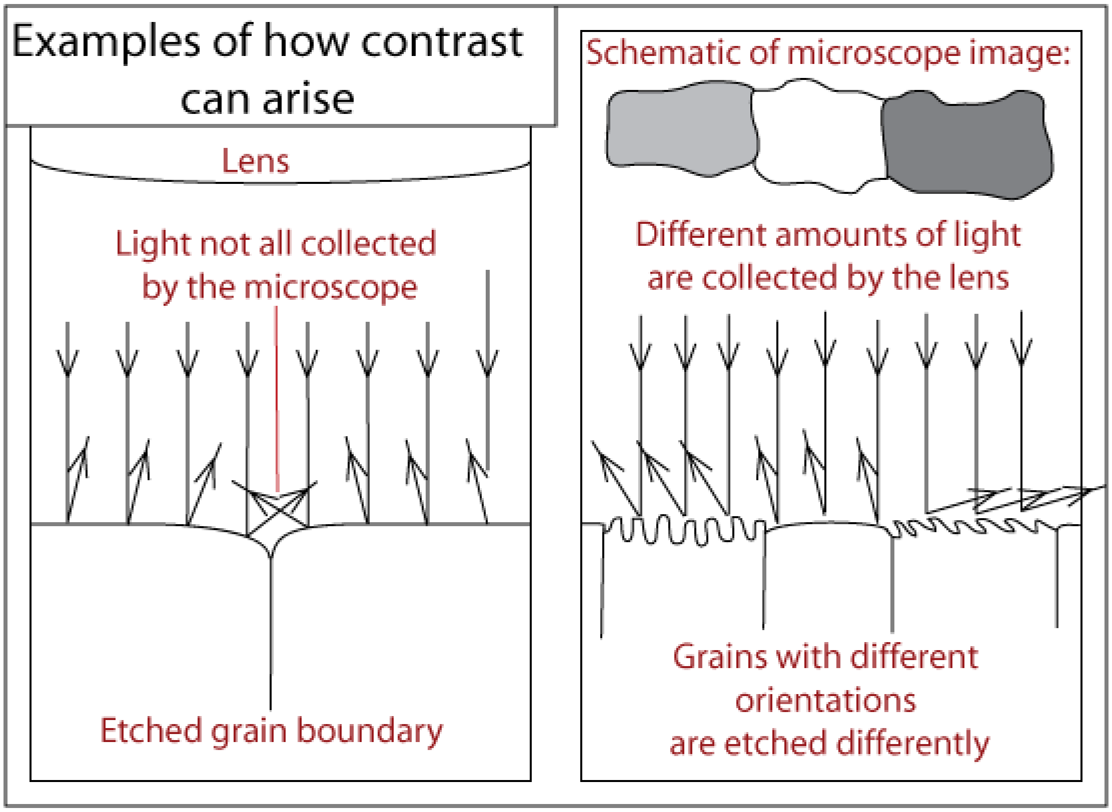
Mechanical polishing will always leave a layer of disturbed material on the surface of the specimen, if the specimen is particularly susceptible to mechanical damage (or excessive force is used in the grinding and polishing stages) debris can become embedded in the surface and plastic deformation may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

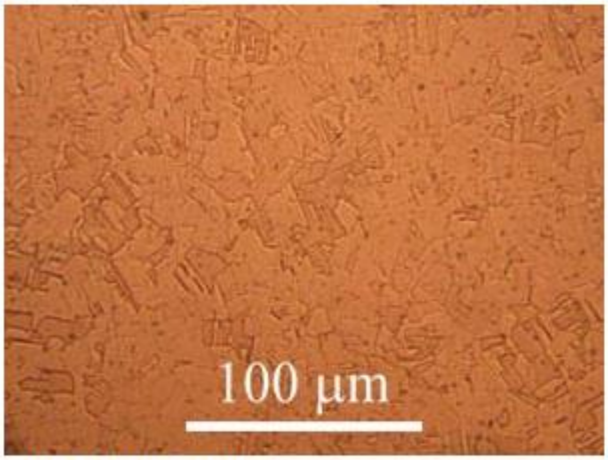
In all samples, etchants will preferentially attack high energy sites, such as boundaries and defects.



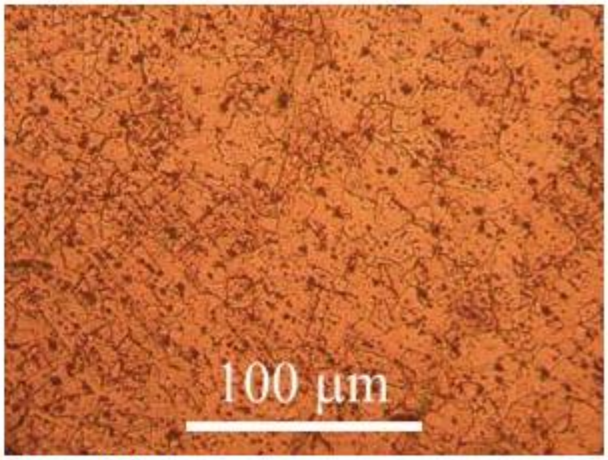
The specimen is etched using a reagent. For example, for etching stainless steel or copper and its alloys, a saturated aqueous solution of ferric chloride, containing a few drops of hydrochloric acid is used. This is applied using a cotton bud wiped over the surface a few times (Care should be taken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

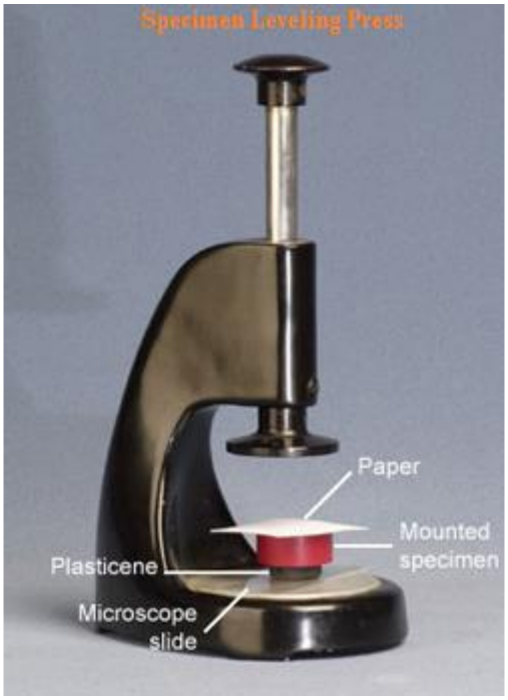


Etched copper specimen



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 75ºC; 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?

EXPERIMENT 4

IRON-CARBON PHASE DIAGRAM

1. Draw Fe-Fe_3C Phase Diagram and identify the phases

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

- **α-ferrite** – Interstitial solid solution of carbon in α-iron. α-ferrite has BCC crystal structure. It is a low temperature phase. The solubility of carbon in α-Fe is low. Maximum solubility of carbon is 0.025% at 723ºC. Ferrite is a soft phase with a hardness of 80-150 HV.
- **Austenite** – Interstitial solid solution of carbon in γ-iron. Austenite has FCC crystal structure, permitting high solubility of carbon – up to 2.1% at 1147 ºC. Austenite does not exist below 723ºC and maximum carbon concentration at this temperature is 0.83%. Austenite is a normally a metastable phase however, it can be made into a stable phase by alloying elements. Hardness of Austenite is 200-250 HB.
- **δ-ferrite** – Solid solution of carbon in δ-iron. Maximum concentration of carbon in δ-ferrite is 0.1% at 1493ºC i.e. at temperature of the peritectic transformation. The crystal structure of δ-ferrite is BCC.
- **L** - Liquid solution of carbon in iron;
- **Cementite** – Iron carbide, intermetallic compound, having fixed composition Fe_3C. 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 cooing 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 δ-ferrite. Carbon content in δ-ferrite increases up to 0.1% in course solidification, and at 1493ºC remaining liquid phase and δ-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 stars 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 ºF (1147 ºC). 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 ºC, 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 723ºC. The eutectoid concentration of carbon is 0.8%.

When the temperature of an alloy reaches 723ºC, 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 α-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

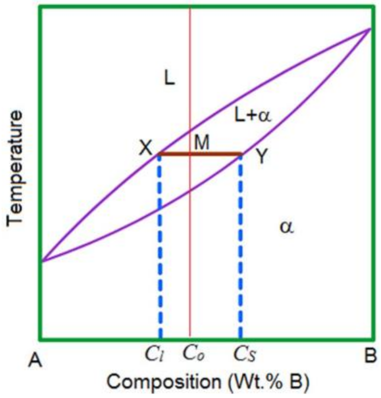
Lever rule derived from mass balance gives relative amounts of the co-existing phases. The tie-line is treated as a lever arm, with the fulcrum at the overall composition. For the arm to be horizontal, the weight to be hung at each end must be proportional to the arm length on the other side of the fulcrum.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

$$C_L = \frac{MY}{XY} \;, \qquad C_S = \frac{Uc}{UV} \;, \qquad C_S + C_L = 1$$

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 β phase is not appeared in the Fe-Fe_3C phase diagram?

2. Why A_{CM} line is steeper than A_3 line?

3. Why does proeutectoid phase (ferrite or cementite) form along austenite grain boundaries?

4. Compute the mass fractions of α 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?

6. Compute the mass fractions of proeutectoid ferrite and pearlite that form in an iron carbon alloy containing 0.35%C

7. The microstructure of an iron-carbon alloy consists of proeutectoid ferrite and pearlite; the mass fractions of these two microconstituents are 0.174 and 0.826, respectively. Determine the concentration of carbon in this alloy.

