

**PHASE EQUILIBRIA IN MATERIALS
(MME 330)**

LABORATORY MANUAL



**Department of Materials and Metallurgical Engineering
Indian Institute of Technology, Kanpur**

**5th Semester
(2009-2010)**

MME330: Phase Equilibria in Materials (2009-10 Sem I)

Laboratory Schedule

Turn→ Section ↓													
	1 st	2 nd	3 rd	4 th	5 th	6 th	7 th	8 th	9 th	10 th	11 th	12 th	13 th
Wednesday	7/29	8/5	8/12	8/19	8/26	9/2	9/9	9/16	9/23	10/7	10/21	10/28	11/4
Friday	7/31	8/7	8/21	8/22	9/4	9/11	9/18	9/25	10/9	10/16	10/23	10/30	11/6

1st Mid Semester Examination : August 27 – August 29, 2009
Mid Semester Recess : September 27 - October 04, 2009
2nd Mid Semester Examination : October 12 – October 14, 2008
Laboratory Examination : Group 1: November 11 and Group 2: November 13, 2009
End Semester Examination : November 16 – November 26, 2009

Important reminders: 1.1ST TURN: FORMATION OF GROUPS

2. HANDLE THE MICROSOPEs WITH CARE

3. POLISH AND ETCH EACH SAMPLE CAREFULLY TO GET MICROSTRUCTURE IN SINGLE RUN

4. TAKE INITIAL ON THE MEASURED DATA/OBSERVATIONS

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GENERAL INSTRUCTIONS

1. Every student should obtain a copy of the MME 330 laboratory manual.
2. Dress code: Students must come to the laboratory wearing: (1) trousers, (ii) half-sleeve tops and (iii) Leather shoes. Half pants, loosely hanging garments and slippers are not allowed.
3. To avoid any injury, the student must take the permission of the laboratory staffs before handling the machines.
- 4. EVERY STUDENT IS REQUIRED TO HANDLE THE OPTICAL MICROSCOPE WITH CARE.**
5. Students must ensure that their work areas are clean.
6. At the end of each experiment, the student must take initials from the staff on your data/observations.
7. Laboratory report must be submitted in standard sheet, available at the shopping center in the subsequent lab turn. Reports on ordinary sheets and computer papers will not be accepted.
8. Each member of any group must submit lab report even if the experiment has been performed in a group.
9. The lab report must contain: (1) Title of the experiment, (ii) Three to four lines stating the objectives, (iii) A few lines on background; (iii) Name of all equipments/tools used along with one line description of its use and (iv) Neatly labeled sketches of the observed microstructures with few lines of description.
10. Student can check their laboratory reports after correction for discussion.
- 11. Careless handling of machine, especially optical microscope will not be tolerated.**

GENERAL INFORMATION

In this laboratory you will be exposed to various aspects of phase diagrams, microstructure, quantitative metallographic techniques and phase transformation behaviour of alloys. You will be doing experiments on different types of materials and thereby understanding the phase formation and microstructure of the materials. You will also carry out experiments on determination of phase diagram using cooling curves of different alloys. You are required to submit a report on each experiment you will be performing. You will be asked questions on various aspects of the experiments. You need to refer to the following text books.

RECOMMENDED READING

- 1. PHYSICAL METALLURGY PRINCIPLES, *R. Abbaschian and R.E. Reed-Hill*, University Series in Basic Engg. , 1991**
- 2. INTRODUCTION TO PHYSICAL METALLURGY, *S.H.Avner*, Tata McGraw Hill Publications, 1997**
- 3. PHASE DIAGRAMS IN METALLURGY, *F.Rhines*, McGraw-Hill, 1956**
- 4. ALLOY PHASE EQUILIBIRA, *Allan Prince*, Elsevier Publishing Company, 1966**
- 5. PHASE EQUILIBRIA IN MATERIALS, *Shant P.Gupta*, Allied Publishers Private Limited, 2003**
- 6. QUANTITATIVE MICROSCOPY, *R.T. DeHoff and F.N.Rhines*, McGraw Hill Book Company, 1968**
- 7. PRACTICAL STEREOLOGY; Second Edition, *John C. Russ and Robert T.Dehoff*, Kluwer Academic/Plenum Publisers, 2001**

DETAILS OF EXPERIMENTS:

SET I

- 1) Structure of pure metals and solid solutions: Cu, Fe, Zn(as cast), Cu-Ni(as cast) and Cu-Ni (annealed)
- 2) Eutectic, Hypo- and Hyper-eutectic alloys: Al-Si (unmodified), Al-Si(modified), Pb-6wt%Sb, Pb- 11.1 wt%Sb and Pb- 20 wt%Sb
- 3) Fe -C Alloys: Steels: 0.2wt%C, 0.6 wt%C, 0.8wt%C, 1.2 wt%C, 0.7wt%C quenched from 850⁰C, 0.7 wt%C steel quenched and tempered
- 4) Alloy Steels: 0.23 wt%C and 0.85 %Mn Alloy Steels, Free cutting steels, 18/8 Austenitic stainless steel, High Speed Tool steel
- 5) Cast iron: Grey Cast iron, White cast iron, Malleable Iron with ferritic matrix, Malleable iron with pearlitic and ferritic matrix, Nodular iron
- 6) Peritectic and Montectic Alloys: 60:40 ($\alpha+\beta$) Brass, 70:30 (α) Brass, Cu-10wt%Sn, Cu- 36wt% Pb, Cu- 50 wt% Pb

SET II

- 1) Measurement of volume fraction, surface area in two phase and grain size in single phase materials
- 2) Measurements of particle size and its distribution
- 3) Heat treatment of 0.6 wt% C steels: Annealing (Furnace cooled, Normalizing (Air cooled), Water quenched, Oil quenched
- 4) Phase Transformation of Pb-Sn Eutectic Alloy using DTA
- 5) Cooling curves of Pb-Sn Eutectic Alloy
- 6) Cooling curve of Pb-Sn Hypo-eutectic Alloy

SET I: EXPERIMENT 1

STRUCTURE OF PURE METALS AND SOLID SOLUTIONS

Objective

To study and observe the microstructure of pure metals and continuous solid solutions

Background

Microstructure of materials is the 'heart' of materials science and engineering. Understanding microstructure and correlating it with properties of materials form the basis for studying the microstructure. Phase diagram dictates different phases that can be formed under the equilibrium condition for a particular metals or alloy. Therefore, the microstructure in a material is interlinked with the phase diagram under study. By definition the structure that is observed when a polished and etched specimen of metal or alloy is viewed in an optical microscope at magnifications more than x25 is called microstructure.

Pure metals such as Cu, Zn, and Fe are commonly used in various applications. Pure Cu finds applications in electronics, biomedical industry. Pure Fe is used extensively in the making steels and cast irons as well as biomedical applications. Pure Zn finds major applications in making corrosion resistance coatings, fuel cells, batteries. Microstructure of the pure metals consists of grains of different sizes and shapes when viewed under optical or visible light microscope (VLM). The grains are separated from each other by what is known as grain boundaries. The size and shape of the grain depends on the processing conditions. As cast metals show characteristic tree-like structure (as large as several mm which can even be viewed with naked eyes) known as *dendrite*. On the other hand microstructure of deformed metals will show finer elongated grains.

Solid solutions are alloys formed by two or more metals. In solid solutions, one metal get dissolved into other (just like in case of sugar in water!). Therefore, solid solution is solid-state solution of one or more solutes in a solvent. Such a mixture is considered a solution rather than a compound when the crystal structure of the solvent remains unchanged by addition of the solutes, and when the mixture remains in a single homogeneous phase. The solute may incorporate into the solvent crystal lattice substitutionally, by replacing a solvent atoms in the lattice, or interstitially, by fitting into the space between solvent atoms. Both Cu and Ni have face centered cubic (fcc) crystal structure. When these two are mixed in any proportions, they form a solid solution having fcc structure. The figure 1 shows phase diagram of Cu-Ni. You can clearly observe that Cu and Ni forms solid solution at all proportions, called continuous solid -solutions. Like pure metals, the solid solution will also have microstructure consisting of dendrite which can be observed because of segregation of solute at regions among the dendrites, called interdendritic space. Therefore, as cast microstructure will show large dendrite of different sizes with lots of segregation (due to coring effect). Annealing (heating to high temperature followed by furnace cooling) will lead to homogenization and thereby reducing the segregation.

