

MSE 311
Physical Metallurgy Lab
 2015-2016

Instructor

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 (Venue- Physical Metallurgy Lab WL-201)

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General Information

Total Laboratory Turns: 12 [6 experiments in first session (before midsem) and 6 experiments in next session (after midsem)]

Lab Exam/Lab Viva/Lab Quiz will be either after both sets of experiments or after each set of experiment. Each day, viva will be taken on the experiment.

Marks Distribution of Lab:

- Laboratory (Total 100 %)
 - a) Daily Lab Report 10%
 - b) Daily Lab Viva 10%
 - c) Assignment 15%
 - d) Project 25%
 - e) Final Lab Exam/ Lab Viva 40%

SAFETY:

- To avoid injury, the students must take the permission of the laboratory staffs before handling any machine. Careless handling of machines may result in serious injury.
- Students must ensure that their work areas are clean.
- At the end of each experiment, students must clear off all tools and materials from the work area.

RULES:

- Follow the lab timing and come in proper attire. There will be two attendances: Initial attendance (at sharp 2 PM) to be taken by TAs at the beginning of lab session and final one after the experiments over. Late comers will not be allowed to enter.
- Do not use cell phone inside the lab during lab timing.
- Students must come to the laboratory wearing trousers and shoes. Half pants, loosely hanging garments and slippers are strictly prohibited.
- Every student should obtain a copy of Laboratory Manual. You are requested to bring your lab manual on the day of experiment.
- The Lab report should contain: (i) Title of the experiment, (ii) Three to four lines stating the objectives, (iii) Name of all equipments/tools used along with a one line description of their use, and (iv) Observations and (v) Answer to questions specifically asked in the section “to report” The report should be short and nicely prepared.

RECOMMENDED READING:

- The Principles of Metallographic Laboratory Practice by George L. Khel
- Physical Metallurgy by V. Raghvan
- Engineering Physical Metallurgy by Yu. Lakhtin
- Material Science by S P Gupta

Experiment 1

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 the Metallurgical Engineering.

A well prepared metallographic specimen is:

- a. A representative sample
- b. 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.
- c. Free from polishing scratches and pits and liquid staining
- d. 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 specimen has 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 convenient 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 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 means 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 into 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.

Particle Size vs. Common Grit Sizes for Abrasive Papers

GRIT NUMBER		
European (P-grade)	Standard grit	Median Diameter, (microns)
60	60	250
80	80	180
100	100	150
120	120	106
150	150	90
180	180	75
220	220	63
P240	240	58.5
P280		52.2
P320	280	46.2
P360	320	40.5
P400		35
P500	360	30.2
P600	400	25.75
P800		21.8
P1000	500	18.3
P1200	600	15.3
P2400	800	6.5
P4000	1200	2.5

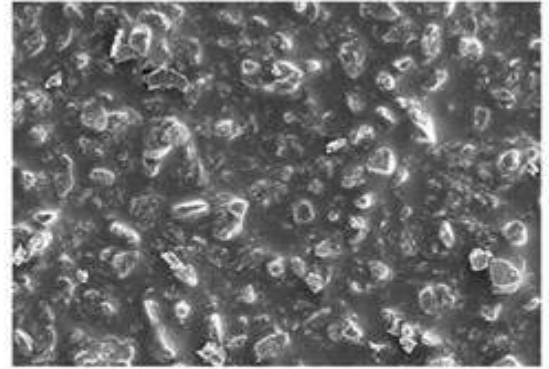


Figure SEM micrograph of 600 grit SiC Abrasive Paper (original mag. 150x)

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 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 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 next stage grinding is always helpful.

Grinding Mediums

Grinding media are silicon carbide (SiC), aluminium oxide (Al₂O₃), emery (Al₂O₃-Fe₃O₄), and diamond particles. All except diamond 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 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 Al_2O_3 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 abrasive 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 the amount, 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 surface of the sample which results in a leveling and brightening of its surface.

Electro polishing 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 works 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 as polished mirror like surface in the metal. It can be used for phase identification, for dislocation density caculation (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 times. 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 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 glass bottle. Why?
8. Which type of precaution should be taken care of before etching?

EXPERIMENT 2

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 first day 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 microstructures of Fe-C alloys, which undergo solid state transformations, such as eutectoid transformations, martensitic transformations, etc. Fe-C alloys with C content less than 2 wt% 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 compounds 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 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 723⁰C, 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 Fe₃C with lamellar pearlite.