8. The mass fraction of eutectoid cementite in an iron-carbon alloy is 0.109. on the basis of this information, is it possible to determine the composition of the alloy? If so, what is its composition? If this is not possible, explain why.

9. α-ferrite and δ-ferrite, both contain BCC structure. Why the solubility limit varies from α-ferrite to δ-ferrite?

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

EXPERIMENT 5

MICROSTRUCTURAL ANALYSIS OF CARBON AND LOW ALLOY STEELS

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 carbon and low alloy 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

A sub-division of the major class and the percentage of the major alloying elements

Specifications	Types and classes
10XX	Non resulphurized carbon steels (Plain carbon steel)
11XX	Resulfurized carbon steels, (Free cutting steel)
12XX	Carbon steels, resulfurized and rephosphorized
13XX	Manganese steels
2XXX	Nickel steels
23XX	Nickel steels 3.50% Ni
25XX	Nickel steels 5.0% Ni
3XXX	Nickel chromium steels
30XX	Ni-Cr steels 0.7% Ni, 0.7% Cr
31XX	Ni-Cr steels 1.25% Ni, 0.6% Cr
32XX	Ni-Cr steels 1.75% Ni, 1.0% Cr
33XX	Ni-Cr steels 3.50% Ni, 1.50% Cr
40XX	Carbon-molybdenum steels
41XX	Chromium-molybdenum steels
43XX	Chromium-nickel-molybdenum steels
46XX	Nickel-moly steels 1.65% Ni, 0.25% Mo

48XX	Nickel-moly steels 3.25% Ni, 0.25% Mo
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

4% picral	4 gms picric acid 96 ml ethyl alcohol 5 ml zephiran chloride (wetting agent) per 75 ml of solution	For structures consisting of ferrite and carbides (and pearlite). For bainitic steels and tempered martensite Does not reveal ferrite grain boundaries For pearlite and carbides, usually etch for 20 s
4% picral + 2% nital	4 ml picric acid 96 ml ethyl alcohol 5 ml zephiran chloride 2 ml nitric acid (conc)	For ferrite plus carbide (pearlite) structures and ferrite plus bainite or martensite etch first in picral followed by nital, or mix the two solutions. Either technique produces good results.
Sodium metabisulfite	10 - 12 gms sodium metabisulfite 100 ml distilled water	Tint etch. Darkens martensite in quenched steels and dual phase steels
Beraha's reagent	10 gms sodium thiosulfate (anhydrous) 3 gms potassium metabisulfite 100 ml water	Tint etch. For ferrite grain boundaries
Klemm's reagent	50 ml water saturated with sodium thiosulfate + 1 gm potassium metabisulfite	Tint etch. Colors ferrite grains and ferrite in pearlite Etching time, 40–120 s

5. 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.

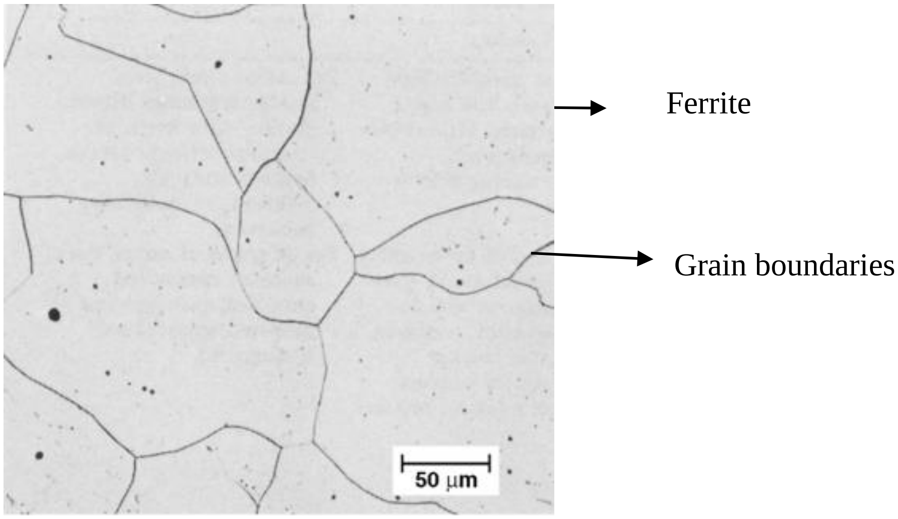


Fig.1: Microstructure of low carbon steel etched with nital

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

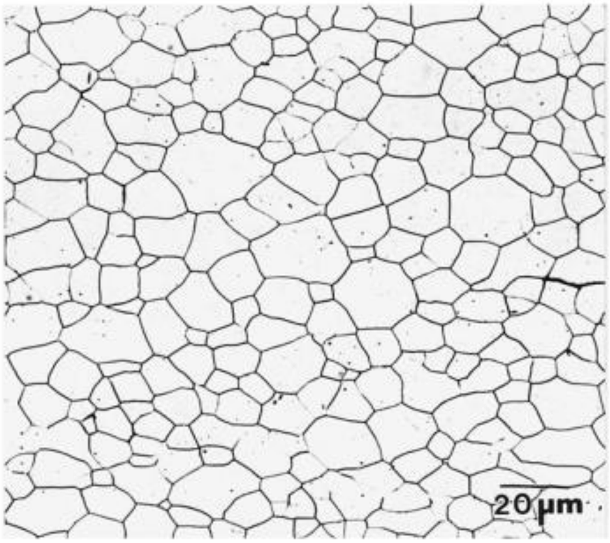


Fig. 2: Ferrite grains in a low-carbon (0.02% C) steel. Marshall's reagent.

Microstructure of steel containing 0.02- 0.4% C

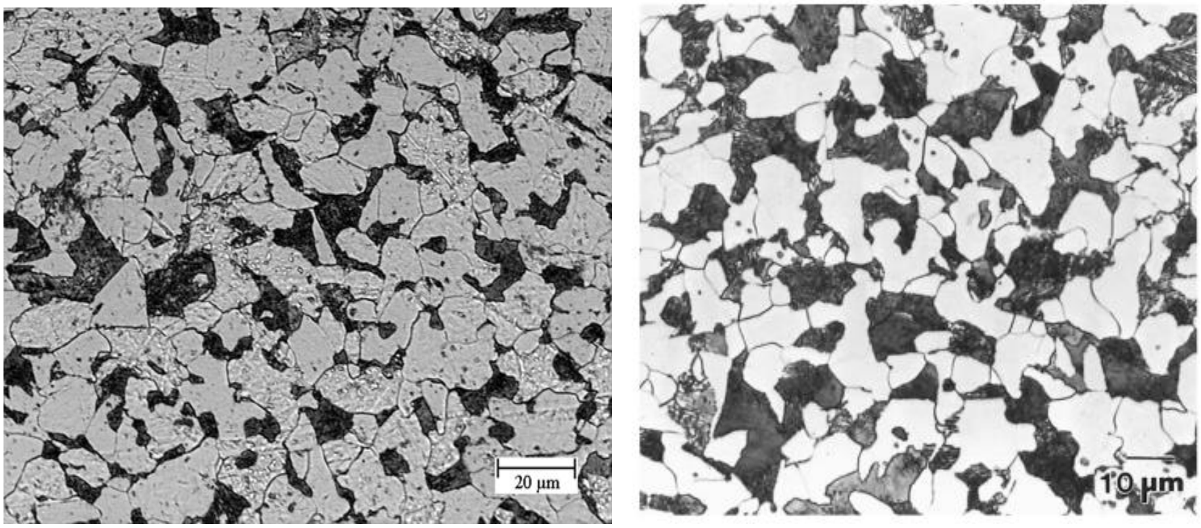


Figure 3. The microstructure of AISI 1040 steel showing ferrite and pearlite. The mean grain diameter is 26.173 µm. Etchant 2% Nital.

Microstructure of eutectoid steel containing 0.8% C

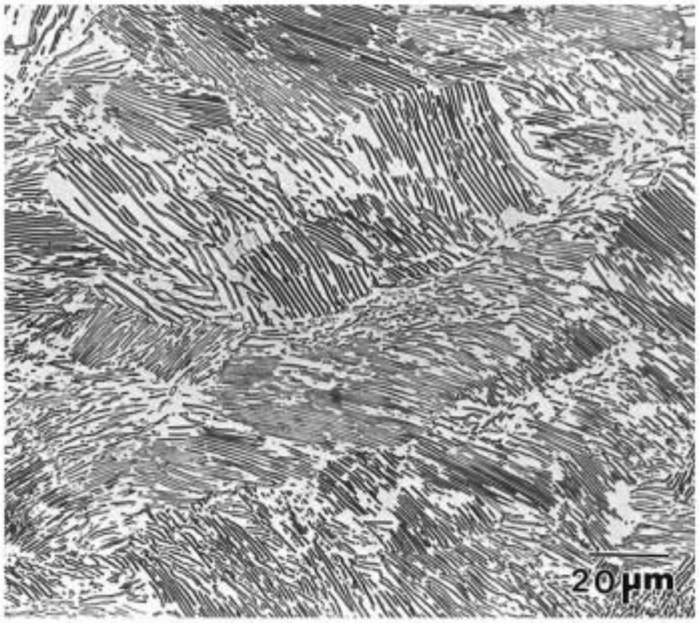


Figure 4. The microstructure of AISI/SAE 1080 steel showing pearlite etched with 4% Picral.