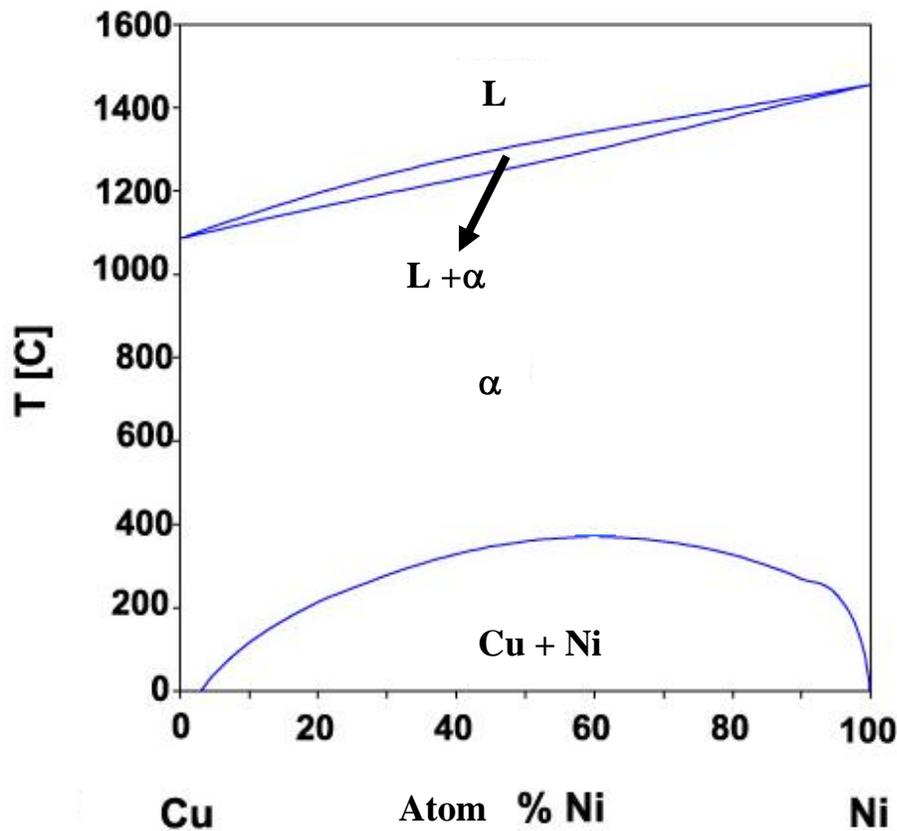


Figure 1: Cu-Ni binary phase diagram

LABORATORY EXERCISE

1. Materials: Cu, Fe, Zn(as cast), Cu-Ni(as cast) and Cu-Ni (annealed)

2. Procedure:

- (a) Polish samples using emery papers of 1/0, 2/0, 3/0, 4/0 followed by cloth polishing using 5 μm Al₂O₃ slurry.
- (b) Etch using proper chemical etchant (Fe : Nital, Cu : FeCl₃, (Cu-Ni): FeCl₃ and Zn: 50vol% HCl)
- (c) Look at the sample in optical microscope

Report the following

- 1. Draw neatly the microstructure of all the samples as observed under optical microscope. Highlight the major features and magnifications used
- 2. Machines and materials used
- 3. Etchants used and optimum etching time required to obtain reasonably good microstructure.

SET I: EXPERIMENT 2

EUTECTIC, HYPO- AND HYPER-EUTECTIC ALLOYS: Al-Si (UNMODIFIED), Al-Si (MODIFIED), Pb-6wt%Sb, Pb- 11.1 wt%Sb and Pb- 20 wt%Sb

Objective:

To study and observe the microstructure of eutectic alloys: Al- Si and Pb-Sb

Background

The second type of alloys we will study is known as eutectic alloys. 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, Fe-C (cast iron) 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 crystallizes 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 a 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.

Figure 2 shows phase diagrams of both Al-Si and Pb-Sb alloys systems. Al-Si alloy phase diagram consists of a primary solution solution (α -Al) and eutectic and pure Si. Al and Si forms eutectic alloy at a composition of 11.7 at% Si and temperature of 577°C . During solidification of Al-Si eutectic alloy, α -Al and Si crystallizes simultaneously from the liquid and thereby forming eutectic microstructure that is characterized by coarse Si – phase dispersed in α -Al (solid solution of Al and Si) matrix. The Si can crystallize in the form of discrete particles or needle shaped crystals or sheets or flakes. The shape as well as size of the Si-phase dictates the mechanical properties of the Al-Si eutectic alloys. Coarse Si-particles, flakes or needles are found to have detrimental effect on the mechanical properties (strength, ductility etc). Therefore, these alloys are modified by adding Na, Sr in minute quantities (up to 0.05 at %). The shape, size, distribution of Si-phase can be modified by addition of such minute quantities of Na or Sr during casting of Al-Si eutectic alloys. The microstructure of the modified alloys becomes finer than unmodified and thus leads to improvement in mechanical properties. The mechanism of such modification is still controversial.

Pb-Sb phase diagram consists of two terminal solid solution (α and β) and a eutectic point at a composition of 17.8 at% Sb 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% Sb to pure Sb) 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.

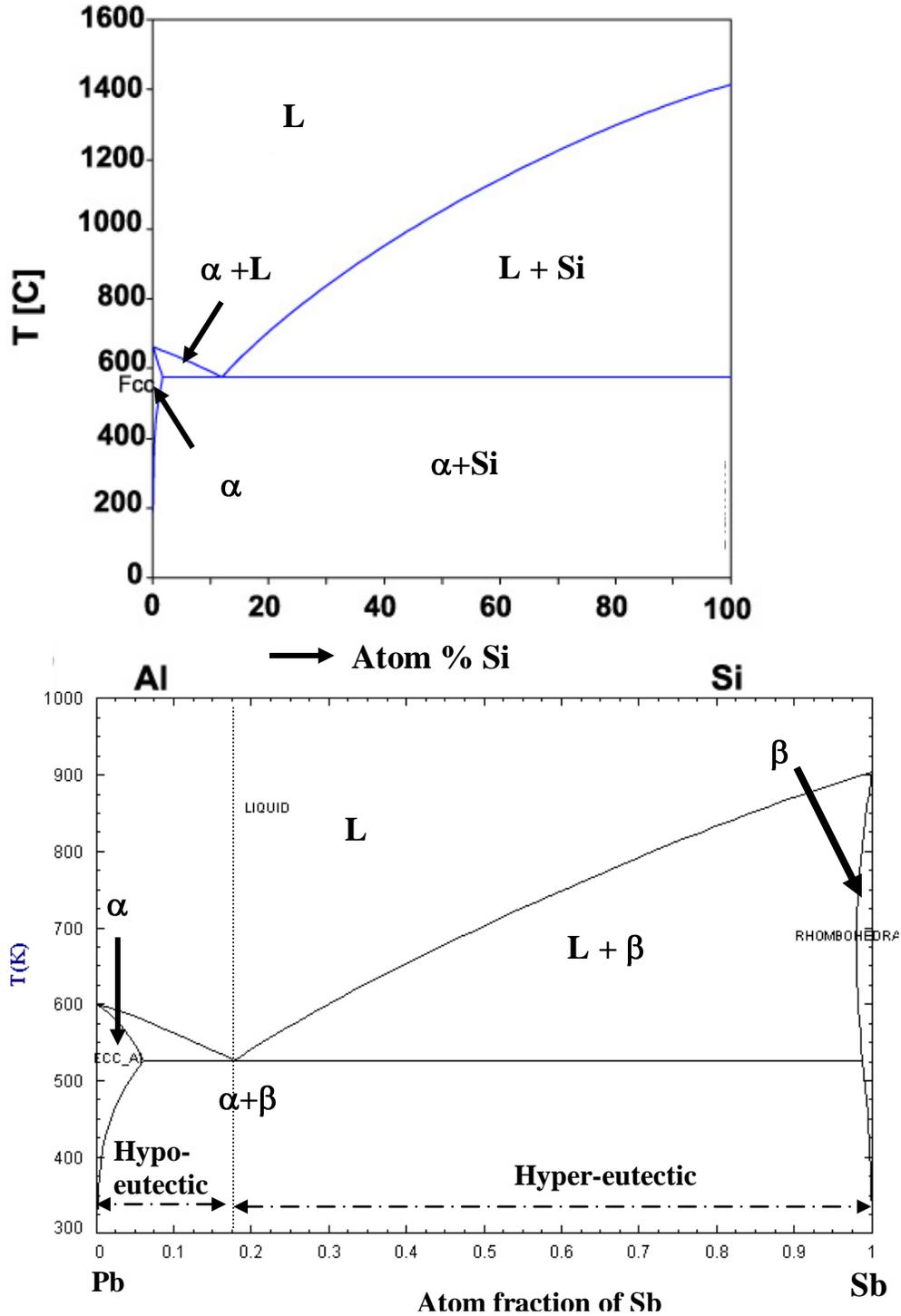


Figure 2: Phase diagrams of Al-Si and Pb-Sb binary systems

LABORATORY EXERCISE

1. Materials:

Al-Si (unmodified), Al-Si (modified), Pb-6wt%Sb (HYPO-EUTECTIC), Pb- 11.1 wt%Sb (EUTECTIC) and Pb- 20 wt%Sb (HYPO-EUTECTIC)

