

Lab Manual

PHYSICAL METALLURGY LABORATORY



Department of Metallurgical and Materials Engineering

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EXPERIMENT 1

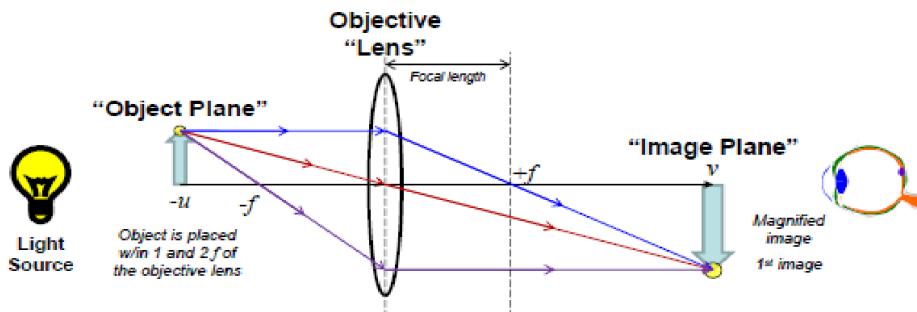
METALLURGICAL MICROSCOPE

Objective: To study the components and functions of a 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.



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:

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

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 nosepiece 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.



To report:

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 can one 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 $20 \mu\text{m}$, what is the drawing magnification? For this drawing magnification, how long would a scale bar have to be to represent a distance of $10 \mu\text{m}$?
10. What is the significance of this experiment? How is it related to your course of study?

Experiment 2

Introduction of Metallographic Specimen Preparation

Objective:

1. Metallographic Sample Preparation.
2. To study the importance of the various steps in sample preparation.
3. To understand the need of mounting, polishing and etching.

Introduction:

Metallography is the study of the microstructure of metals and alloys by means of microscopy. It is an art and science of preparing, interpreting, and analyzing microstructures in materials, to better understand materials behavior and performance. The method is used for the evaluation of metallic materials in the various industries, including the aerospace industry, the automotive industry and parts of the construction industry. It is also used for process control including the examination of defects that appear in finished or partly finished products, as well as the studies of parts that have failed during service. It is an important branch of Metallurgical Engineering.

A well prepared metallographic specimen is:

- A representative sample
- Sectioned, ground and polished so as to minimize disturbed or flowed surface metal caused by mechanical deformation, and thus to allow the true microstructure to be revealed by etching.
- Free from polishing scratches and pits and liquid staining
- Flat enough to permit examination by optical microscope or SEM

Various steps involved in the specimen preparation, and the significance of each step:

Many steps in the preparation of metallographic specimens described here are also applicable in other types of metallographic studies, such as electron microscopy, micro hardness testing, quantitative measurement of constituents of structures, and electron microprobe analysis. Preparation of metallographic specimens generally requires five major operations:

- a. Sectioning
- b. Mounting (which is necessary when the sample cannot be held properly due to its

shape and/or size, while polishing)

- c. Grinding
- d. Polishing
- e. Etching

These operations are listed below in the order they are performed

Sectioning

Separate test pieces or coupons attached to castings or forgings should be designed so that a minimum of sectioning is required for producing metallographic specimens. Sectioning becomes necessary when studying parts that have failed in service where specimens have to be taken from a large block of material. Therefore, metallographic studies of such samples often involve more than one sectioning operation.

Many metallographic studies require more than one specimen. For example, a study of deformation in wrought metals usually requires two sections- one perpendicular to, and the other parallel to, the major axis of the direction of deformation. Failed parts may best be studied by selecting a specimen that intersects the origin of the failure, if the origin can be identified on the surface. Depending on the type of failure, it may be necessary to take several specimens from the area of failure and from adjacent areas.

Mounting of Specimens

The primary purpose of mounting is to make it easier to handle specimens of arbitrary shape and/or small sizes during various steps of metallographic sample preparation and examination. A secondary purpose is to protect and preserve extreme edges or surface defects during metallographic preparation. Specimens may also require mounting to accommodate various types of automatic devices used in metallographic laboratories or to facilitate placement on the microscope stage. An additional benefit of mounting is the identification of the sample (name, alloy number or laboratory code number) without damaging the specimen.