Hypereutectoid steel

Microstructure of High Carbon Steel containing >0.8% C

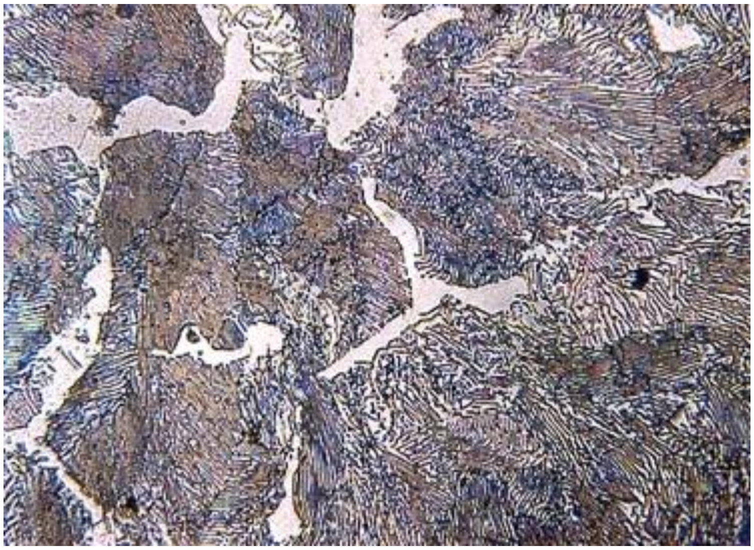


Figure 5: The microstructure of high carbon steel showing proeutectoid cementite around pearlite grains.

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 …………………………………………………………………….
……………………………………………………………………………………….....
……………………………………………………………………………………………….

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?

EXPERIMENT 6

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).

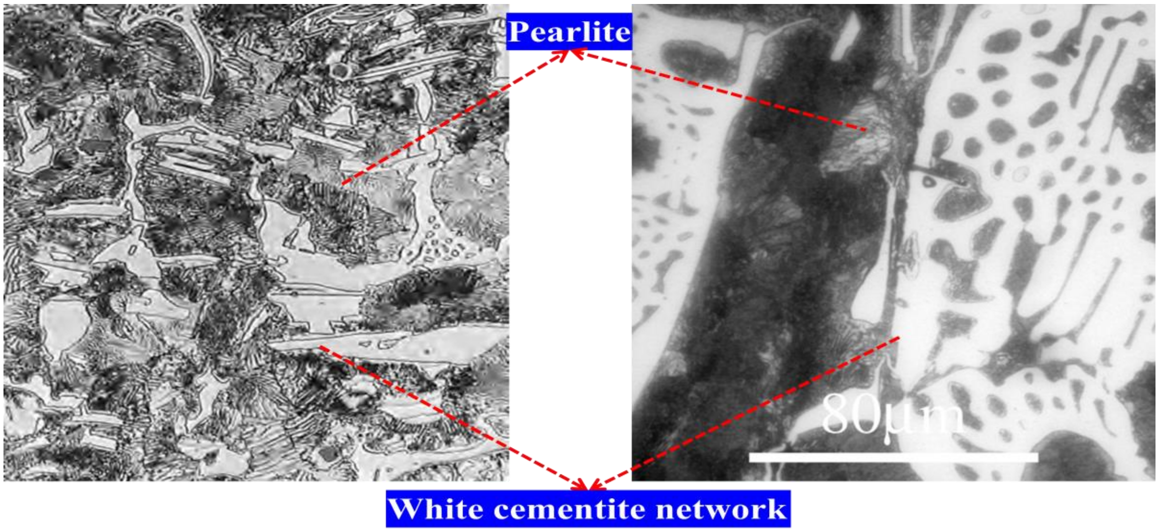
Types of cast iron

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

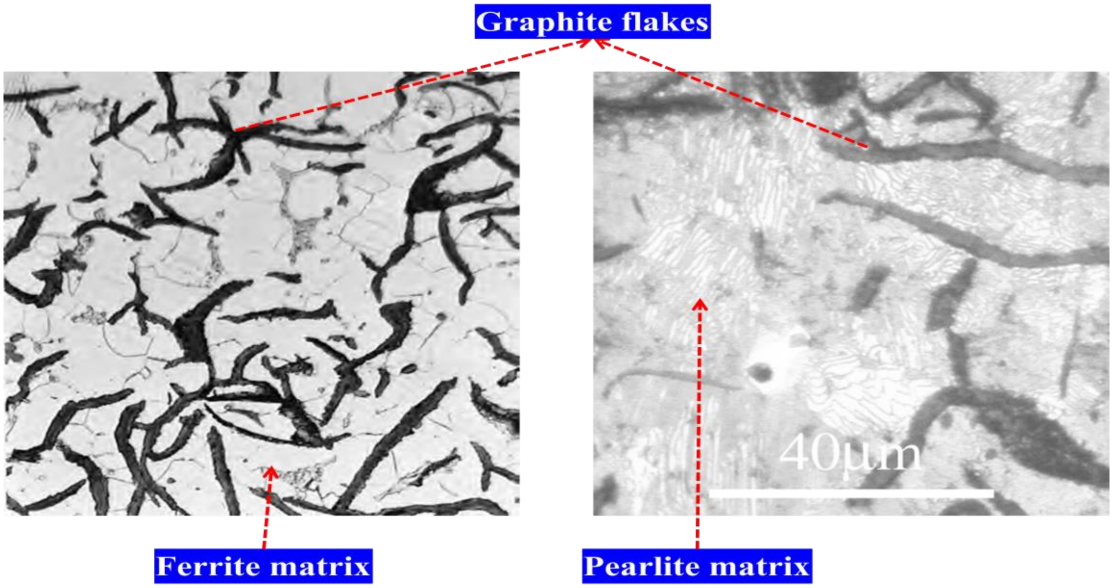
Table 1. Range of compositions for typical unalloyed Cast Irons