2. Procedure:

- (a) Polish samples using emery papers of 1/0, 2/0, 3/0, 4/0 followed by cloth polishing using 5 μm Al_2O_3 slurry.
- (b) No etchant is required for Al-Si and Pb-Sb samples
- (c) Look at the sample in optical microscope

Report the following

1. Draw neatly the microstructure of all the samples as observed under optical microscope
2. Machines and materials used
3. Mention why no etchant is required for revealing microstructure of these specimens

SET I: EXPERIMENT 3

Fe –C ALLOYS: STEELS: 0.2wt%C, 0.6 wt%C, 0.8wt%C, 1.2 wt%C, 0.7wt%C QUENCHED FROM 850⁰C, 0.7 wt% QUENCHED AND TEMPERED

Objectives

To study and observe microstructure of carbon steels with different chemical compositions and processing conditions.

Background

We shall now consider alloys of Fe and C in our experiment. We will look at microstructure of Fe-C alloys, which undergoes solid-state transformation such as eutectoid transformation. 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 constitute the greatest tonnage of alloy used by man. Secondly, solid-state phase changes in Fe-C phase diagram are varied and very interesting. Furthermore, solid-state phase changes in Fe-C system in much respect similar to those occur in other alloy system. These are the reason why Fe-C phase diagram and the microstructure in carbon steels have been studied so extensively.

Let us first look at the Fe-rich portion of classic Fe-C phase diagram. You can consult textbooks for evolution of the phase diagram, which is of utmost importance for ‘making, shaping and treating’ of steels and cast iron. Figure 3 shows the phase diagram. As you can observe that the phase diagram is not a complete one (unlike earlier cases), and it is only plotted for concentration of carbon less than 6.67 wt%, which is corresponding to the composition of cementite, Fe₃C. Cementite is an intermetallic compounds having negligible solubility limits with orthorhombic crystal structure. This phase diagram depicts a number of invariant points. However, for the sake of the experiments, we will consider only the ‘eutectoid’ point, which is marked on the diagram. If we consider an alloy with 0.8 wt% C and heat it to 950⁰C (1223K), it forms single-phase austenite (γ) having fcc crystal structure. γ 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) will have primary α and pearlite in the microstructure because on cooling such a steel from γ region will deposit α first until the composition of the remaining γ reaches 0.8 wt% at temperature of 723⁰C below which pearlite forms. Therefore, microstructure of hypoeutectoid steels will consists of preeutectoid α with lamellar pearlite in varying proportions depending on the alloy composition. Similarly, Fe-C alloy compositions more 0.8 wt% C are termed as hypereutectoid steels. The microstructure of such steels consists of preeutectoid Fe₃C with lamellar pearlite.

The microstructure of steels depends on heat-treatment conditions. If we heat a steel with 0.7 wt% 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 transform 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 microstructure shows twin laths in different orientations. As stated, martensite is a metastable phase and it is very hard but brittle. Therefore, heating at high temperature (300 -500°C) leads to transformation of martensite to various other carbides and ferrites, which reduce its brittleness. This treatment is called tempering. The tempered microstructure will contain various carbides precipitated from the parent martensite in α -ferrite matrix.

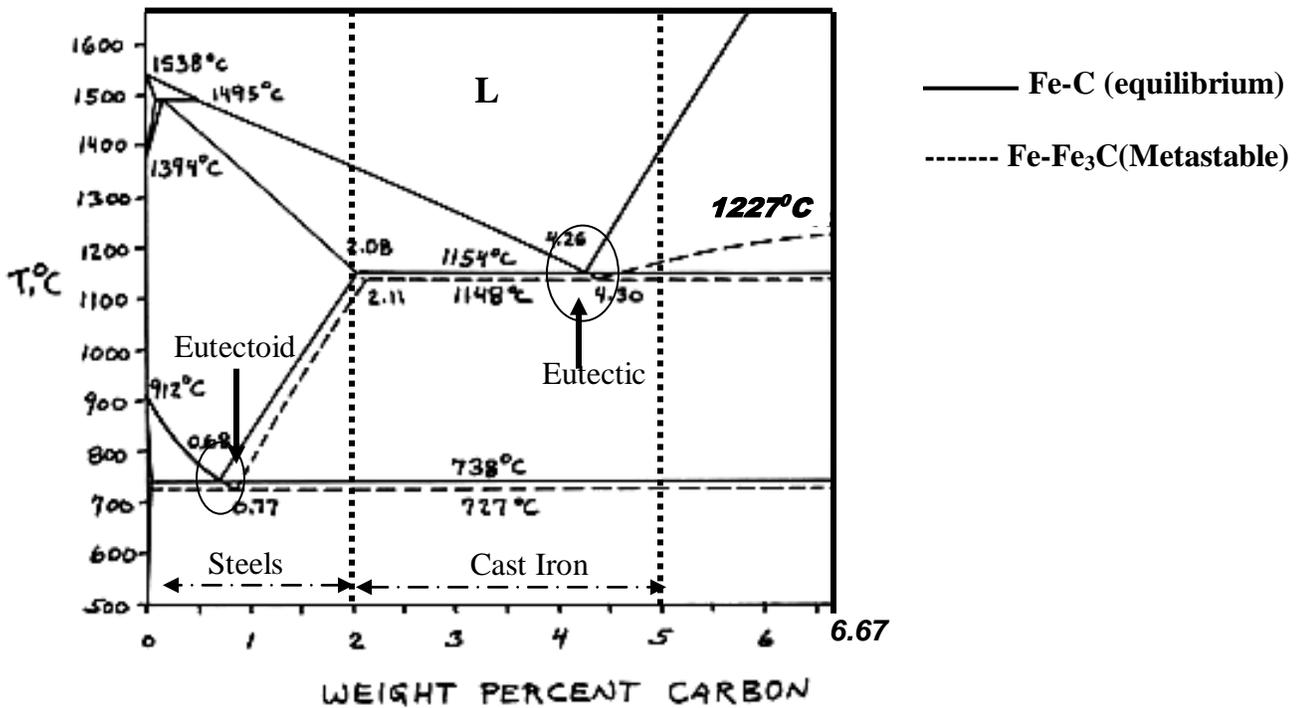


Figure 3: Fe-C and Fe-Fe₃C phase diagrams. Fe₃C is a metastable phase where as the stable phase is graphite. Fe₃C contains 6.67 wt% carbon and thus the phase diagram is reported up to 6.67 wt% carbon. The phase diagram is basis for steel and cast iron. Two important reactions are marked on the diagram.

LABORATORY EXERCISE

1. Materials:

Steels: 0.2wt%C, 0.6 wt%C, 0.8wt%C, 1.2 wt%C, 0.7wt%C quenched from 850⁰C, 0.7 wt% quenched and tempered

2. Procedure:

- (a) Polish samples using emery papers of 1/0, 2/0, 3/0, 4/0 followed by cloth polishing using 5 μm Al₂O₃ slurry.
- (b) Etch using proper chemical etchant (Nital: 4 vol% HNO₃ in methanol)
- (c) Look at the sample in optical microscope

Report the following

1. Draw neatly the microstructure of all the samples as observed under optical microscope
2. Machines and materials used
3. Etchants used and optimum etching time required to obtain reasonably good microstructure

SET I: EXPERIMENT 4

ALLOY STEELS: 0.23 wt% C AND 0.85 % Mn ALLOY STEELS, FREE CUTTING STEELS*, 18/8 AUSTENITIC STAINLESS STEELS, HIGH SPEED TOOL STEELS

Objectives

To study and observe microstructure in various alloy steels

Background

In this experiment we will study the microstructure of alloys steels. Plain carbon steels are Fe-C alloys in which properties are primarily derived from the presence of carbon. Some incidental elements such as Mn, Si, P,S are present in very minute quantities due to the method of making steels, not to modify the mechanical properties. Alloy steels are those steels in which one or more alloying elements are intentionally added to plain carbon steels to enhance or induce some properties. Even though plain carbon steels constitute the largest tonnage of metallic material used by man, they have certain limitations, such as low hardenability (ability to form martensite), low corrosion and oxidation resistance, poor high temperature properties etc. These limitations are overcome by use of alloy steels. The presence of different alloying elements not only enhances the properties of carbon steels, but also induces new properties. Alloy steels can be classified into different types depending on alloying elements added. Classification of alloying elements is done based on whether they stabilize ferrite or austenite.