Compression mounting: It is the most common mounting method, which involves molding around the metallographic specimen by heat and pressure using the molding materials such as Bakelite, Diallyl Phthalate resins, and acrylic resins. Bakelite and Diallyl phthalate are thermosetting, and acrylic resins are thermoplastic.

Not all materials or specimens can be mounted in thermosetting or thermoplastic mounting. The heating cycle may cause changes in the microstructure, or the pressure may cause delicate specimens to collapse or deform. The size of the selected specimen may be too large to be accepted by the available mold sizes. These difficulties are usually overcome by cold mounting.

Cold Mounting requires no pressure and little heat, and is a mean of mounting large numbers of specimens more rapidly than possible by compression mounting. Epoxy resins are most widely used cold mounting materials. They are hard, and adhere tenaciously to most metallurgical, mineral and ceramic specimens.

Grinding

Grinding is a most important operation in specimen preparation. During grinding, the operator has the opportunity of minimizing mechanical surface damage that can be removed by subsequent polishing operations. Even if sectioning is done in a careless manner resulting in a severely damaged surface, the damage can be eliminated by prolonged grinding. However, prolonged grinding should be avoided since it might lead to excess heating or surface damage.

FEPA (Europe)		ANSI/CAMI (US)		
Grit Number	Micron	Grit Number	Micron	Emery Grit
P60	269	60	268	
P80	201	80	188	
P100	162	100	148	
P120	127	120	116	
P180	78	180	78	3
P240	58.5	220	66	2
P280	52.2	240	51.8	
P320	46.2			
P360	40.5	280	42.3	1
P400	35	320	34.3	0
P500	30.2	360	27.3	
P600	25.8			
P800	21.8	400	22.1	00
P1000	18.3	500	18.2	000
P1200	15.3	600	14.5	
P1500	12.6	800	12.2	0000
P2000	10.3	1000	9.2	
P2500	8.4	1200	6.5	
P4000*	5.0*			

The grit size numbering systems differ above 180 grit [P180], but equivalent sizes can be determined using Table

Grinding is accomplished by abrading the specimen surface through a sequence of operations using progressively finer abrasive grits. Grit sizes ranging from 60 mesh to 150 mesh are usually regarded as coarse abrasives, and grit sizes ranging from 180 mesh to 600 mesh as fine abrasives. Grinding should commence with the coarse grit size for making the initial flat surface and remove the effects of sectioning. Hack-sawed, band-sawed, or other rough surfaces usually require abrasive grit sizes in the range of 80 to 150 mesh. Grinding should be done sequentially starting from the coarser one to finer one. A satisfactory grinding sequence might involve grit sizes of 180, 240, 400 and 600 mesh. The purpose of grinding is to remove the oxide layer or damaged layer or uneven surfaces that might have formed during the last sectioning operation. However, the depth of cold worked metal is roughly inversely proportional to the hardness of the specimen and may be 10 to 50 times the depth of penetration of the abrasive particle.

To ensure the complete elimination of the previous grinding scratches found by visual inspection, the direction of grinding must be changed by 90° from the one stage of grinding to the next stage. In addition, microscopic examination of the various ground surfaces during the grinding sequence may be worthwhile in evaluating the effect of grinding. Each ground surface should have scratches that are clean-cut and uniform in size, with no evidence of previous grinding scratches. Cleaning before going to the next stage grinding is always helpful.

Grinding Mediums

Grinding media are silicon carbide (SiC), aluminium oxide (Al_2O_3), emery ($\text{Al}_2\text{O}_3\text{-Fe}_3\text{O}_4$), and diamond particles. All except diamonds are generally bonded to paper or cloth backing material of various weights in the form of sheets, disks, and belts of various sizes. The abrasives may be used also in the powder form by charging the grinding surfaces with loose abrasive particles.

Automatic grinding, as the name implies, is done without hand assistance. All automatic grinding devices use lap surfaces on which paper-based disks are placed or abrasive powder is charged. The lap is either a rotating or a vibrating disk, where the use of the latter is described as vibratory grinding.

Polishing

Polishing is the final step in producing a surface that is flat, scratch free, and mirror-like in appearance. Such a surface is necessary for subsequent accurate metallographic interpretation, both the qualitative and quantitative. The polishing technique used should not introduce extraneous structure, such as disturbed metal, pitting, dragging out of inclusions, and staining.