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

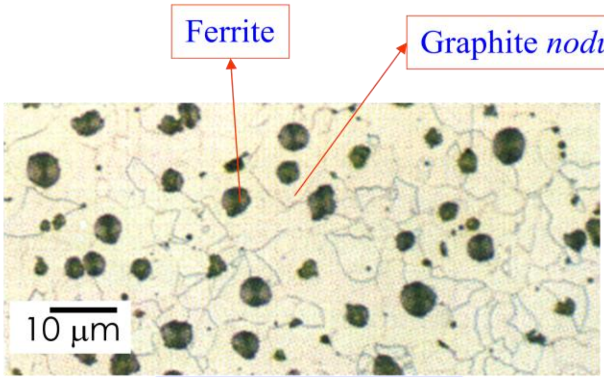
White Cast Iron



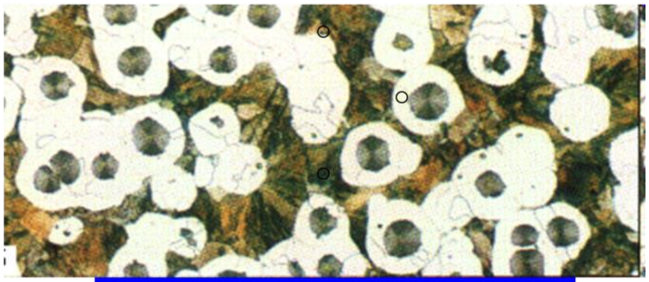
Grey Cast Iron



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



With Ferritic Matrix



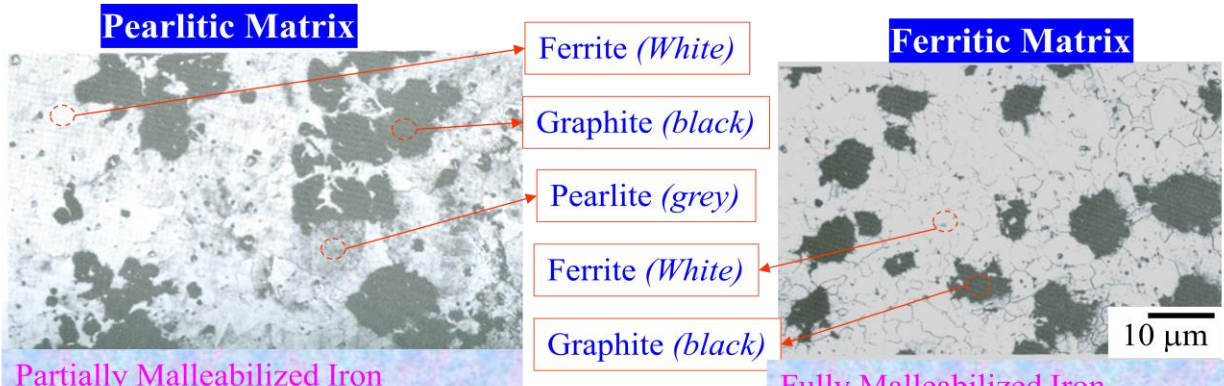
With (Ferrite + Pearlite) Matrix

With Pearlitic matrix

Malleable cast iron

Pearlitic Matrix

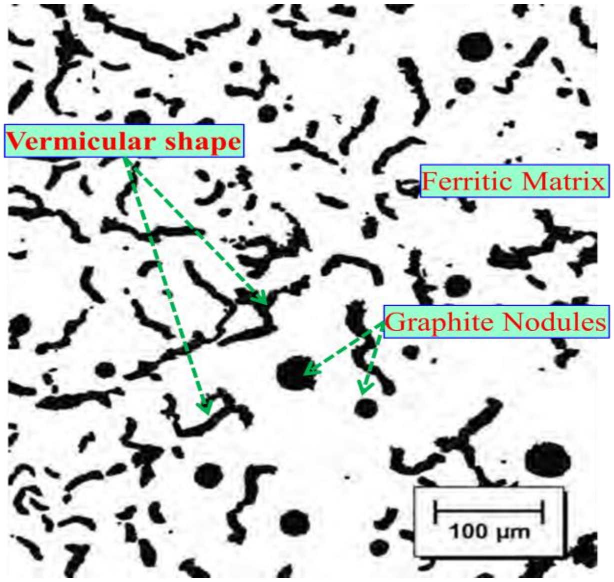
Ferritic Matrix

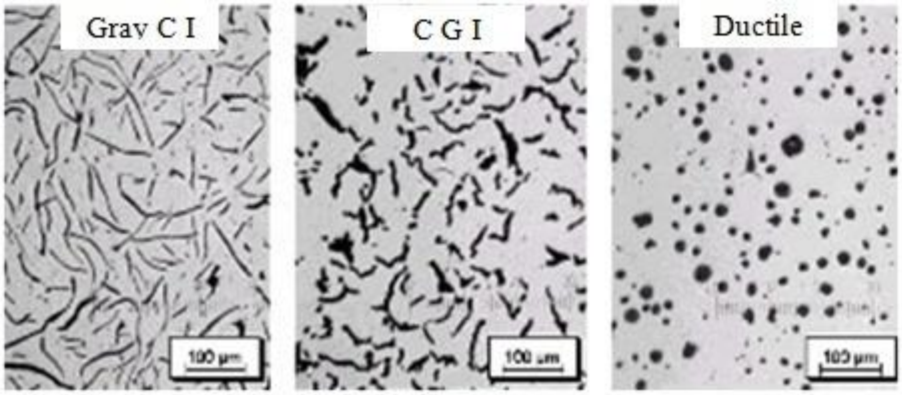


Partially Malleabilized Iron
→ Incomplete Ferritizing Anneal

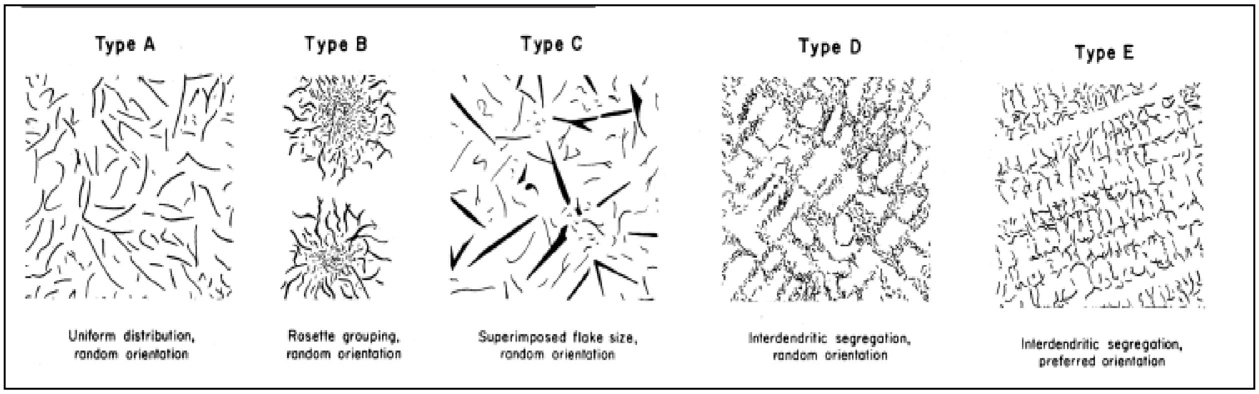
Fully Malleabilized Iron
→Complete Ferritizing Anneal

Compacted graphite cast iron

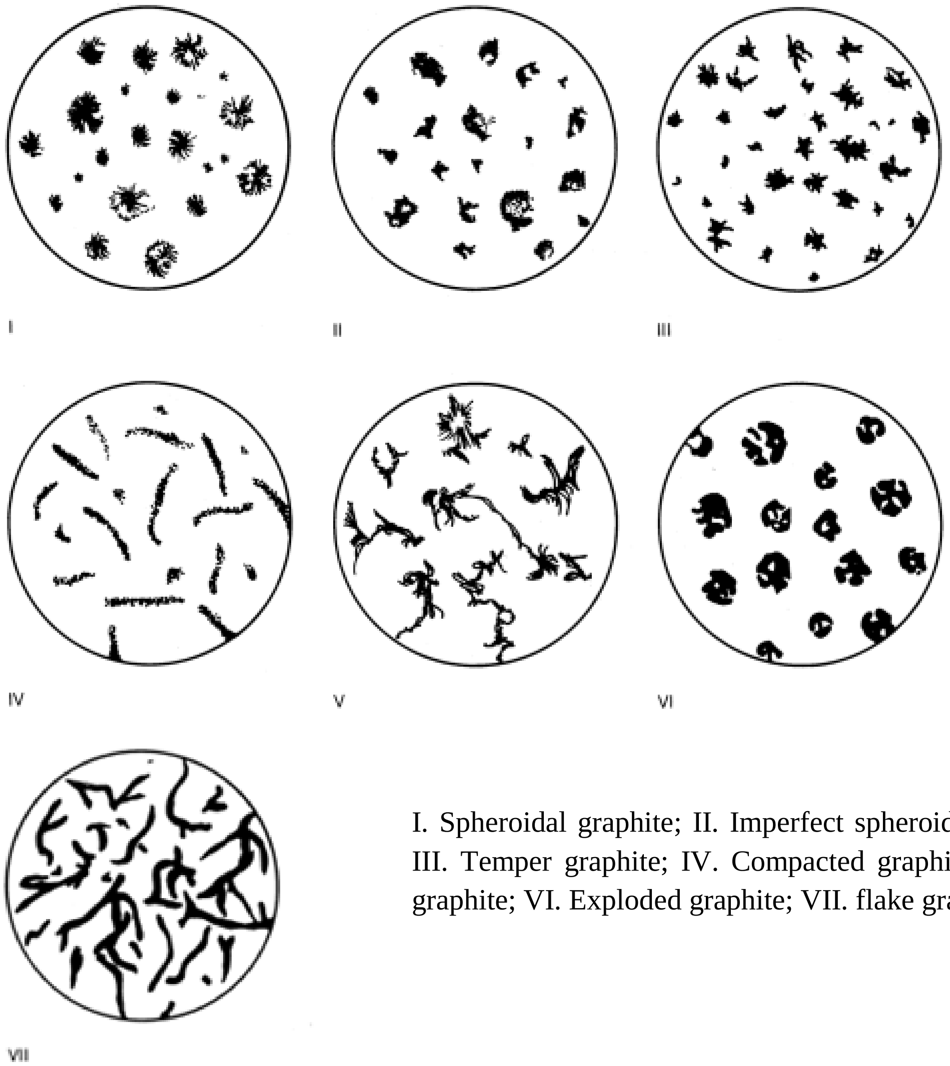




Types of Graphite flakes in gray iron



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.