We shall observe microstructure of some specific steels. Mn additions stabilize austenite phase. Mn also increases hardenability. Mn forms MnS particles in the form of globules and does not allow detrimental FeS to form. Free cutting steels contain 1.5 wt%Mn and 0.2 wt% S. The presence of large number of MnS globules renders the steel cut easily. This kind of steel is known as free cutting steel.

The most important alloy steel is stainless steel. Stainless steels are stainless as they have minimum 11.5 wt% Cr, which having more affinity to O₂ than iron, forms very thin, protective and stable oxide (preferably Cr₂O₃) film on the surface. This film is continuous, impervious and passive to stop further reaction between steels and atmosphere. Stainless steels can be different types depending on the microstructure: ferritic, austenitic, martensitic, duplex types. We will study 18/8 stainless austenitic steels, containing 18 wt% Cr and 8 wt% Ni. The microstructure shows grains of austenite.

High speed tool steels contain other alloying elements such as W,V,Ni, Cr,Mo etc. The microstructure shows presence of various carbides, such as WC, VC, Cr₂₃C₂, (Fe,Mo)₃C in a matrix of ferrite, pearlite or tempered martensite. The presence of these carbide in

the microstructure provides retaining hardness at elevated temperature attained during high speed cutting because of the fact that WC, VC, Cr₂₃C₂ have very high melting temperatures. These carbides are present in the form of fine scale precipitates and thereby provide resistance against deformation.

LABORATORY EXERCISE

1. Materials:

Alloy steels: 0.23 wt% C and 0.85 % Mn alloy steels, Free cutting steels *, 18/8 austenitic stainless steels, high speed steels

2. Procedure:

(a) Polish samples using emery papers of 1/0, 2/0, 3/0, 4/0 followed by cloth polishing using 5 μm Al₂O₃ slurry.

(b) Etch using proper chemical etchant (Nital for Mn steel and High speed steels, FeCl₃ for Free cutting steels and Marbie's reagent for 18/8 steel)

(c) Look at the sample in optical microscope

Report the following

1. Draw neatly the microstructure of all the samples as observed under optical microscope
2. Machines and materials used
3. Etchants used and optimum etching time required to obtain reasonably good microstructure

SET I: EXPERIMENT 5

CAST IRONS: GREY CAST IRON, WHITE CAST IRON, MALLEABLE IRON WITH FERRITIC MATRIX, MALLEABLE IRON WITH PEARLITIC AND FERRITIC MATRIX AND NODULAR IORN

Objective

To study and observe microstructure in different types of cast iron

Background

Cast iron is also Fe-C alloy with carbon content varying from 2.1 to 4 wt% depending on type of cast iron. Cast irons contain appreciable amounts of silicon, normally 1-3 %wt, and consequently these alloys should be considered ternary Fe-C-Si alloys. Despite this, the principles of cast iron solidification are understood from the binary iron-carbon phase diagram, where the eutectic point lies at 1154 °C and 4.3-wt% carbon. Presence of Si in the cast iron makes cementite unstable and thereby promoting formation of graphite in different shapes and sizes with the remaining iron-rich matrix becoming ferritic, pearlitic depending on residual carbon present (**Refer to Figure 3 for Fe-C phase diagram**). Since cast iron has nearly this composition, its melting temperature of 1150 to 1200 °C is about 300 °C lower than the melting point of pure iron. With its low melting point, it has good castability. Cast iron can be cast into any complicated shapes. Cast irons are brittle, except for malleable and nodular iron. However, the presence of graphite gives them machinability and damping capacity. The cast iron is the 'work horse' of automobile industry.

There are different varieties of cast irons. When carbon concentration is low (~2-2.4 wt%) and Si concentration is about 1-1.5 wt% white cast iron forms during solidification. With faster cooling and lower Si concentration, carbon precipitates out as metastable cementite (Fe_3C) instead of graphite. The cementite, which precipitates from the melt, forms as relatively large particles, usually in a eutectic mixture where the other phase is austenite (which on cooling might transform to pearlite or martensite). These eutectic carbides are much too large to provide precipitation hardening (as in some steels, where cementite precipitates might inhibit plastic deformation by impeding the movement of dislocations through the ferrite matrix). Rather, they increase the bulk hardness of the cast iron simply by virtue of their own very high hardness and their substantial volume fraction, such that the bulk hardness can be approximated by a rule of mixtures. In any case, they offer hardness at the expense of ductility and toughness.

In case, Si concentration is sufficiently high (about 2 wt %), it causes carbon to crystallize as graphite from the liquid during solidification with the remaining liquid becoming Fe-rich solidifying as ferrite. Therefore, the microstructure of grey iron shows the presence of flakes of graphite in a ferritic matrix. Grey cast iron has high strength, hardness. However, it has low toughness because of the fact that graphite remains in the

form of flakes, acting as areas of stress concentration and thereby causing the material to fail in service. Therefore, it is desirable to have graphite crystallized as spherical nodules, instead of long flakes. Addition of Mg, Ce into the melt during solidification leads to formation of spherical nodular graphite with remaining matrix solidifying as ferrite or pearlite depending on carbon concentration. This type of cast iron is called nodular iron or spheroidal graphite iron because of the shape of the graphite. Due to their lower aspect ratio, spheroids are relatively short and far from one another. They also have blunt boundaries, as opposed to flakes, which alleviates the stress concentration problems faced by grey cast iron. Nodular iron has high strength, ductility as well as toughness and is used extensively in automobile industry.

There is another category of cast iron called malleable iron. Malleable iron starts as a white iron casting, that is then heat treated at about 900 °C for about 2 days. Graphite separates out much more slowly in this case, so that it forms irregularly shaped graphite particles rather than flakes, called rosettes. Because of the fact that malleable iron forms by solid-state heat-treatment, the graphite forms as irregular spheroids, unlike the nodular iron. In general, the properties of malleable cast iron are more like mild steel. There is a limit to how large a part can be made in malleable iron, since it is made from white cast iron.

LABORATORY EXERCISE

1. Materials:

Cast irons: Grey cast iron, White cast iron, Malleable iron with ferritic matrix, Malleable iron with pearlitic and ferritic matrix and Nodular iron

2. Procedure:

- (a) Polish samples using emery papers of 1/0, 2/0, 3/0, 4/0 followed by cloth polishing using 5 μm Al_2O_3 slurry.
- (b) Etch using proper chemical etchant (Nital)
- (c) Look at the sample in optical microscope

Report the following

1. Draw neatly the microstructure of all the samples as observed under optical microscope
2. Machines and materials used
3. Etchants used and optimum etching time required obtaining reasonably good microstructure.

SET I: EXPERIMENT 6

PERITECTIC AND MONOTECTIC ALLOYS: 60:40 ($\alpha+\beta$) BRASS, 70:30 (α) BRASS, Cu- 10wt%Sn, Cu- 36wt% Pb, Cu- 50 wt% Pb*

Objective

To study and observe the microstructure of peritectic and monotectic alloys.

Background

The next category of alloy we will study is peritectic and monotectic alloys. Peritectic alloys occupy an outstanding position among the engineering materials. Many technically important alloy systems such as steels, Cu alloys (Brass and Bronz), 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 is given by $\alpha + L \rightarrow \beta$ 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 4 shows phase diagram of Cu-Zn binary system. 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 alloys Cu:Zn =70:30 and Cu:Zn =60:40. The microstructure of the first alloy will show predominant presence of the α phase whereas the second alloy will reveal presence of both the α and β phases because of incomplete peritectic reaction.

Similarly, Cu-Sn alloys (popularly known as Sn bronze) undergo a peritectic reaction. These alloys contain other impurities, such as phosphorous in phosphorous bronze and zinc in coinage bronze and Gun metal etc. They are strong and tough and find application in myriad of industrial applications, such as springs, turbines, bearings, clips as well as household applications. Cu-Sn phase diagram is shown in figure 4.