Mechanical Polishing: Mechanical polishing is frequently used to describe the various final polishing procedures involving the use of cloth-covered laps and suitable polishing abrasives, basically Al₂O₃ slurry. The laps have either a rotating or a vibrating motion, and the specimens are held by hand, held mechanically, or merely confined within the polishing area. Polishing should be done in a relatively dust-free area, preferably removed from the area for sectioning, mounting and rough grinding. Any contamination of a polishing lap by abrasive particles carried over from preceding operations or by dust, dirt or other foreign matter in the air cannot be tolerated. Carryover as a result of improper cleaning between final polishing steps is another prime source of contamination. It is just as important for the operator to wash his/her hands meticulously as it is for him/her to remove all traces of polishing abrasives from the specimen before proceeding to the next finer polishing operation.

Electrolytic Polishing: Even with the most careful mechanical polishing, some disturbed metal, even very small amounts, will remain after preparation of a metallographic specimen. This is no problem if the specimen is to be etched for structural investigation because etching is usually sufficient to remove the slight layer of disturbed metal. However, if the specimen is to be examined in the as polished condition, or if no surface disturbance can be tolerated, then either electrolytic polishing or chemical polishing is preferred. The basic principle involved in this technique is anodic dissolution of the surface of the sample which results in a levelling and brightening of its surface.

Electropolishing does not disturb any metal on the specimen surface, and therefore, ideally suited for the metallographic preparation of soft metals, most single phase alloys, and alloys that work harden readily. The disadvantages of electro polishing include preferential attack in multiphase alloys caused by differences in electrical potential between phases. Proper choice of electrolyte and operating conditions will minimize these disadvantages.

Etching

Metallographic etching is used to reveal particular structural characteristics of a metal. This is essential since these structural characteristics are not visible in the polished mirror-like surface in the metal. It can be used for phase identification, for dislocation density calculation (etch pitting), and for orientation studies. The principle of etching multiphase alloys is based on the preferential attack (different rates of electrochemical dissolution of phases in the etchant) or preferential staining of one or more phases, because of differences in chemical composition and, because of differences in grain-orientation. Before being etched, a specimen should be inspected for polishing defects, such as scratches, pits, relief polish, comet tails, pulled out inclusions, and voids.

Chemical Etching: Chemical etching is accomplished by immersing the specimen in (or swabbing it with) a suitable etchant until the required structure is revealed. Etching is done in Petri-dishes or in other suitable containers with loose covers to prevent excessive evaporation of the solvent, particularly alcohol solutions. Glass containers can be used for all etchants except hydrofluoric acid solutions, where the container should be made of polyethylene or other suitable material. By the use of tongs or other convenient handling device, the surface of the specimen is immersed in the etchant with some agitation to ensure that fresh etchant is in contact with the specimen all the time. During etching, most metals lose their bright appearance, indicating that etching is taking place. With practice, one can ascertain the completion of etching by the degree of dullness of the surface. If the etching procedure calls for swabbing, the surface of the specimen can be swabbed with cotton saturated with the etchant, or the specimen can be immersed and swabbed while in the solution. When etching is complete, the specimen is rinsed in running water and then in alcohol, followed by drying in a stream of warm air (hand dryer). After etching the specimen surface is observed under the optical-microscope for studying its microstructure. Care should be taken while etching so that the hand is not affected by the etching.

To report:

1. What is mounting?
2. Differentiate between hot and cold mounting?
3. Define terms for polishing, grinding, and mesh size?
4. Explain the importance of etchant?
5. Which types of materials are basically polished by electrolytic polishing?
6. What are the effects you have observed on changing etchant time?

7. HF should not be kept in a glass bottle. Why?
8. Which type of precaution should be taken care of before etching?

EXPERIMENT 3

Optical Microscopy of ferrous samples

Objective

Optical Microscopy of different ferrous samples of Steel (Mild steel, Stainless Steel) and Cast Irons

Methodology

- Follow all the steps for making the specimen for etching (as per previous lab)
- Etch with the proper etchant (list of etchants is given in the Appendix)
- See the microstructure with appropriate magnification.
- Draw the microstructure and label.