8. Compare gray 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 7

MICROSTRUCTURAL ANALYSIS OF NON-FERROUS METALS

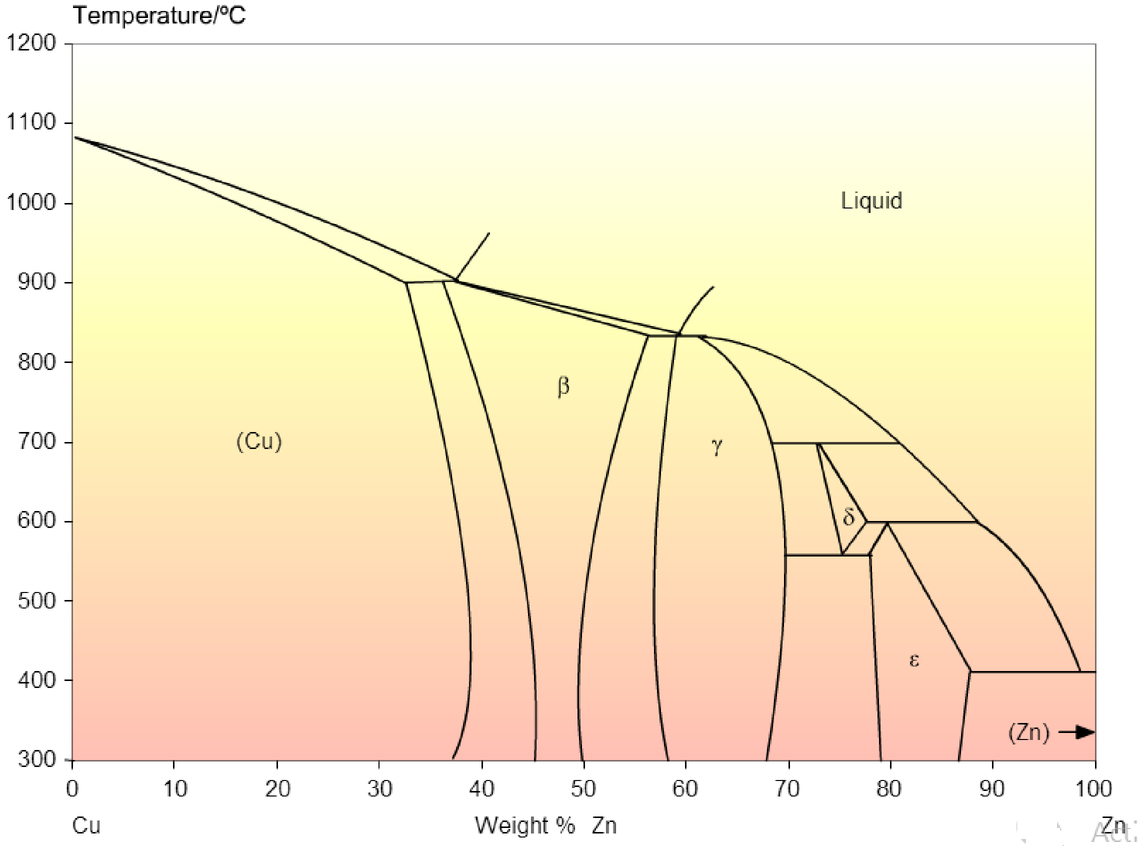
Objective

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

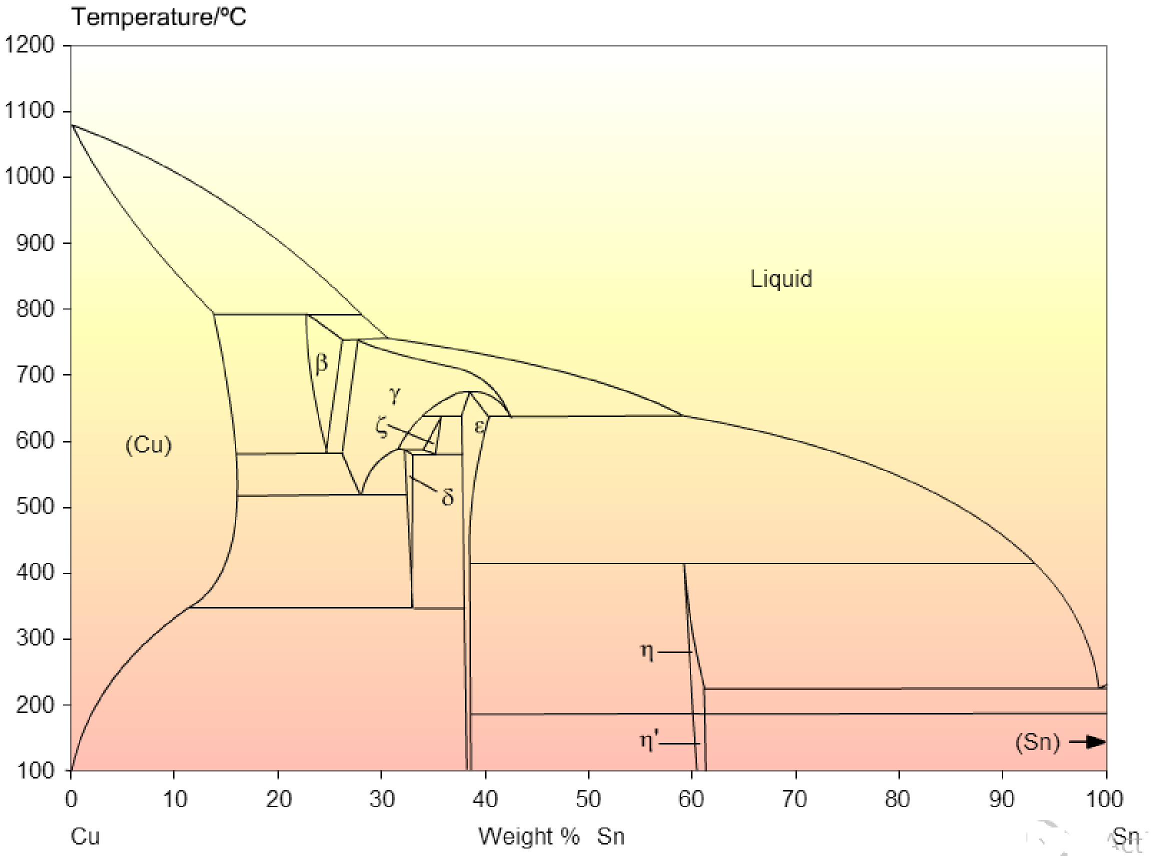
Theory

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

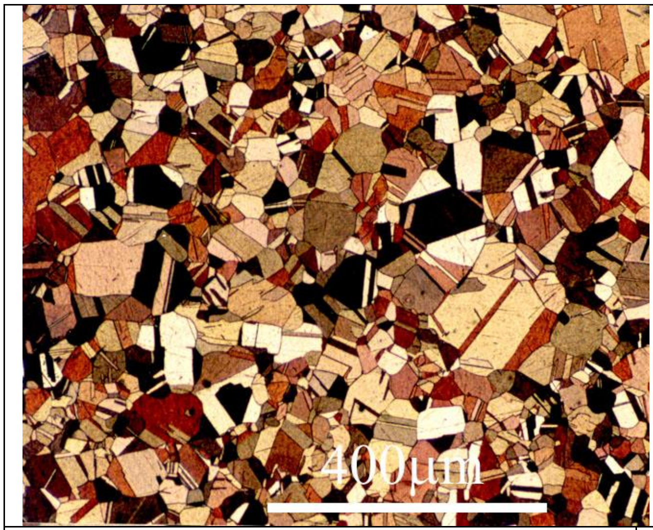
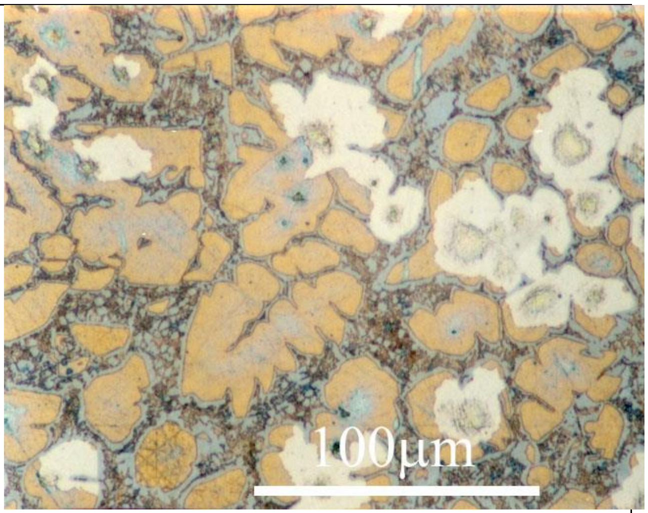
Observations



Identify the Phases, Temperatures & 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

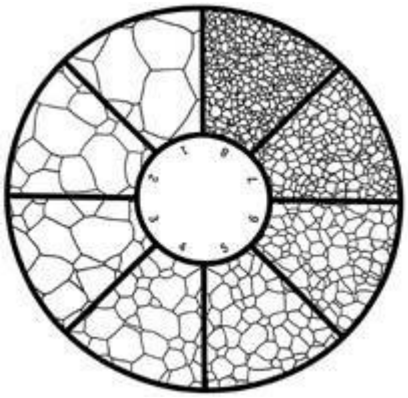
8. How is the core structure homogenised?

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

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

EXPERIMENT 8

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 = $\frac{1200\ mm^2}{250\times 250} = 0.0192\ mm^2$

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

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

$$n = \frac{30}{0.0192} = \frac{30\times 10^4}{192} = 1.5\times 10^4 = 1500$$

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”

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

$$G = -2.9542 + 1.421 \times 9.6158 = 10.76 \approx 11$$

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

iii. Jeffery's Planimetric Method

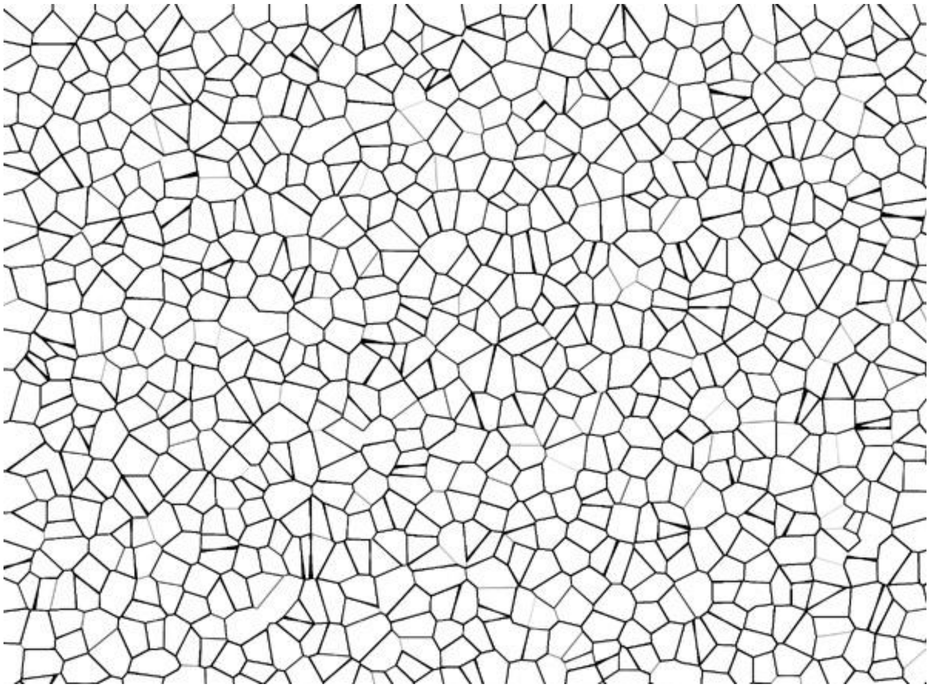
In this method a rectangle or a circle of known area commonly 5000 mm^2 is engraved or drawn, as the case may be, on the eye piece of the microscope or on the photomicrograph. The magnification which gives about 50 grains inside the circle or rectangle is used for the study. All the grains which intersect 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)