The monotectic alloys show promising applications in tribology of automotive components. As a part of your laboratory exercise, you will observe microstructure of Cu-Pb alloys. Cu and Pb are immiscible in liquid and solid. Figure 5 shows the Cu-Pb binary phase diagram. The compositions of alloys are marked on the figure. As you can observe during cooling such an alloy, liquid, L separates into two liquids (Cu-rich and Pb-rich) below monotectic temperature, T_M . One of the liquids (Cu-rich) solidifies first and traps the other liquid (Pb-rich). The Pb-rich liquid then solidifies as droplets. Therefore, the microstructure shows the presence of Pb-droplets in Cu matrix.

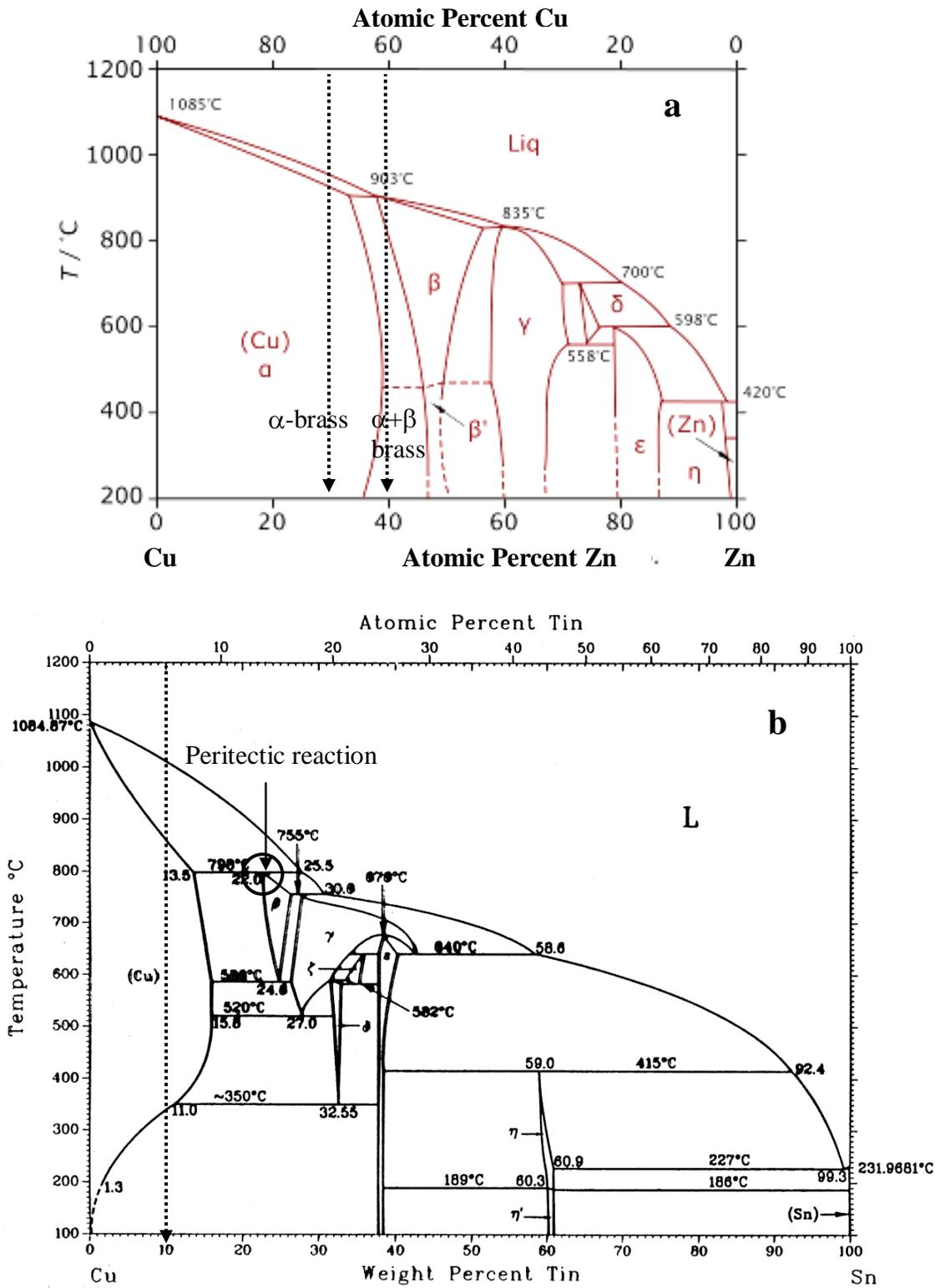


Figure 4: Phase diagrams of peritectic alloys: (a) Brass: Cu-Zn and (b) Bronze: Cu-Sn

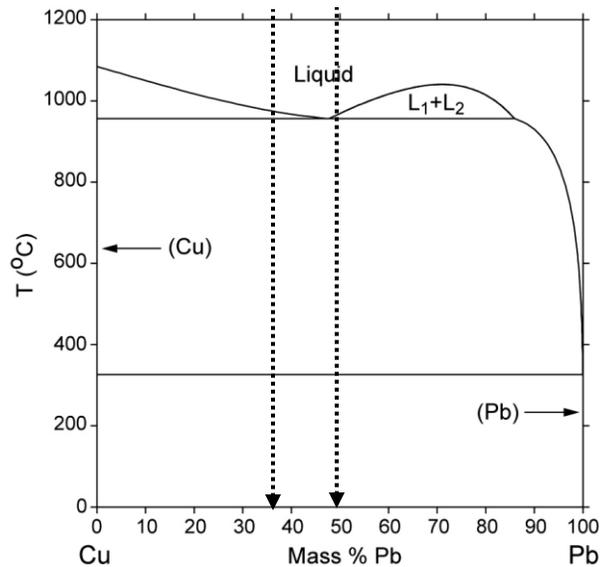


Figure 5: Phase diagram of monotectic alloy: Cu-Pb

LABORATORY EXERCISE

1. Materials:

Peritectic alloys: 60:40 ($\alpha+\beta$) Brass 70:30 (α) Brass, Cu- 10wt% Sn Bronze

Monotectic alloys: Cu- 36wt% Pb, Cu- 50 wt% Pb*

2. Procedure:

- (a) Polish samples using emery papers of 1/0, 2/0, 3/0, 4/0 followed by cloth polishing using 5 μm Al_2O_3 slurry.
- (b) Etch using proper chemical etchant (FeCl_3 solution)
- (c) Look at the sample in optical microscope

Report the following

1. Draw neatly the microstructure of all the samples as observed under optical microscope
2. Machines and materials used
3. Etchants used and optimum etching time required to obtain reasonably good microstructure.

SET II: EXPERIMENT 1

MEASUREMENT OF VOLUME FRACTION, SURFACE AREA IN TWO-PHASE AND GRAIN SIZE IN SINGLE-PHASE MATERIALS

Objective:

Understanding the basic tools for surface and volume fraction of phases

Background

Quantitative metallography plays very important role in materials science and engineering. It can provide quantitative relationships between processes, microstructures and physical as well as mechanical properties and supply the first hand data necessary to establish a reasonable mathematical model for microstructure. It enables the costly trial and error method to be replaced by a scientifically based experimental procedure resulting in the improvement of known materials and the development of new ones. The study on quantitative metallography is as known 'Stereology'. You have observed microstructures of different materials, such as steels, cast iron, eutectic, peritectic and monotectic alloys. You must have observed single phase as well as two-phase microstructures. In many cases, we need to have information of the grain size, surface area and volume fraction of phases present in the microstructure. During this laboratory practice, you will be exposed to some of the basic quantitative metallographic techniques, such as measurement of volume fraction and surface area for two-phase material as well as grain size of single-phase material. Grain size is a very important parameter to characterize the microstructure. It can set up the quantitative relationship between the microstructure and mechanical properties. A famous example is the Hall-Patch equation:

$\sigma = \sigma_0 + kd^{-\frac{1}{2}}$, where σ is flow stress, σ_0 is lattice friction stress, k is constant and d is grain size. Therefore, grain size measurement can provide us a good estimate of yield strength of different material. Grain size measurement also provides information regarding ductile-to-brittle transition, creep behaviour of materials etc. Similarly, the precipitation hardening due to presence of second phase particles embedded in a matrix depends on the volume fraction of the precipitates, precipitate size, inter-precipitate distance, interfacial area, mean free path etc. There are many ways one can measure grain size in a single-phase microstructure. You will use the method known as 'Linear Intercept method'. Figure 6 depict the way the method is used. You draw a straight line on the single-phase microstructure and then count number of intersection the line makes with the grain boundaries. By knowing the length of line you can easily calculate the average grain size of the material. By carrying out such measurement for large number of grains (approximately 80), you can get a better statistics.