Brief description about iron carbon diagram:

Read: The Principles of Metallographic Laboratory Practice by George L. Khel
Physical Metallurgy by V. Raghvan and Engineering
Physical Metallurgy by Yu. Lakhtin

We shall now consider alloys of Iron and Carbon in our experiment. We will look at the microstrucrures of Fe-C alloys, which undergo solid state transformations, such as eutectoid transformations, martensitic transformations, etc. Fe-C alloys with C content less than 2wt%C are known as plain carbon steels. There are many reasons for studying the microstructure of plain carbon steels. Carbon steels by far find the maximum use in industries and daily life. With the variation of different phases, the processing, properties and use of the Fe-C alloys also vary. Hence, Fe-C phase diagram and the microstructure in carbon steels are to be studied so extensively.

Let us first look at the Fe rich portion of classical Fe-C phase diagram (Figure 2). Cementite is an intermetallic compound orthorhombic crystal structure. This phase diagram depicts a number of invariant points. If we consider an alloy with 0.8wt% C and heat it to 950⁰C (1223K), it forms a single phase austenite having FCC crystal structure. Austenite has high solubility of carbon (max. 2.11wt% at about 1150⁰C) On cooling such an alloy below 723⁰C, the alloy undergoes eutectoid transformation forming two phases, ferrite(α) and cementite, simultaneously. This structure is known as pearlite, which consists of alternate lamellae of α and Fe₃C. Steels having composition less than 0.8 wt% C (known as

hypoeutectoid steels) has primary α and pearlite in the microstructure because on cooling such a steel from γ region, it forms α first until the composition of the remaining γ reaches 0.8 wt% at temperature of 723°C , and this austenite undergoes eutectoid transformation. Therefore, microstructure of hypoeutectoid steels consists of proeutectoid α with lamellar pearlite in varying proportions depending on the alloy composition. Similarly, Fe-C alloy compositions more than 0.8 wt% C are termed as hypereutectoid steels. The microstructure of such steels consists of pro eutectoid Fe_3C with lamellar pearlite.

The microstructure of steels depends on heat treatment conditions. If we heat a steel with 0.7wt% C to 950°C and suddenly quench the steel in water (or brine solution), the steel becomes very hard. Quenching of steel is the rapid cooling from a suitably elevated temperature. This is because γ transforms to a new phase called martensite, which is a metastable phase with body centered tetragonal crystal structure. Martensite has lath shaped morphology, which are internally twinned. The microstructures show twin laths in different orientations. The most important alloy steel is stainless steel. Stainless steels are stainless as they have minimum 11.5wt% Cr, which forms very thin, protective, adherent and stable oxide (preferably Cr_2O_3) film on the surface. This film is continuous, impervious and passive to stop further reaction between steels and atmosphere. Stainless steels can be of different types depending on the microstructure: ferrite, austenitic, martensitic, duplex types. We will study 18 wt% Cr and 8 wt% Ni, famously known as 18-8 austenitic stainless steel. The microstructure shows grains of austenite.