The microstructure of steels depends on heat treatment conditions. If we heat a steel with 0.7wt% C to 950⁰ 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 center tetragonal crystal structure. Martensite has lath shaped morphology, which are internally twinned. The microstructures shows 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.

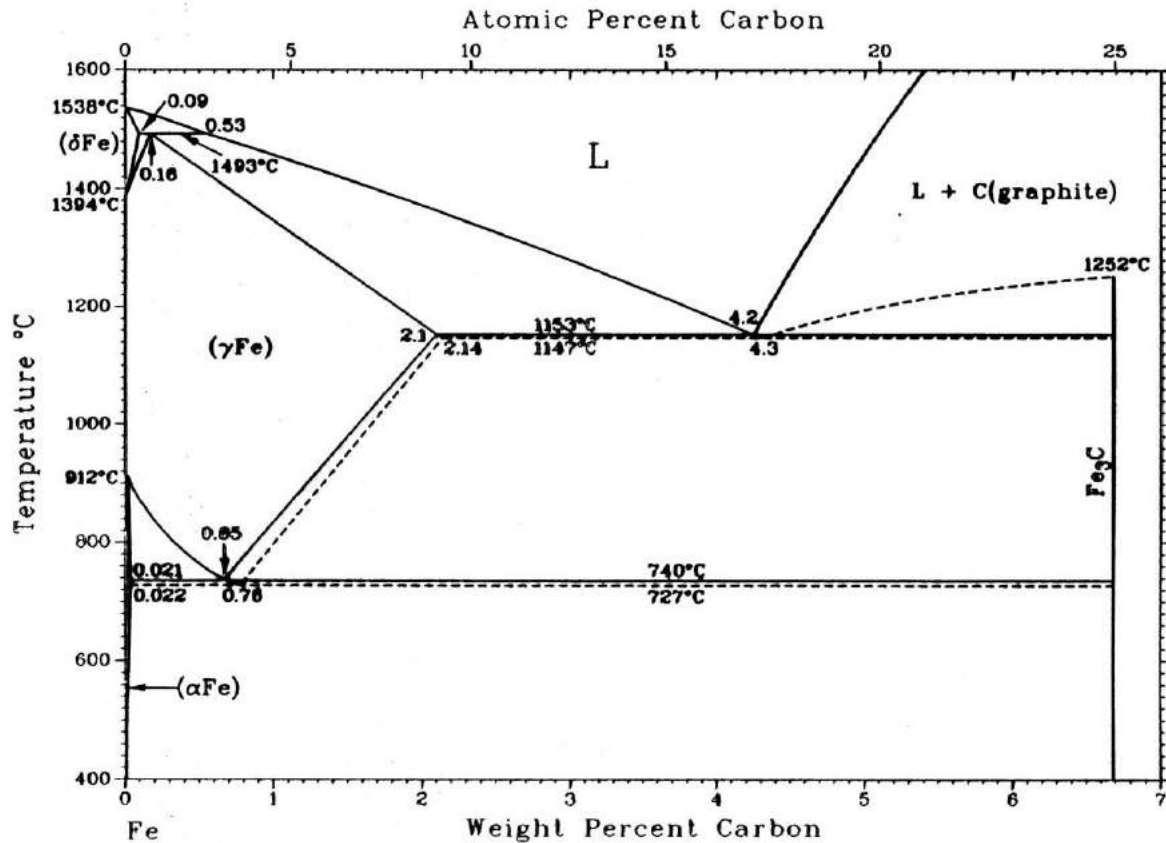


Figure 2: Phase diagram of Fe-C system

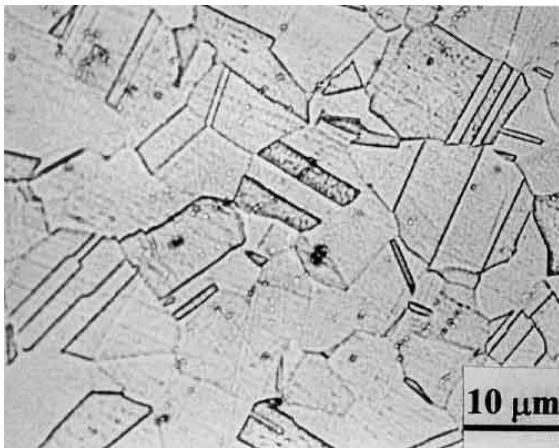
Cast Irons

Gray Cast iron: When a cast iron is solidified slowly, the tendency of carbon to separate out as graphite increases. In gray cast iron the graphite precipitates as irregular, elongated, curved and interconnected plates. Although the flakes appear as if separated in the two dimensional microstructure, they are interconnected in three dimensional structures. The large graphite flakes not only interrupt the continuity of the matrix but also act as stress risers. As a result, the strength and ductility of the gray cast iron decreases. However, the presence of flake graphite provides good wear and thermal properties along with good damping capacity. Gray cast iron is widely used for gear and pump housings, machines beds, brake drums, motor frames and electrical equipment.