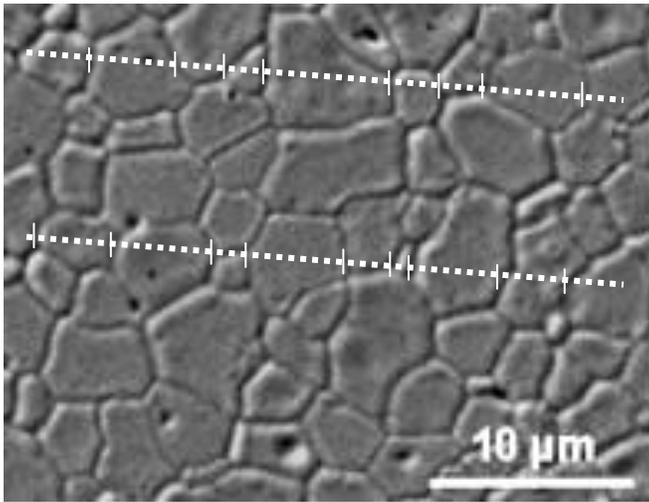


Figure 6: Linear Intercept method.

For measurement of surface area of a single-phase material (grain boundary area in unit volume), similarly, we will draw a reference straight line on the microstructure (as shown on the figure above) and count the number of intersection the line makes with the grain boundaries. The surface area can then be calculated using the formula, $S_V = 2.N$, where N is the number of intersection the line makes with the grain boundaries. The factor 2 is due to that line makes two intersections per grain. For a two-phase material, measurement of interfacial area requires counting the number of intersections with two phase boundaries.

To measure the volume fraction of the phases in a two-phase microstructure, we will adopt a method popularly known as systematic point counting. The most commonly measured property of a two-phase (or multi-phase) is the volume fraction of phases. The volume fraction can be estimated using plane, line or point probes. The simplest one relies on point probe. In a nutshell, the method can be described as follows. Figure 7 shows a two-phase microstructure consisting of α and β phases. The population of the points in microstructure is probed by a $n \times n$ (8×8) grids of points superimposed on the microstructure. Count the grid points that hit the β phases. This count is repeated on a series of fields on the set of section. The mean point fraction, $\left(\frac{\bar{P}_\beta}{P_T} \right)$, where \bar{P}_β is the points hitting the β phase and P_T is the total number of points, represents the volume fraction of the β phase. As you can understand the simplest method can be applied to even microstructure consisting of 3 or more number phases.

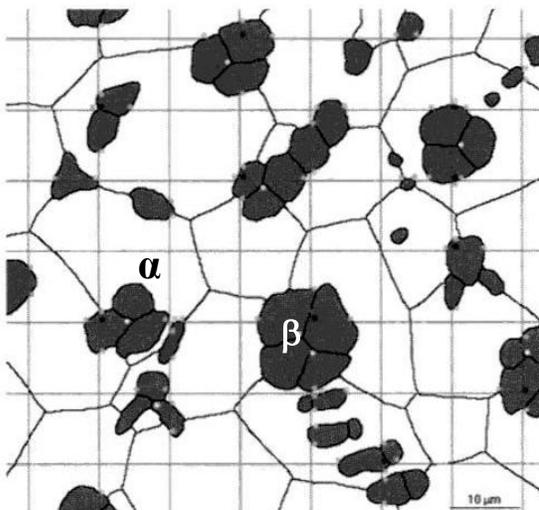


Figure 7: Measurement of volume fraction of phases using systematic point counting method.

LABORTAORY EXCERCISE

1. Materials:

1. Pure iron sample for the measurement of grain size and grain boundary surface area
2. α - β brass for the measurement of the volume fraction of phases

2. Procedure:

- (a) Polish samples using emery papers of 1/0, 2/0, 3/0, 4/0 followed by cloth polishing using 5 μm Al_2O_3 slurry.
- (b) Etch using proper chemical etchant (pure iron: Nital and Brass : FeCl_3)
- (c) Look at the sample in optical microscope at a magnification of 100
- (d) For measurement of grain size of single phase material, draw a reference line of fixed length and measure the number of intersections with grains and calculate the average grain size. Repeat the process for large number of grain (at least 80) to obtain a better statistics.
- (e) For measurement of surface area, use (d)
- (f) For measurement of volume fraction of phases for a two-phase material, place a (nxn) grid on the microstructure and count the points hitting both the phases and use the formula mentioned in the text to obtain the volume fraction. (**Check the calibration of the microscope carefully before taking measurements!**)

Report the following

1. Draw neatly the microstructure of all the samples as observed under optical microscope with the reference line on the microstructure.
2. Machines and materials used
3. Calculation of grain size, surface area of single-phase material and volume fraction of the two-phase material
4. Precautions needed to taken to obtain
5. Comment on ease on usage of software for such calculations

SET II: EXPERIMENT 2

MEASUREMENT OF PARTICLE SIZE AND ITS DISTRIBUTION

Objective

To measure particle size and size distribution of powder using metallographic technique

Background

In many metallurgical systems, precipitates or dispersoids of different sizes form during processing of materials. For example, in case of monotectic alloys, minor phase (Pb in case Cu-Pb alloy) can be obtained as dispersoid within the major phase or matrix. In case of precipitation-hardened alloys (Al-Cu, Al-Li alloys), precipitates (GP zones, θ'' , θ' , θ etc) of different sizes are formed in a matrix. In steels, certain heat-treatments are carried out to obtain spherical carbides (Fe_3C). This kind of heat-treatments is known as Spheroidization annealing. The mechanical, electrical as well as electronic properties of the materials depends the size and distribution of the second phase, i.e., dispersoids, precipitates or spheroids. Spheroidization annealing involves heating the steel below the eutectoid temperature (723°C) for sufficiently long times (approx. 30 hours). The result is a structure of rods or spheres of cementite within primary structure (ferrite or pearlite, depending on which side of the eutectoid reaction you are on). The purpose is to soften higher carbon steels and allow more formability. The properties of these materials depend on size and distribution of the dispersoids or precipitates or spheroids within the primary phase matrix. Therefore, it is imperative to obtain the size and distribution of the second phase within the primary phase matrix.

During this experiment, you will learn the technique of measuring the size and the distribution of second phase particles using optical microscope. The basic principle utilized in this experiment is quite similar to those of previous experiments. The technique utilized in this experiment can be summarized as the following. Figure 8 shows microstructure of spheroidized perlite in hyper-eutectoid steel (~1% C) with Fe_3C near spherical particles in the ferrite matrix. This microstructure is obtained upon heat-treating the steel at about 700°C for 30 hours. A reference line of specified length 'L' is drawn on the microstructure. The size of each particle (ΔL_i , $i=1, 2, 3, \dots$) intercepted by the straight line is measured by a micrometer attached to the optical microscope. Such measurement is carried out for sufficiently large number (approximately 80) particles and the average size of the particle is calculated to obtain a good statistics.

In order to obtain the distribution of particle size, we follow the following procedure. The raw data obtained during the measurement, (ΔL_i , $i=1, 2, 3, \dots$) is arranged in ascending order of ΔL . The sample size (the largest size to smallest size) range is divided into several convenient size intervals based on the data. The number of particles measured in each size intervals is counted. The number of particle in each interval is plotted as a function of particle size.

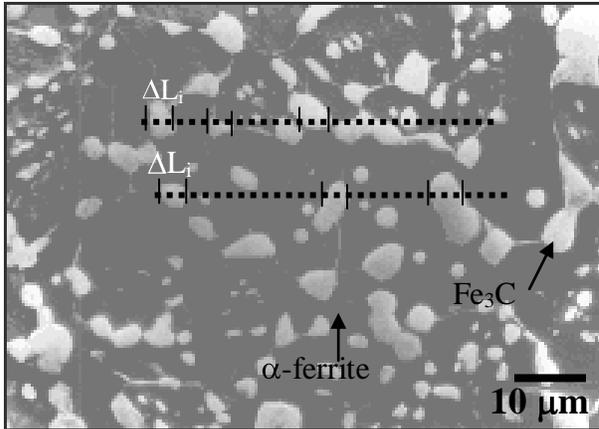


Figure 8: Spheroidized pearlite in hyper -eutectoid steel (1 wt % C) with Fe_3C near spherical particles in the ferrite matrix.