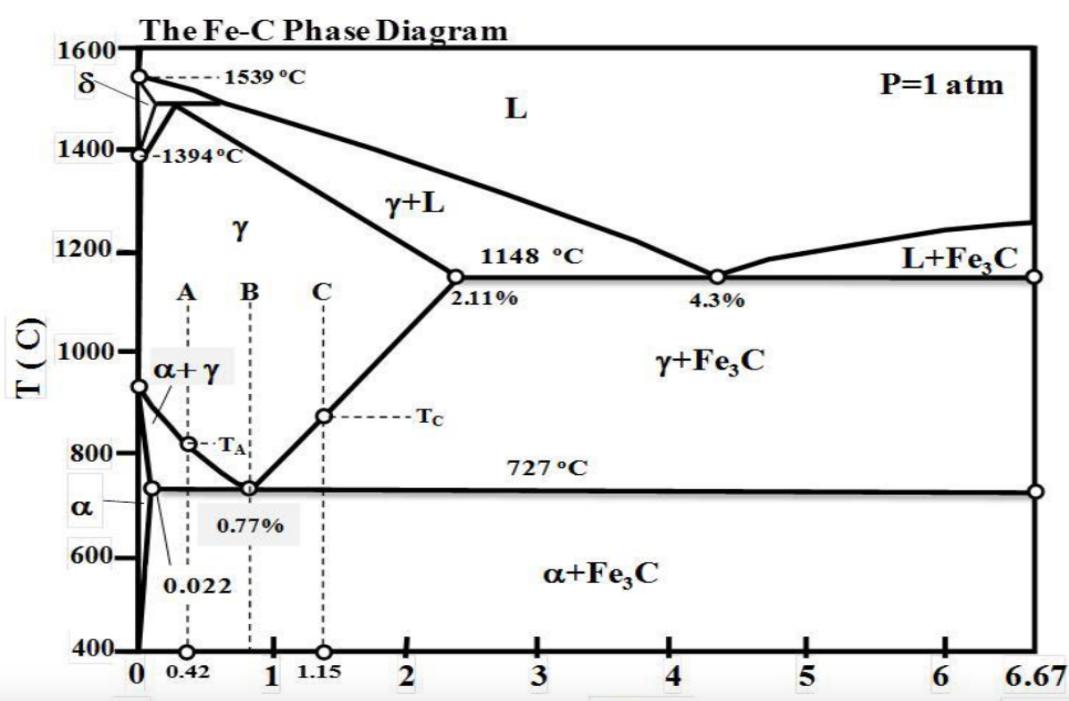
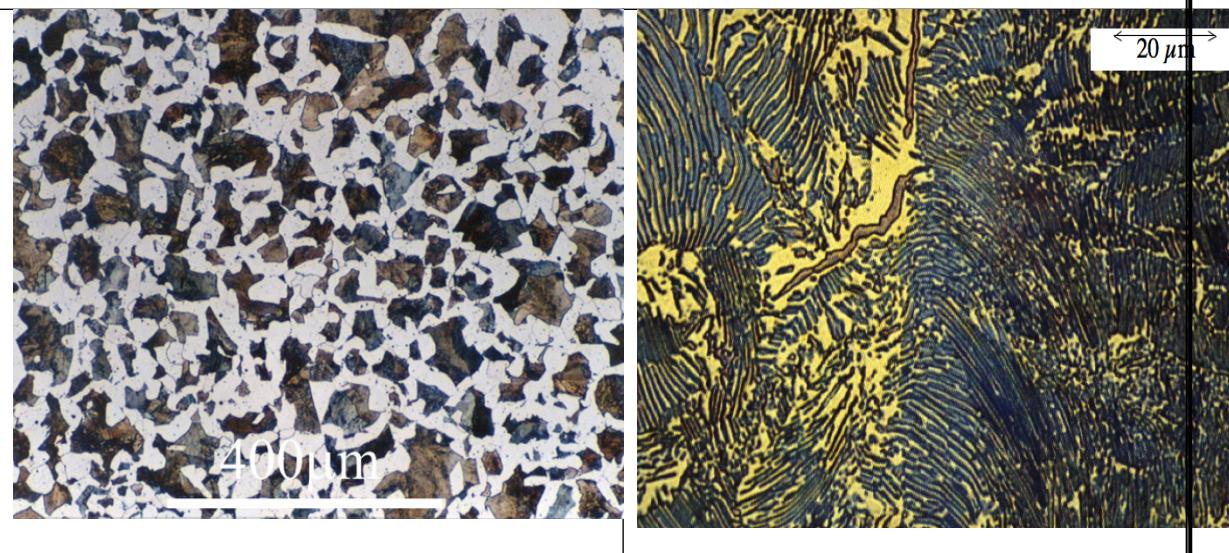
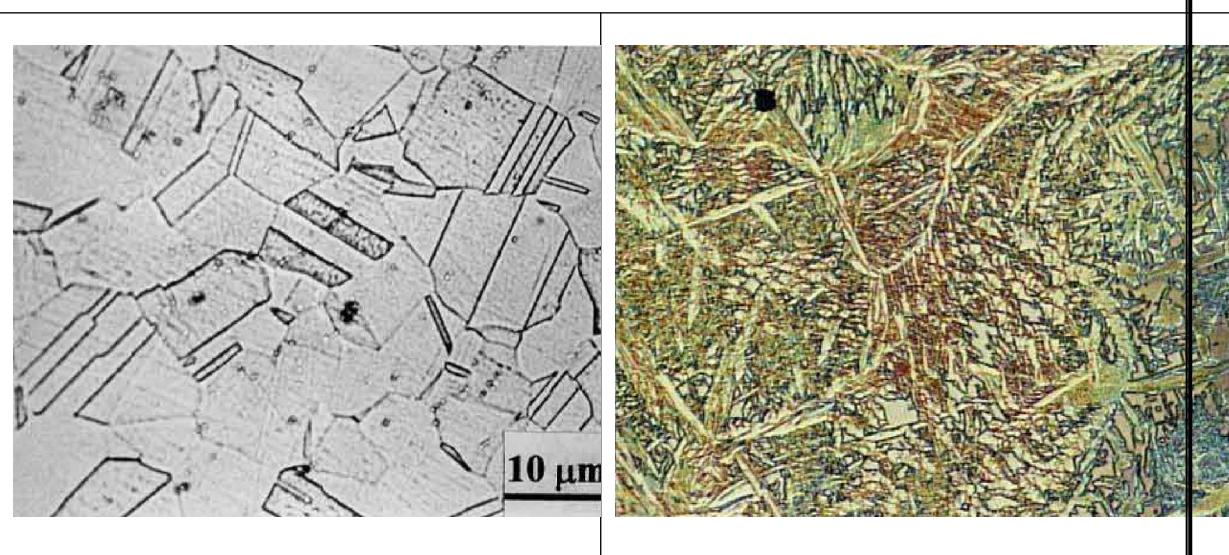