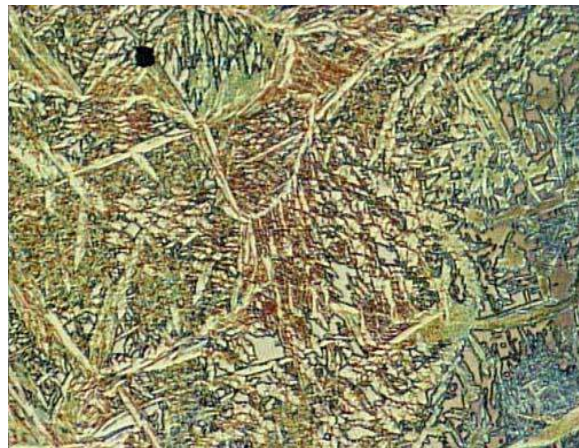
White cast iron: White cast irons are very brittle and contain eutectic mixture of austenite and cementite (known as ledeburite) upon cooling. A typical microstructure of white cast iron

consists of dendrites of transformed austenite (later to pearlite) in white interdendritic network of cementite. Cementite is a hard, brittle interstitial compound. Since white cast iron contains relatively large amounts of cementite in the form of continuous interdendritic network, it makes the white cast iron “hard” and “wear resistant”. However, it is extremely brittle and difficult to machine. White cast irons have limited in engineering applications, because of inherent brittleness and lack of machinability. They are used where wear resistance is most important, and service condition does not require ductility such as liners for cement mixtures, ball mills, certain type of drawing dies and extrusion nozzles.

Nodular/ Spheroidal cast iron: In the nodular cast iron the graphite is present as tiny balls or nodules. Nodular iron is produced by the addition of one or more elements, such as as Mg, Ce, Ca, Li, Na, and Ba to the molten metal. Mg plays an important role in the formation of graphite nodules. The spheroids result in higher strength and toughness compared to gray cast iron. Some typical application of nodular iron includes crank shafts, pistons, cylinder heads, fly wheels, work rolls, lever, etc.



Microstructure of 304 stainless steel phases



Colour mixture of austenite, ferrite and sigma

To report:

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

EXPERIMENT 3

Optical Microscopy of Non Ferrous Sample

Objective:

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

Methodology

- Follow all the steps for making the specimen for etching (as per first day lab)
- 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 are little tricky since they are soft and 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, 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 pipe lines for water.

Zinc (Zn):

Zinc is hexagonal closed packed structure with limited ductility. However, when formed alloys with Cu, it forms one of the exciting engineering alloy, called Brass. Zinc finds some crucial engineering applications, like galvanization of steel (for corrosion protection), sacrificial anode for protecting underground metallic structures (cathodic protection), etc.

Brass:

Figure 4 shows the Cu-Zn phase diagram with different phases. Cu-rich portion of the phase diagram reveals a cascade of peritectic reactions. The peritectic reactions corresponding to $\alpha + \beta$ brass is 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 T_c 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 $a + L \rightarrow \beta$ 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.

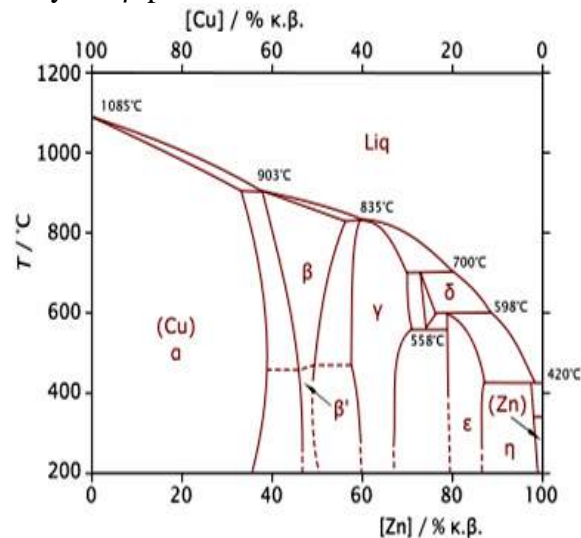


Figure 3: Phase diagram of Cu-Zn system

Lead-Tin alloys (Pb-Sn):