LABORATORY EXERCISE

1. Materials:

Plain carbon steel containing 0.4 wt% C processed by spheroidization annealing

2. Procedure:

- (a) Polish samples (annealed, normalized and quenched) using using emery papers of 1/0, 2/0, 3/0, 4/0 followed by cloth polishing using $5\ \mu\text{m}$ Al_2O_3 slurry.
- (b) Etch using proper chemical etchant (2 % Nital)
- (c) Look at the sample in optical microscope
- (d) Follow the above mentioned procedure

Report the following

1. Plot particle size distribution and calculate the mean particle size
2. Draw microstructure giving details of the technique
3. Precautions needed to taken during measurement and drawbacks of this technique

SET II: EXPERIMENT 3

HEAT-TREATMENT OF 0.6 wt% C STEELS: ANNEALING (FURNACE COOLED), NORMALIZING (AIR COOLED), WATER QUENCHED, OIL QUENCHED

Objective

To study and observe the microstructure of 0.6 wt% C steel processed under different experimental conditions

Background

In this experiment we will study the microstructure of plain carbon steel containing 0.6 wt % C, processed under various conditions. Plain carbon steels are Fe-C alloys in which properties are primarily derived from the presence of carbon as interstitial solute. Some incidental elements such as Mn, Si, P, S are present in very minute quantities due to the method of making steels, not to modify the mechanical properties. You have already observed microstructure of 0.2 and 0.6 wt % carbon steel in Experiment 3 in Set I. Refer to Fe –C phase diagram (Figure 3) to understand the microstructure of plain carbon steels. The microstructure of 0.6 wt % C steel normally consists of grains of α -ferrite with pearlite (approx. 75 volume %) at the ferrite grain boundaries. The microstructure of steels also depends on heat-treatment conditions, i.e, cooling rates. During the course of this experiment, you will observe change in the microstructure of 0.6 wt% C steel under different cooling conditions. During annealing, the steel sample is heated to high temperature ($\sim 950^{\circ}\text{C}$ in the austenite region). After keeping the sample at this temperature for some time (2-3 hours), the furnace power is switched off and sample is allowed to cool inside the furnace. Obviously, the cooling rate will very low (0.5 to 1 K/min) because the furnace cools very slowly. During normalizing, the sample is heated to high temperature and held for some time (2-3 hours) as before and then removed from the furnace to cool in air. In this case, the cooling rate will be higher (10-100 K/min.) than that of annealing. Quenching means rapid cooling (cooling rate $\sim 10^4$ K/sec). Quenching of sample is done by rapidly dropping the hot sample in water or oil. Water has higher thermal conductivity and therefore, the heat transfer from the sample to water is faster than that to oil. Therefore, quenching rate can be varied using different types of quenching media. In a nutshell, we vary the cooling rate of the sample and observe the microstructural change in the steel sample containing 0.6 wt% carbon. During annealing the primary ferrite grains grows substantially before the sample temperature reaches eutectoid temperature ($\sim 723^{\circ}\text{C}$) below which pearlite forms. Normalizing involves faster cooling as compared to annealing and therefore, grain size of primary ferrite is smaller than that for annealing. The smaller grain size of ferrite helps in pearlite formation because of presence of larger grain boundary area. Quenching of the steels in water leads to formation of a new phase known as martensite which is very hard and brittle. This new phase is a metastable one which forms bypassing the eutectoid reaction.

You can understand that how we, the material engineers can vary the properties of the steels by adopting different heat –treatment conditions. This is the unique characteristics of ours! Measurement of hardness using Rockwell hardness tester allows us to compare the hardness of 0.6 wt% C plain carbon steel processed under different heat-treatment

conditions. Hardness measurements provide us elegant way of measuring mechanical property using a small piece of sample.

LABORATORY EXERCISE

1. Materials:

Plain carbon steel containing 0.6 wt%C

2. Procedure:

- (a) Polish samples (annealed, normalized and quenched) using emery papers of 1/0, 2/0, 3/0, 4/0 followed by cloth polishing using 5 μm Al_2O_3 slurry.
- (b) Etch using proper chemical etchant (2% Nital)
- (c) Look at the sample in optical microscope
- (d) Measure the hardness on Rockwell Scale, HRC using diamond cone and 150 kg load.

Report the following

1. Draw neatly the microstructure of all the samples as observed under optical microscope
2. Machines and materials used
3. Etchants used and optimum etching time required to obtain reasonably good microstructure.
4. Compare the hardness with different heat-treatment.

SET II: EXPERIMENT 4

PHASE TRANSFORMATION OF EUTECTIC Pb-Sn ALLOY

Objective

To study the melting and solidification of $\text{Pb}_{38.1}\text{Sn}_{61.9}$ eutectic alloy using Differential Thermal Analyzer (DTA)

Background

During the following three experiments, you will be learning about determination of phase diagram. In this experiment you will study the melting and solidification of a solder alloy, which is a Pb-Sn eutectic alloy. Let us first discuss about the Pb-Sn phase diagram. We shall use the Pb-Sn phase diagram for last three experiments. Figure 9 shows Pb-Sn binary alloy phase diagram. The phase diagram shows that there are two terminal solid solutions, Pb-rich α -phase and Sn-rich β -phase with a eutectic reaction at a temperature of 183°C and composition of Pb – 61.9 wt % Sn. This eutectic phase has the lowest melting temperature in the phase diagram. The solder alloy is widely used in semiconductor industry for soldering applications. We also use it for day-to-day soldering needs.

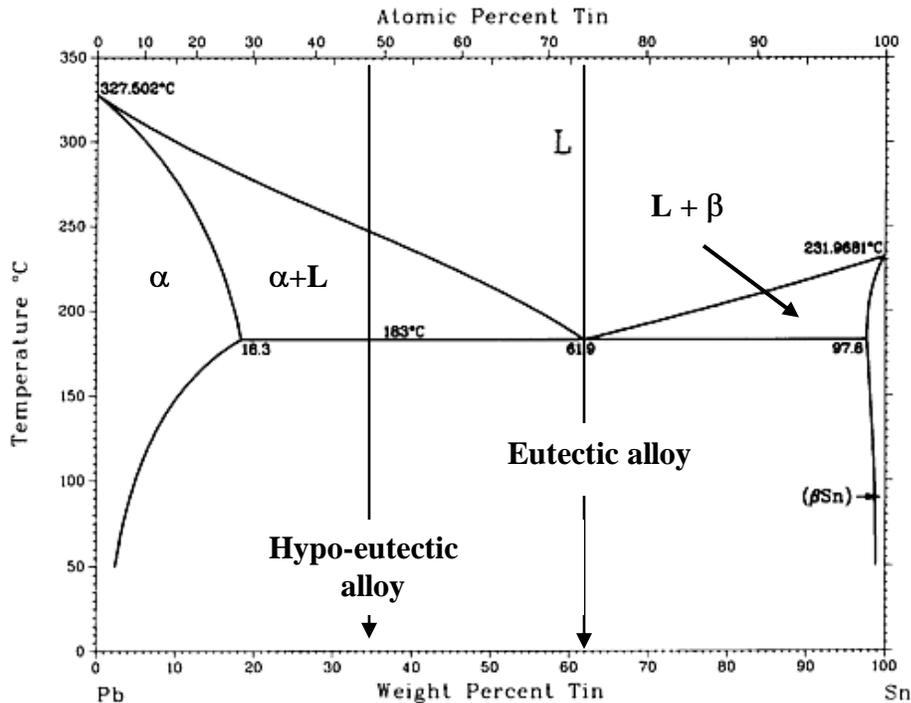


Figure 9: Pb-Sn binary phase diagram

During cooling such an alloy from the molten state, it undergoes the eutectic reaction, $L \rightarrow \alpha + \beta$, releasing the latent heat for solidification (exothermic). On the other hand,

during heating, the opposite reaction, $\alpha + \beta \rightarrow L$ takes place, which absorbs latent heat required from the surrounding (endothermic).

We shall study the phase transformation (melting and solidification) of the alloy using an apparatus known as Differential Thermal Analyzer or DTA. DTA analysis is a technique in which temperature of a sample is compared with that of an inert reference material during a programmed change of temperature. The temperature of the sample and reference should be the same until some thermal event occurs in the sample, in which case sample temperature either lag (if the change is endothermic) or leads (if the change is exothermic) the reference temperature. During the experiment, both the sample and reference are heated at constant rate and the sample temperature is monitored as a function of time (differential temperature). The plots remains linear until an endothermic event (such as melting) happens at $T = 183^{\circ}\text{C}$. The sample temperature remains constant until event is completed. It then increases rapidly to catch up with temperature required by the programmer. The thermal event in the sample thus appears as a rather broad deviation from sloping baseline. Similarly, the sample temperature is monitored during cooling of molten alloy. The plot remains linear until solidification begins (solidification begins at temperature slightly below 183°C because of nucleation of the solid phase require undercooling). The sample temperature remains same during the solidification event after which sample temperature drops. Therefore, solidification event can be observed as a deviation from the slopping baseline.