Figure 2: Phase diagram of Fe-C system



Ferrite in a Fe-0.4C steel which is slowly cooled; the remaining dark-etching microstructure is fine pearlite, The appearance of a pearlitic microstructure (right image).



Microstructure of 304 stainless steel,
phases

Colour mixture of austenite, ferrite and sigma

To report:

1. Draw the structures that you see under microscope and label.

EXPERIMENT 4

Optical Microscopy of Non Ferrous Sample

Objective:

Optical Microscopy of different non ferrous samples (Lead-Tin alloy, Copper, Brass)

Methodology

- Follow all the steps for making the specimen for etching.
- Etch with the proper solutions (list of etchant is given at the end of the document)
- See the microstructure with appropriate magnification.
- Draw the microstructure and label.

Brief description about eutectic alloys.

Read: The Principles of Metallographic Laboratory Practice by George L. Khel

Physical Metallurgy by V.Raghvan and Engineering Physical Metallurgy by Yu Lakhtin

The metallography of non-ferrous metals is a little tricky since they are soft and in some of the cases, special reagents are required for etching.

Copper (Cu):

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

Brass:

Figure 4 shows the Cu-Zn phase diagram with different phases. The Cu-rich portion of the phase diagram reveals a cascade of peritectic reactions. The peritectic reactions corresponding to $\alpha + \beta$ brass are marked. In the laboratory exercise, you will study the microstructure of composition of alloy Cu:Zn = 60:40. The microstructure will reveal presence of both the α and β phases because of incomplete peritectic reaction. Apart from Brass, many other technically important alloy systems such as steels, CuSn (Bronze), rare earth permanent magnets (Nd-Fe-B) and high Tc superconductors (Yb-B-Cu-O) display peritectic reactions, where phase and microstructure selection plays an important role for

the processing and the properties of the material. The peritectic reaction can be written as $\alpha + L \rightarrow \beta$ on cooling, where α and β are solid phases and L stands for liquid. Therefore, a primary solid phase reacts with liquid leading to formation of another solid phase. The second solid phase is called peritectic phase. The equilibrium microstructure of such alloys will show β as dendrites (tree-like structure). During normal freezing of the peritectic alloy, the reaction never goes to completion and hence, some amount of primary α phase remains in the microstructure surrounded by the β phase.