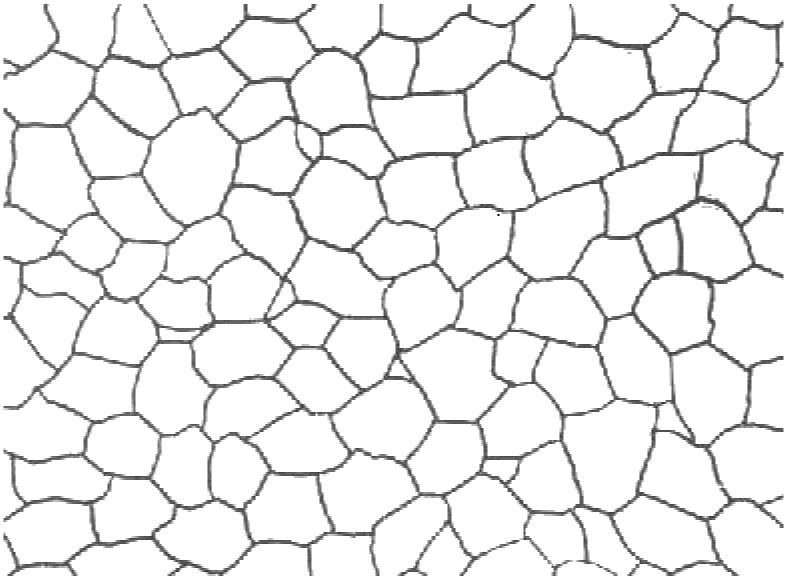
Magnification Used, X	**Jeffries Multiplier, k, to obtain Grains/mm^2**
1	0.0002
10	0.02
25	0.125
50	0.5
75	1.125
100	2.0
150	4.5
200	8.0
250	12.5
300	18.0
500	50.0
750	112.5
1000	200.0

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?

PROJECT

COLOR METALLOGRAPHY

Objective

To determined the colored microstructure for a given sample.

Theory

Refer the following article on “Color Metallography” by George F Vander Voort

Observation

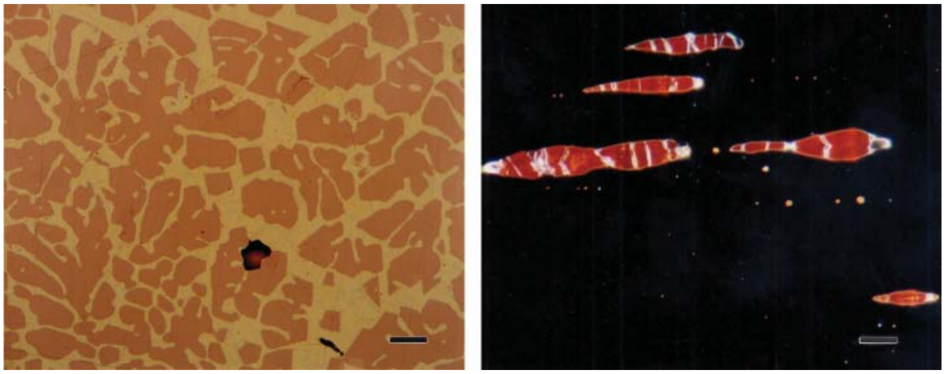
Paste the microstructure and write down the phases
Magnification used ____________ Etchant used ________________________________ *Phases present*: …………………………………….. ………………………………………………………………………………………………. Any other observation …………………………………………………………………………. …………………………………………………………………………………………….....

Paste the extra microstructures and/or write extra information (if any) related to "color metallography"

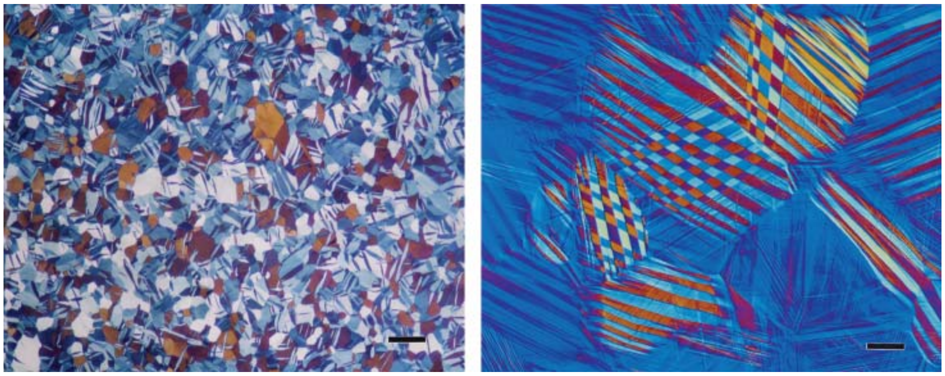
Color Metallography

George F. Vander Voort
Buehler Ltd, Lake Bluff, Illinois USA
george.vandervoort@buehler.com

Color has historically seen limited use in metallography, mainly due to the cost of film and prints and the difficulty and cost of reproducing images in publications. However, with the growth of digital imaging, capturing color images is much simpler and cheaper. Also, printing images in color is inexpensive for in-house reports, and can be distributed cheaply on CDs, although reproduction in journals is still expensive. Color does have many advantages over black and white. First, the human eye is sensitive to only about forty shades of gray from white to black, but is sensitive to a vast number of colors. Tint etchants reveal features in the microstructure that often cannot be revealed using standard black and white etchants. Color etchants are sensitive to crystallographic orientation and can reveal if the grains have a random or a preferred crystallographic texture. They are also very sensitive to variations in composition and residual deformation. Further, they are usually selective to certain phases and this is valuable in quantitative microscopy.



Figures 1 (left) and 2 (right) showing natural reddish-purple color of the AuAl$_2$ intermetallic (left) in bright field and cuprous oxide's characteristic ruby red color in dark field illumination (tough-pitch arsenical copper specimen). The magnification bars are 50 and 10 µm, respectively.



Figures 3 (left) and 4 (right): Grain structure on high-purity Zr (left) that was hot worked and cold drawn (note mechanical twins) and viewed in polarized light and of Spangold (Au – 19Cu-5Al) that was polished and cycled through the shape-memory effect to produce martensite and Nomarski differential interference illumination was used to image the surface upheaval due to the shear reaction at the free surface. The magnification bars are 100 and 50 µm, respectively.

The use of color in metallography has a long history with color micrographs published over the past eighty-some years. Examples of natural color in metals are rare (Figure 1). Gold and copper exhibit yellow color under bright field illumination. Color can be produced using optical methods, as in dark field illumination (Figure 2), polarized light (Figure 3) and differential interference contrast illumination (Figure 4). The microstructure of metals with non-cubic crystal structures can be examined without etching using polarized light but color is not always observed. The specimen must be prepared completely free of residual damage for color to be observed, and even then, some non-cubic metals still exhibit little color. However, many metals and alloys can be etched with reagents that deposit an interference film on the surface that creates color in bright field illumination. If it is difficult to grow such a film to the point where the color response is excellent, the color can be enhanced by examination with polarized light, perhaps aided with a sensitive tint filter (also called a lambda plate or first-order red filter).

Anodizing

There are a number of electrolytic etching reagents that can be used to produce color. Second-phase constituents can be colored and viewed with bright field. Anodizing aluminum specimens with Barker's reagent, or similar solutions, does not produce an interference film, as color is not observed in bright field. This procedure produces fine etch pitting on the surface. The grain structure can be seen in black and white in polarized light, and in color if a sensitive tint plate is added. Figure 5 show an example of anodizing to reveal the grain structure of super-pure aluminum.

Color Etching

Many metals etched with standard reagents to reveal the grain boundaries often yield only a high percentage of the boundaries, rather than all of the boundaries. Color etchants, however, reveal the grain structure completely. In the case of metals with annealing twins, it can be very difficult to rate the grain size when a standard etchant reveals a portion of the grain and twin boundaries. In fact, it can be quite difficult to make a precise measurement of the grain size, even manually, with such a specimen, as distinguishing between grain and twin boundaries (the latter must be ignored in the measurement), is not simple. However, with a color etched microstructure it is relatively easy to separate grain from twin boundaries, at least manually. Further, the films grow as a function of crystal orientation. Therefore, one can detect any preferred crystallographic orientation by the narrowness of the color range present. If a wide range of colors is present in a random pattern, the crystal orientation is