This alloy system is important since it finds application as solder material due to low melting eutectic. The phase diagram of Pb-Sn system is shown in Fig. 4. The word eutectic originates from a Greek word 'eutektos' meaning 'easily melted'. These alloys can be easily melted because the melting temperature of these alloys is lowest in the concerned phase diagram. Eutectic alloys are used extensively in industries such as automobile, electronic industry. The classical examples are Pb-Sn, Al-Si, Cu-Zn etc. For a binary alloy, the eutectic reaction is given by $L \rightarrow \alpha + \beta$ during cooling, where L stands for liquid and α and β are two solid phases. Hence, two solid phases start to crystallize simultaneously from the liquid phase at the eutectic temperature. Such a simultaneous crystallization of a eutectic mixture is known as a eutectic reaction. The temperature at which it takes place is the eutectic temperature, and the composition and temperature at which it takes place is called the eutectic point. The eutectic point is an invariant point in the phase diagram because it has zero degrees of freedom. Therefore, the composition as well as temperature of the eutectic reaction is fixed for a particular alloy system.

Pb-Sn phase diagram consists of two terminal solid solution (α and β) and a eutectic point at a composition of 17.8 at% Sn and temperature of 183°C. Alloy compositions to the left of the eutectic (pure Pb to 17.8) are called hypo-eutectic and alloy compositions to the right of the eutectic composition (17.8 at% Sn to pure Sn) are known as hyper-eutectic alloys. The microstructure of the alloys depends on the alloy composition. For a hypo-eutectic alloy, the microstructure consists of primary α -dendrite and eutectic mixture whereas hyper-eutectic alloys shows primary β -dendrite and eutectic mixture.

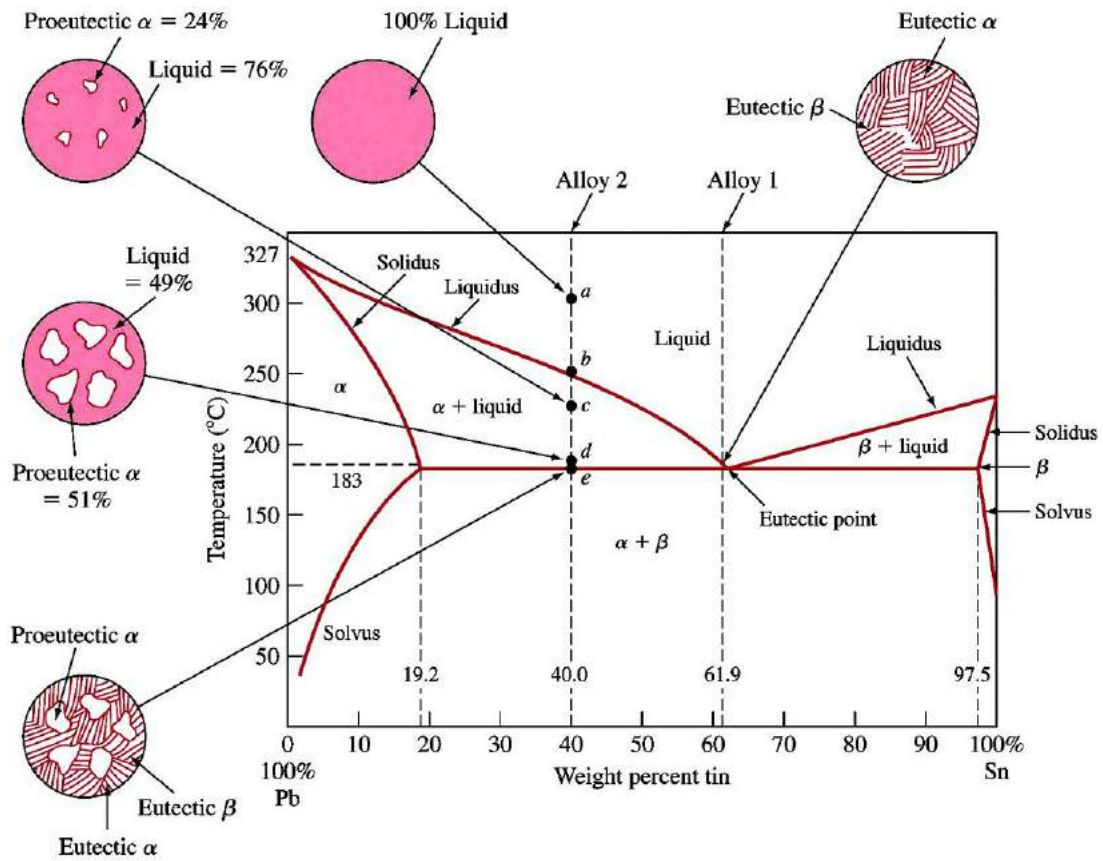


Figure 4: Phase diagram of Pb-Sn system.

