PHASE TRANSFORMATIONS & HEAT TREATMENT

LABORATORY OBSERVATION BOOK

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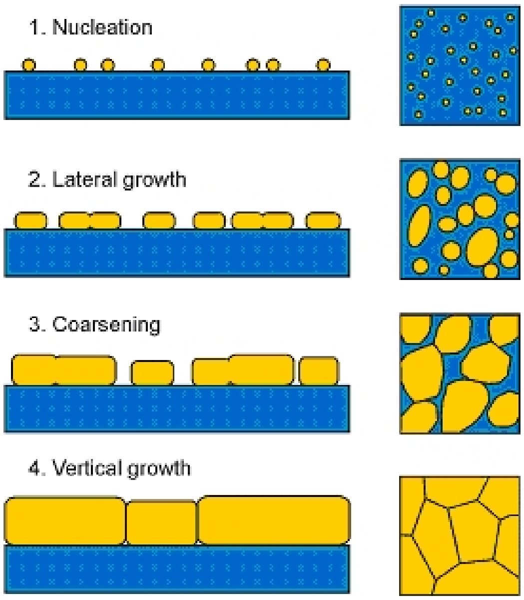

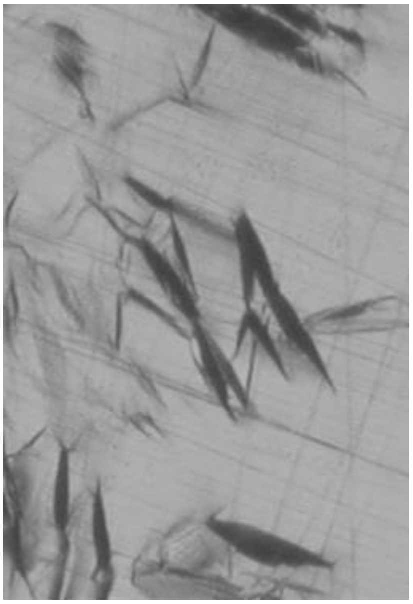

Department of Metallurgy & Materials Engineering

VEER SURENDRA SAI UNIVERSITY OF TECHNOLOGY, BURLA

Odisha – 768018

PHASE TRANSFORMATIONS & HEAT TREATMENT

LABORATORY OBSERVATION BOOK

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

Roll No: ....

LABOROTARY COORDINATOR(S)

A. LAVA KUMAR
Assistant Professor

Department of Metallurgy & Materials Engineering

VEER SURENDRA SAI UNIVERSITY OF TECHNOLOGY, BURLA

Odisha - 768018

FOREWORD

This lab manual is designed to suit the needs of the under graduate metallurgist in acquainting with the heat treatment principles and transformations in materials. The metallographic techniques are stressed, as these practices are essential for a metallurgical engineer. In this observation book an attempt is made to clarify the concepts of Annealing, Normalizing, Hardening, Tempering and Hardenability. The students shall get clarity about microstructures of carbon steels, cast irons and non-ferrous alloys, in particular to differentiate between various phases present in metals and alloys. The student shall also be trained in grain size measurements, and quantitative metallography.

Instructor will indicate the experiments to be conducted and advise on the nature of the lab report and time of submission. However, a typical format for a lab report is included in this observation book. It is important that all the information necessary to complete the lab report is obtained before students leave the lab.

Laboratory Notebook Grading: 20% Neatness and Legible, 20% Experimental Description, 20% Observations and Comments, 20% Data Collection, 20% Data Analysis including plots, calculations, and conclusions drew.

Mr. A.Lava Kumar
Assistant Professor

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SAFETY IN THE LABORATORY

All students must read and understand the information in this document with regard to laboratory safety and emergency procedures prior to the first laboratory session. ***Your personal laboratory safety depends mostly on you***. Effort has been made to address situations that may pose a hazard in the lab but the information and instructions provided cannot be considered all-inclusive. Students must adhere to written and verbal safety instructions throughout the academic term.

Common Sense

Good common sense is needed for safety in a laboratory. It is expected that each student will work in a responsible manner and exercise good judgment and common sense. If at any time you are not sure how to handle a particular situation, ask your Teaching Assistant or Instructor for advice. **DO NOT TOUCH ANYTHING WITH WHICH YOU ARE NOT COMPLETELY FAMILIAR**!!! It is always better to ask questions than to risk harm to yourself or damage to the equipment.