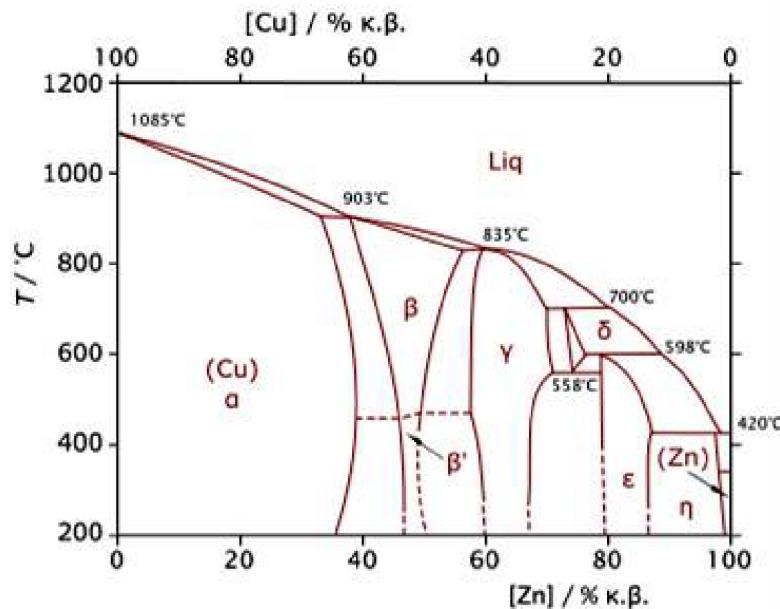


Figure 3: Phase diagram of Cu-Zn system

To Report:

1. Draw the microstructures and label

EXPERIMENT 5

Quantitative Metallography

Objective

- 1) To analyse the microstructure of a specimen quantitatively.
- 2) To verify the basic stereological equations $Vv=AA=LL=PP$.
- 3) To measure the volume fraction of a phase in a polycrystalline material.
- 4) The whole process requires proper metallographic sample preparation and use of image analyser.

Background:

The quantitative analysis of a microstructure is performed on a two dimensional plane (also called plane of polish) which is cut through the three dimensional structure of the material. This technique is known as stereology. The basic measurements which are done in this technique are grain size measurement and the determination of volume fraction of a phase or constituent in polycrystalline materials. The following methods are used for this purpose:

Grain size measurement:

1) Jefferies method:

This method involves the counting of the number of grains which lie in a rectangular or circular area marked on the microstructure. The grains, which are partly inside the marked area, are counted as half. From this, number of grains per unit area (NA) is determined and the grain diameter is calculated from average grain area (A) using the formula:

$$d = (A)^{1/2} = 1/(NA)^{1/2}$$

The grain size is often computed from an ASTM grain size number as: $N=2^{n-1}$

Where n is ASTM number and N is number of grains per square inch at 100X.

2) Mean Intercept length Method:

In this method, the polycrystalline microstructure is superimposed by a grid of parallel lines and the number of intercepts per unit length (PL) is obtained. The mean intercept length is given by:

$$L = 1/PL$$

And the grain size N, is related with L as:

$$N = -6.6457 \ln L - 3.298 \quad (L \text{ in mm})$$

Volume Fraction Determination:

1) Point Counting Method:

In this method, a set of points is overlaid on a microstructural field and the number of points contained in the phase of intercept is counted. The PP is calculated by dividing the number of test points in the phase of intercept by the total number of test points. The average value of PP is equal to the volume fraction (VV).

$$VV = \langle PP \rangle$$

This method is called systematic point counting, when a regular array of points is used and is called random point counting, when random test points are used.

2) Lineal Analysis:

In this method, the test length of randomly placed lines within the phase of intercept $L\alpha$ is calculated and is divided by the total line length (LT)

$$LL = L\alpha / LT$$

And also

$$VV = \langle LL \rangle$$

3) Areal Analysis:

This method involves measurement of the fraction of the area of representative metallographic planes AA occupied by the phase of intercept. The average value of this area fraction is equal to the volume fraction.

$$VV = \langle AA \rangle$$

Observations You Need to Make:

Take readings individually for quantification

Counting of changes as you select regions ‘randomly’ (unbiased observation).

Comparing the lineal, areal and volume fraction.

To Report:

1. What do you mean by quantitative measurement?
2. What is stereology?
3. Define volume fraction?
4. How will you calculate grain diameter with the help of Jefferies method?

Appendix

List of Etchants

S. No	Etching Reagent	Composition	Suitable for
1	Nitric Acid (Nital)	HNO ₃ 1 to 5 ml Ethyl or methyl Alcohol 100 ml	For low carbon and medium carbon steel
2	Picric Acid (Picral)	Picric Acid 4 gm Ethyl or methyl alcohol 100ml	For all grades of carbon steel
3	Ferric Chloride and Hydrochloric acid	FeCl ₃ 5g HCl 50 ml H ₂ O 100 ml	Stainless Steel
4	Ferric Chloride	FeCl ₃ 5 to 10 g HCl 15 to 20 ml Water 100 ml	Copper, Brass