To Report:

1. Draw the microstructures and label

EXPERIMENT 4

Quantitative Metallography

Objective

- 1) To analyze the microstructure of a specimen quantitatively.
- 2) To verify the basic stereological equations $V_v=AA=LL=PP$.
- 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 analyzer.

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 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 interest is counted. The PP is calculated by dividing the number of test points in the phase of interest 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 you will calculate grain diameter with the help of Jefferies method?

EXPERIMENT No - 05

Objective

To identify an unknown sample (metallic) by indexing of peaks with the help of obtained XRD pattern and precision lattice parameter measurement via Nelson-Riley Function.

Procedure:

I. Spectrum Analysis

1. You will receive two XRD spectra after doing XRD of two different cubic samples.
2. Calculate the d-spacing's of each peak.
3. Identify the sample and crystal structure by doing proper calculation.

II. X-ray Diffraction Experiment of unknown sample

1. You will learn the procedure of running XRD experiment from TA/Instructor.
2. Read the instructions for the powder X-ray diffractometer. TA/Instructor will explain the operation.
3. TA/Instructor will record the X-ray diffraction patterns of two unknown samples one for each group.
4. Analysis the X-ray diffraction patterns.
5. Please list the observed differences between experimental data and standard spectrum. (This will be done after you complete the spectrum analysis).

Background/Basic Principle:

Please read "Elements Of X Ray Diffraction : Cullity, B. D"

To Report:

1. Calculation sheet for identification of structure
2. Calculation sheet and graph sheet for lattice parameter measurement

Appendix

List of Etchants

Sr	Etching Regent	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
5	Mixed Acids	HNO ₃ 5ml Water 95ml	Lead Tin Alloy
6	Mixed Acids	HCl 50 ml Water 50 ml	Zinc and its alloys

MSE 311 : Physical Metallurgy Lab

(SET-1)

Till Mid Semester

		Experiment 1	Experiment 2	Experiment 3	Experiment 4	Experiment 5	Experiment 6 & Lab Viva	Remarks
Monday	Introduction, Group & Day Distribution 27/7/2015	3-Aug-15	10-Aug-15	17-Aug-15	24-Aug-15	31-Aug-15	For Experiment No 6 and Lab Viva date will announce later	Gr. A to Gr. D
Tuesday		4-Aug-15	11-Aug-15	18-Aug-15	25-Aug-15	1-Sep-15		Gr. E to Gr. H
Thursday		6-Aug-15	13-Aug-15	20-Aug-15	27-Aug-15	3-Sep-15		Gr. I to Gr. L
MONDAY	SET 1							
Experiment → Date↓		Experiment 1	Experiment 2	Experiment 3	Experiment 4	Experiment 5	Experiment 6 & Lab Viva	Remarks
03-Aug		all students will complete their first experiment					For Experiment No 6 and Lab Viva date will announce later	
10-Aug	All Students of Monday Batch (03/08/2015)	Group A	Group B	Group C	Group D			
17-Aug		Group D	Group A	Group B	Group C			
24-Aug		Group C	Group D	Group A	Group B			
31-Aug		Group B	Group C	Group D	Group A			
TUESDAY	SET 1							
Experiment → Date↓		Experiment 1	Experiment 2	Experiment 3	Experiment 4	Experiment 5	Experiment 6 & Lab Viva	Remarks
04-Aug		all students will complete their first experiment					For Experiment No 6 and Lab Viva date will announce later	
11-Aug	All Students of Tuesday Batch (04/08/2015)	Group E	Group F	Group G	Group H			
18-Aug		Group H	Group E	Group F	Group G			
25-Aug		Group G	Group H	Group E	Group F			
01-Sep		Group F	Group G	Group H	Group E			
THURSDAY	SET 1							
Experiment → Date↓		Experiment 1	Experiment 2	Experiment 3	Experiment 4	Experiment 5	Experiment 6 & Lab Viva	Remarks
06-Aug		all students will complete their first experiment					For Experiment No 6 and Lab Viva date will announce later	
13-Aug	All Students of Thursday Batch (06/08/2015)	Group I	Group J	Group G	Group L			
20-Aug		Group L	Group I	Group J	Group G			
27-Aug		Group G	Group L	Group I	Group J			
03-Sep		Group J	Group G	Group L	Group I			