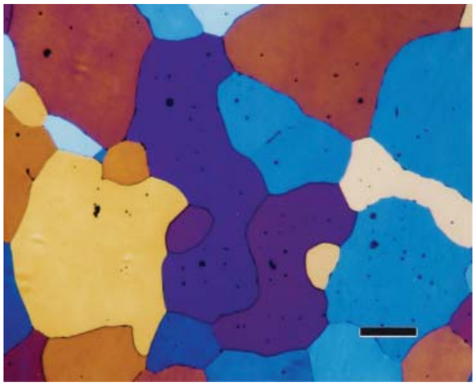
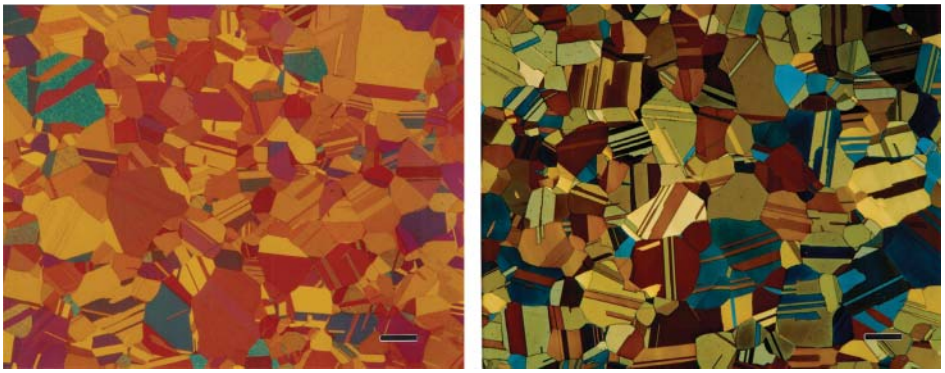
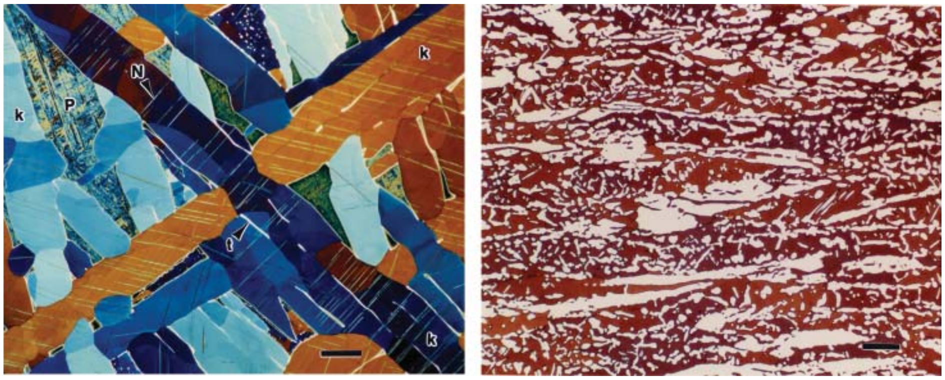


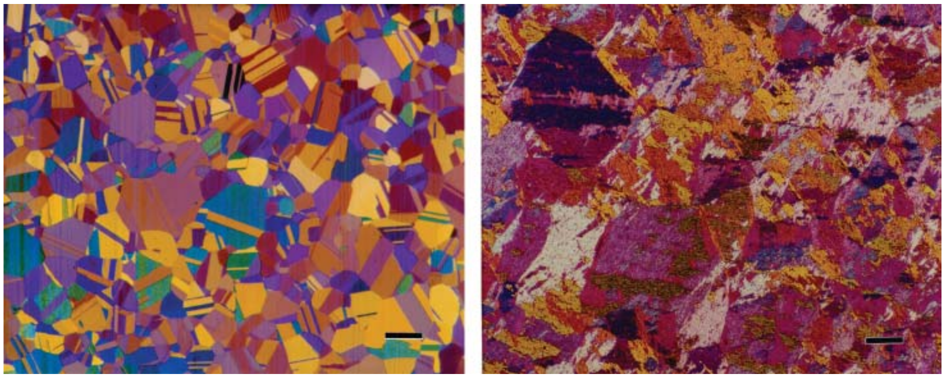
Figure 5. Super-pure aluminum anodized with Barker's reagent (30 V dc, 2 minutes). The magnification bar is 200 µm long.



Figures 6 and 7: FCC twinned grain structure of cartridge brass, Cu – 30% Zn, after cold reduction by 50% and full annealing, tint etched with Klemm's I (left) and Klemm's III (right) reagents and viewed with polarized light plus sensitive tint. Magnification bars are 200-µm long.



Figures 8 and 9: Fine octahedrite grain structure of the Gibeon meteorite (left) revealed with Beraha's reagent (100 mL water, 10 g $Na_2S_2O_3$ and 3 g $K_2S_2O_5$) and ferrite in 7 Mo PLUS duplex stainless steel plate revealed using Beraha's reagent (85 mL water, 15 mL HCl, 1 g $K_2S_2O_5$). The magnification bars are 500 and 50 µm long, respectively.



Figures 10 and 11: FCC twinned grain structure of heading quality Custom Flo 302 stainless steel revealed using Beraha's B1 reagent and lath martensite grain structure of over-austenitized (1093 °C) AerMet 100 ultra-high strength steel revealed using 10% sodium metabisulfite. Both viewed with polarized light plus sensitive tint. The magnification bars are 100 µm long.

random. If a narrow range of colors is present in the grains, then a preferred orientation is present. Tint etch compositions are given at the end of the article.

Specimen preparation must be better when using color methods compared with black and white methods because the epitaxially grown films are sensitive to residual preparation-induced damage that was not removed. This level of preparation is required in image analysis work and can be easily obtained by a knowledgeable metallographer with the proper equipment. Electrolytic polishing is not required to get damage-free surfaces.

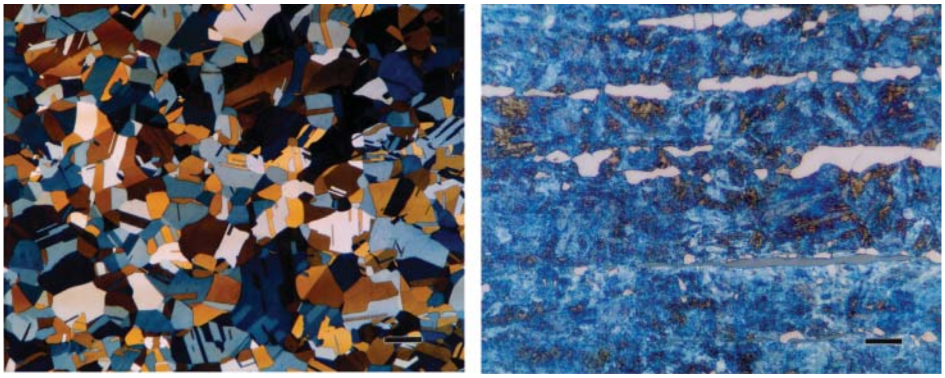
The most common tint etchants are those that deposit a sulfide-based interference film on the specimen. These are the best-known tint etches and usually the easiest to use. Klemm and Beraha have developed the most widely used sulfide-based tint etchants using sodium thiosulfate, $Na_2S_2O_3$, and potassium metabisulfite, $K_2S_2O_5$. Klemm's I, II, III (Figures 6 and 7) and one of Beraha's reagents utilize both ingredients (Figure 8), while Beraha recommends a range of HCl concentrations used with potassium metabisulfite (Figure 9) for etching a variety of iron-based alloys. These etchants can be used to color ferrite and martensite in cast iron, carbon and low-alloy steels. The HCl-based reagents vary widely in concentration and can be used to color the grain structures of stainless steels (Figure 10), Ni-based and Co-based alloys. Sodium metabisulfite has been used in a number of concentrations, from about 1 to 20 g per 100 mL water, and is a safe, reliable, useful color etch for irons and steels (Figure 11).

Beraha also developed etchants based upon sulfamic acid, a weak organic acid, which has not been used much, although they are quite useful, reliable and easy to employ. The sulfamic acid-based reagents are applicable to cast iron, low-carbon and alloy steels, tool steels, and martensitic stainless steels (Figure 12). Beraha also developed two rather specialized tint etches that deposit cadmium sulfide (Figure 13) or lead sulfide (Figure 14) films on the surfaces of steels and copper-based alloys. These two etchants are quite useful, although tedious to make. His CdS reagent is useful for carbon and alloy steels, tool steels, and ferritic, martensitic and precipitation hardenable stainless steels, while the PbS reagent does an excellent job on copper-based alloys and can be used to color sulfides in steels white (the specimen is pre-etched with nital and the etch colors the darkened matrix, so that the white sulfides are visible).

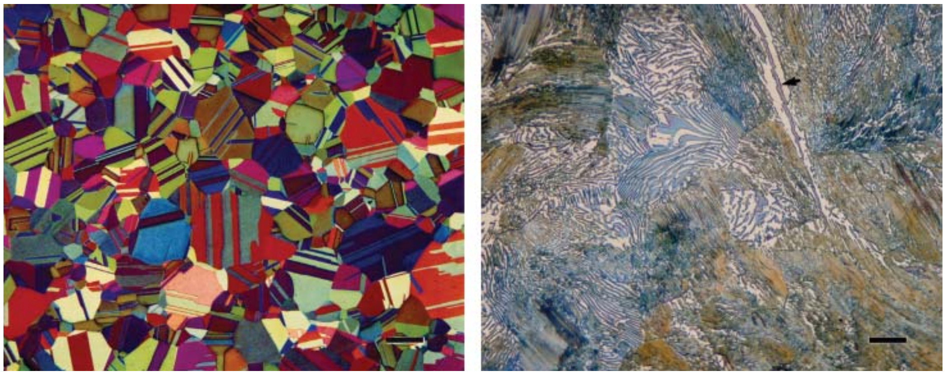
Beraha also developed two tint etchants that utilize molybdate ions in nitric acid. They color cementite in steels (Figure 15). He also developed tint etchants that deposit elemental selenium on the surface of steels (Figure 16), nickel-based alloys and copper-based alloys (Figure 17).

There are a number of other tint etchants that have been developed by a variety of metallographers. Lichtenegger and Blöch, for example, developed an unusual reagent that will color austenite (Figure 18) in duplex stainless steels, rather than ferrite (as nearly all others do).