LABORATORY PRACTICE

1. Materials:

Pb-Sn eutectic alloy (common solder)

2. Procedure: The experimental steps in brief:

- (a) Heat the alloy inside the DTA to 200°C at a predetermined rate (10K/min.) and hold the liquid for 10 minutes.
- (b) Cool the liquid alloy at a determined rate (10K/min.).
- (c) Record the temperature of the sample during cooling

Report the following

1. Plot the change the temperature of the sample (T) with change in temperature (ΔT) during cooling.
2. Machines and materials used
3. Precautions need to taken during the experiment

SET II: EXPERIMENT 5

COOLING CURVE OF A Pb-Sn EUTECTIC ALLOY

Objective

To measure cooling curve of a Pb-Sn eutectic alloy using thermocouple

Background

In the last experiment, you have studied the melting and the solidification behaviour of Pb-Sn eutectic alloy using DTA. In this experiment, you will measure the cooling curve of Pb-Sn as a part of your study on determination of phase diagram of a simple eutectic alloy system. $\text{Pb}_{38.1}\text{Sn}_{61.9}$ alloy sample (solder alloy) is heated to about 200°C to obtain fully molten alloy. The temperature of the sample is monitored using a thermocouple during cooling of the alloy to room temperature. The temperature of the sample is plotted as function of time. The sample temperature drops continuously as function until it reaches 183°C . At 183°C , the sample undergoes solidification by forming eutectic phase mixture from liquid, $L \rightarrow \alpha + \beta$ (refer to Pb-Sn binary phase diagram in Figure 9). The sample temperature remains constant until the whole liquid solidifies and then the sample temperature is dropped again. In real situation, cooling curve looks a bit different from the above scenario. In fact, solidification does not begin at 183°C , because nucleation of solid phases from the liquid requires undercooling (drop in temperature) to provide sufficient driving force (change in volume free energy, G). Therefore, the temperature of the sample drops slightly below 183°C before the solidification begins. As soon as solidification begins, latent heat is released into the sample. This leads to heating up of the sample and therefore, sample temperature increases a bit from the temperature at which solidification begins. Thereafter, the temperature remains constant until whole liquid solidified. Then sample temperature drops again. By measuring the solidification temperature of the sample, you can measure the solidus temperature of the eutectic alloy and this data can then be used to construct the phase diagram. By doing such experiments using alloys of different compositions, we can obtain the phase diagram!

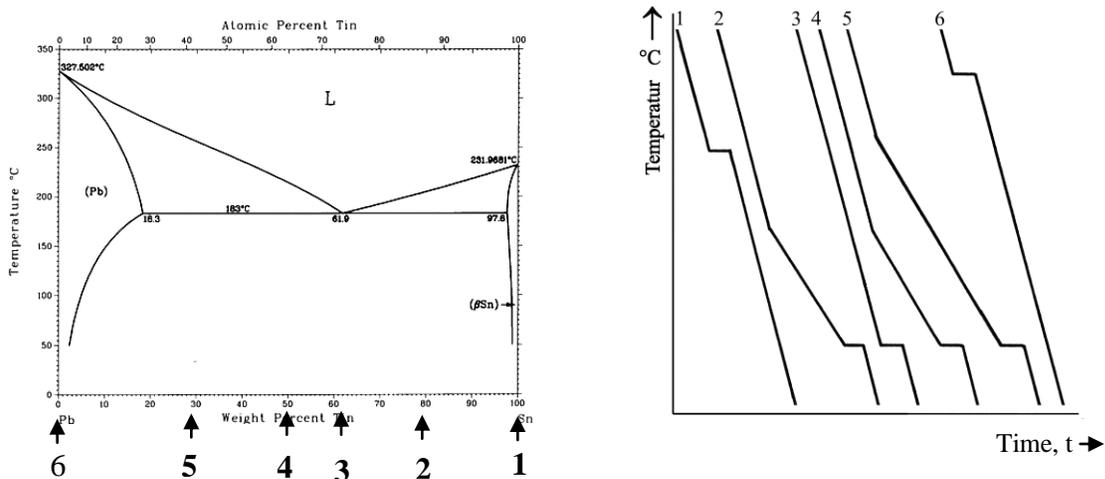


Figure 10: (a) Pb-Sn phase diagram and (b) Typical cooling curves for the alloys shown in (a)

Figure 10 indicates how the phase alloy diagram can be constructed using a set of cooling curves obtained for different alloys compositions ranging from 0wt% Sn to 100%Sn in Pb-Sn alloys. It is evident that these simple experiments can provide us insight into the alloy phase diagram!

For the purpose of the experiment, we shall use a thermocouple of K-type. The thermocouple is a widely used type of temperature sensor. The basic principle of thermocouples is based on the fact that when two dissimilar metals are joined at two ends, a predictable voltage will be generated between the two ends if two ends are kept at different temperatures. The voltage can be related to the difference in temperature between the measuring junction and the reference junction (connected to the measuring device). The selection of the optimum thermocouple type (alloys used in their construction) is based on application temperature, atmosphere, and required length of service, accuracy and cost. In this experiment, you will use a K type Chromel(+)/Alumel(-) thermocouple, which is suitable for measurement of temperature in the range of -200°C to 1200°C in air with precision of $\pm 1.5\text{K}$.

LABORATORY PRACTICE

1. Materials:

Pb-Sn eutectic alloy (common solder): $\text{Pb}_{38.9}\text{Sn}_{61.9}$

2. Procedure: The experimental steps in brief:

- (a) Heat the alloy inside a furnace to 200°C at a predetermined rate (10K/min.) and hold the liquid for 10 minutes.
- (b) Place a thermocouple (Chromel-Alumel K type) to determine the temperature using voltage. Cool the liquid alloy at a determined rate (10K/min.).
- (c) Record the temperature of the sample during heating and cooling as function of time

Report the following

1. Plot the change the temperature of the sample (T) with time (t).
2. Machines and materials used
3. Precautions need to taken during the experiment

SET II: EXPERIMENT 6

COOLING CURVE OF A Pb-Sn HYPO-EUTECTIC ALLOY

Objective

To measure cooling curve of a Pb-Sn non-eutectic alloy using thermocouple

Background

In the last experiment, you have measured the cooling curve of a Pb-Sn eutectic alloy. In this experiment, you will measure the cooling curve of Pb-Sn hypo-eutectic alloy as a part of your study on determination of phase diagram of a simple eutectic alloy system. Hypo-eutectic (as shown in figure 9) alloy sample is heated to about 300⁰C to obtain fully molten alloy. The temperature of the sample is monitored using a thermocouple during cooling of the alloy to room temperature. The temperature of the sample is plotted as function of time. The sample temperature drops continuously as function until it reaches about 250⁰C, at which solidification of Pb-rich α -phase begins. A careful look at the phase diagram will tell you that α -phase do not have a fixed solidification temperature. Rather solidification of the α -phase begins at temperature of ~250⁰C and ends at solidus temperature (183⁰C). Therefore, at 250⁰C the primary α -phase starts forming, the slope of the cooling curve changes. The new cooling curve progresses further until the temperature reaches 183⁰C or eutectic temperature. At 183⁰C, the sample undergoes solidification by forming eutectic phase mixture from liquid, $L \rightarrow \alpha + \beta$ (**Refer to Pb-Sn binary phase diagram in Figure 9 and 10**). The sample temperature remains constant until the whole liquid solidifies and then the sample temperature is dropped again. The resulting cooling curve shows the two stages of solidification with a section of reduced gradient where a single phase is solidifying and a plateau where eutectic is solidifying . By taking a series of cooling curves for the same system over a range of compositions the liquidus and solidus temperatures for each composition can be determined allowing the solidus and liquidus to be mapped to determine the phase diagram.

LABORATORY PRACTICE

1. Materials:

Pb-Sn non-eutectic alloy:

2. Procedure: The experimental steps in brief:

- (a) Heat the alloy inside a furnace to 300⁰C at a predetermined rate (10K/min.) and hold the liquid for 10 minutes.
- (b) Place a thermocouple (Chromel-Alumel K type) to determine the temperature using voltage. Cool the liquid alloy at a determined rate (10K/min.).

(c) Record the temperature of the sample during heating and cooling as function of time

Report the following

1. Plot the change the temperature of the sample (T) with time (t).
2. Machines and materials used
3. Precautions need to taken during the experiment