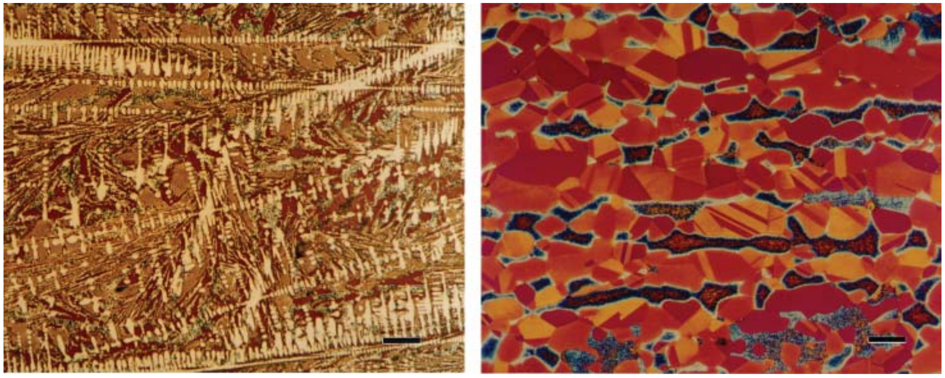
Weck developed a number of tint etchants, while utilizing many of them in her research. Several were developed to color aluminum (Figure 19) or titanium alloys (Figure 20). In each case, it is easier to develop good color with the cast alloys than with the wrought alloys. Two etchants have been found useful for coloring theta phase, $AlCu_2$, in Al-Cu alloys; Lienard developed one of the



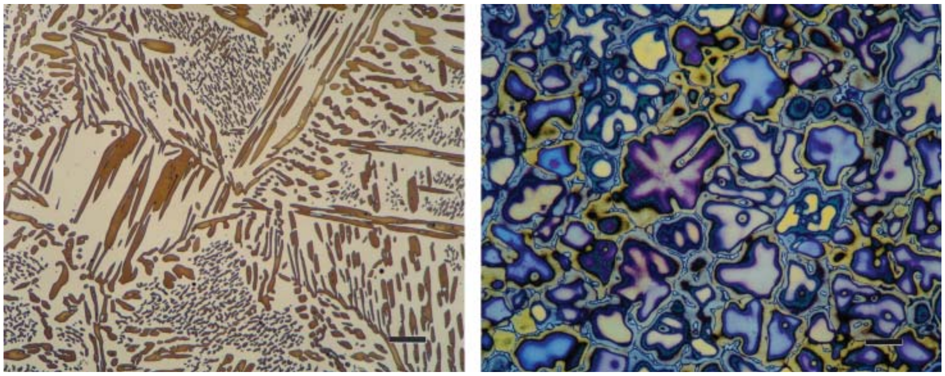
Figures 12 and 13: Twinned FCC grain structure in Fe-39% Ni revealed by Beraha's sulfamic acid reagent (left) and tempered martensite grain structure of Carpenter Project 70 type 416 martensitic stainless steel revealed with Beraha's CdS reagent (right). The white grains are delta ferrite and the gray inclusions are sulfides. Viewed with polarized light plus sensitive tint. The magnification bars are 100 and 200 µm long, respectively.



Figures 14 and 15: Cartridge brass micrograph shown in Figures 6 and 7 tint etched with Beraha's PbS reagent (left) and cementite in a hot rolled Fe-1% C binary alloy colored with Beraha's sodium molybdate reagent (right). Magnification bars are 200 and 20 µm, respectively.



Figures 16 and 17: Cementite in the chill cast surface of gray iron etched with Beraha's selenic acid reagent for cast iron (left) and twinned FCC alpha phase and beta phase (mottled and outlined) in Cu-40% Zn revealed using Beraha's selenic acid reagent for copper alloys. Magnification bars are 50 and 20 µm long, respectively.



Figures 18 and 19: Austenite colored in ASTM A890 Grade 5A cast duplex stainless steel with the LB1 reagent (left) and the cast grain structure of 206 aluminum revealed using Weck's reagent for Al alloys (right). The magnification bars are 100 and 50 µm long, respectively.

easiest to use. Several color etchants have been developed for molybdenum (Figure 21) and for tungsten. Details on these etchants can be found in [1].

Conclusions

The examples shown have demonstrated the great value of color and tint etching for examining microstructures of metals. Solutions exist to develop color with most commercial alloy systems. The examples clearly demonstrate the value of these reagents in revealing the grain structure fully, even for the most difficult to etch specimens. Further, they are selective in nature that can be quite useful for quantitative metallographic studies. Tint etchants reveal segregation very clearly and either EDS or WDS can be performed on a tint-etched surface without any problems from the interference surface layer. ■

Reference

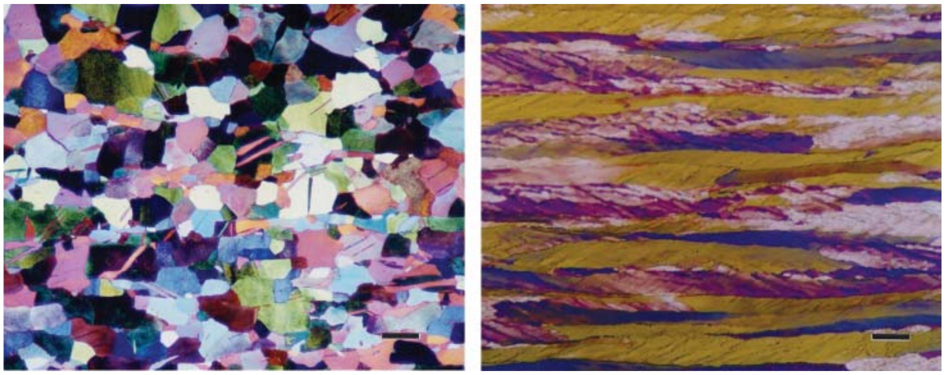
[1]. G. F. Vander Voort, Metallography: Principles and Practice, McGraw-Hill Book Co., NY, 1984 and ASM International, Materials Park, Ohio, 1999.

Appendix

Etch Compositions

- Klemm's I: 50 mL stock solution, 1 g $K_2S_2O_5$ (stock solution is water saturated with $Na_2S_2O_3$
- Klemm's III: 5 mL stock solution, 45 mL water, 20 g $K_2S_2O_5$ (stock solution as for Klemm's I)
- Beraha's 10/3 reagent: 10g $Na_2S_2O_3$, 3g $K_2S_2O_5$ and 100 mL water
- Beraha's BI: 100 mL stock solution (1000 mL water, 200 mL HCl, 24 g NH_4FHF) plus 0.1 – 0.2 g $K_2S_2O_5$ for martensitic stainless steel and 0.3 – 0.6 g $K_2S_2O_5$ for austenitic and ferritic stainless steels.
- Beraha's sulfamic acid reagent No. III: 100 mL water, 3 g $K_2S_2O_5$, 2 g NH_2SO_3H (two other similar compositions were published) for carbon and alloy steels.
- Beraha's sulfamic acid reagent No. IV: 100 mL water, 3 g $K_2S_2O_5$, 1 g NH_2SO_3H, 0.5 – 1 g NH_4FHF for high-Cr tool steels and martensitic stainless steels.
- Beraha's CdS and PbS reagents: CdS stock solution: 1000 mL water, 240 g $Na_2S_2O_3 \cdot 5H_2O$, 20-25 g cadmium chloride (or cadmium acetate), 30 g citric acid; PbS stock solution: 1000 mL water, 240 g $Na_2S_2O_3 \cdot 5H_2O$, 30 g citric acid, 24 g lead acetate.

Mix each in order given. Age solutions in a dark bottle, in darkness, for 24 hours before use. To use the CdS reagent, filter excess precipitates from about 100 mL of solution. CdS regent will color microstructure of a wide



Figures 20 and 21: Grain structure of as-rolled CP Ti (ASTM F67, Grade 2) containing mechanical twins (left) etched with modifided Weck's reagent and cold rolled pure molybdenum (right) colored with the reagent developed by Oak Ridge National Laboratory. Magnification bars are 100 and 20 μm long, respectively.

range of irons and steels in 20 to 90 s. To use the PbS reagent, do not filter excess precipitates when you pour about 100 mL of the stock solution for use. The PbS reagent will color the microstructure of most copper-based alloys. It will also etch steel microstructures.

- Beraha's sodium molybdate reagent: Stock Solution: 1000 mL water, 10 g $Na_2MoO_4 \cdot 2H_2O$. Pour off about 100 mL of the stock solution and add HNO_3 to bring the pH to 2.5 – 3.0. For steels, add small amounts of NH_4FHF to control coloration (none for cast iron). Colors cementite.
- Beraha's selenic acid reagent for cast iron: 100 mL ethanol, 2 mL HCl, 1 mL selenic acid.
- Beraha's selenic acid reagent for Cu alloys: 300 mL ethanol, 2 mL HCl, 0.5-1 mL selenic acid
- Lichtenegger and Blöch LB1 reagent: of 20 g of ammonium bifluoride, NH_4FHF, and 0.5 g potassium metabisulfite, $K_2S_2O_5$, dissolved in 100 mL water (use hot water). Etch at 25-30 ^{o}C.
- Weck's reagent for Al: 100 mL water, 4 g $KMnO_4$ and 1 g NaOH
- Modified Weck's reagent for Ti: 100 mL water, 25 mL ethanol and 2 g ammonium bifluoride. The original formula specified 50 mL of ethanol, but that produces etch artifacts.
- ORNL tint etch for Mo: 70 mL water, 10 mL sulfuric acid and 20 mL hydrogen peroxide (30% conc.). The specimen is immersed for 2-3 minutes. Swab etching produces a flat etched microstructure (no color).