Personal and General laboratory safety

1. Never eat, drink, or smoke while working in the laboratory. Read labels carefully.
2. Do not use any equipment unless you are trained and approved as a user by your supervisor.
3. Wear safety glasses or face shields when working with hazardous materials and/or Equipment. Wear gloves when using any hazardous or toxic agent.
4. Clothing: When handling dangerous substances, wear gloves, laboratory coats, and safety shield or glasses. Shorts and sandals should not be worn in the lab at any time. Shoes are required when working with the heat treatment furnaces.
5. If you have long hair or loose clothes, make sure it is tied back or confined.
6. Keep the work area clear of all materials except those needed for your work. Extra books, purses, etc. should be kept away from equipment, which requires air flow or ventilation to prevent overheating.
7. Disposal - Students are responsible for the proper disposal of used material if any in appropriate containers.
8. Equipment Failure - If a piece of equipment fails while being used, report it immediately to your lab assistant or tutor. Never try to fix the problem yourself because you could harm yourself and others.
9. If leaving a lab unattended, turn off all ignition sources and lock the doors. Clean up your work area before leaving. Wash hands before leaving the lab and before eating.

Cutting & Grinding

1. Power saws should not be operated without eye protection and an apron.
2. Do not push the specimen or materials against the cutting wheel directly with the fingers.
3. Long materials should be supported or clamped before cutting.
4. Hold the sample tightly.
5. Do not overheat the sample while grinding. Cool the sample in water during intermediate inspections.

Heat Treating

1. Use tongs to insert or remove the specimens from the furnace.
2. Use insulating gloves to open or close the doors to the furnaces.
3. Either cool the specimens immediately after removal from the furnace or place in a designated area for slow cooling. Hot specimens should not be left in the open where may be accidentally touched.
4. The furnaces should be turned off when not in use.
5. If specimens are left in a furnace and the area is abandoned by the student, a sign must be left with a name and phone number and time for removal.
6. Quenching samples in oil can cause the oil to ignite. Be prepared to cover the container after immersion.
7. Use baskets or tongs for quenching in oil or water.

Chemical safety

1. Treat every chemical as if it were hazardous.
2. Make sure all chemicals are clearly and currently labeled with the substance name, concentration, date, and name of the individual responsible.
3. Never return chemicals to reagent bottles. (Try for the correct amount and share any excess.)
4. Comply with fire regulations concerning storage quantities, types of approved containers and cabinets, proper labeling, etc. If uncertain about regulations, contact the building coordinator.
5. Use volatile and flammable compounds only in a fume hood. Procedures that produce aerosols should be performed in a hood to prevent inhalation of hazardous material.
6. Never allow a solvent to come in contact with your skin. Always use gloves. Never "smell" a solvent!! Read the label on the solvent bottle to identify its contents.
7. Dispose of waste and broken glassware in proper containers. Clean up spills immediately.
8. Do not store food in laboratories.

I ____ Scholar No ______________________ have read and understood the laboratory safety procedures and policies. I am responsible for following these procedures while in the laboratories.

____

(Signature with date)

PHASE TRANSFORMATIONS & HEAT TREATMENT LABORATORY

EVEN SEMESTER 2016-17

LOGBOOK MARK SHEET

Name: **Section:**

Scholar Number: **Group :**

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S.No	**Experiment Title**	**Marks obtained**	**Instructor Sign.**
1.	Concepts in PT& HT (Revisit)	......./10	
2.	Annealing & Normalizing	......./10	
3.	Spherodising & Hardening	......./10	
4.	Nucleation of Ice from Water : A Modelling Approach	......./10	
5.	Study of nucleation and growth in Eutectoid steel	......./10	
6.	Jominy End Quench Test	......./10	
7.	Carburization of Steel	......./10	
8.	Precipitation Hardening	......./10	
9.	Differential Scanning Calorimetry	......./10	
10.	CASE STUDY	......./10	
	Total Marks obtained		

Unauthorized absences will be credited with a mark of 0/10. Your completed laboratory logbook (with this sheet firmly stuck in and fully completed) must be submitted to your instructor at the end of each experiment. Your instructor will make an assessment and transmit the mark.

A. LAVA KUMAR
Laboratory Coordinator

EXPERIMENT 1

CONCEPTS IN PHASE TRANSFORMATIONS & HEAT TREATMENT

Draw Fe-Fe_3C Phase Diagram and identify the phases

Draw Fe-C Phase Diagram and identify the phases

Draw TTT Diagram for Hypo-Eutectoid, Eutectoid, Hyper-eutectoid Steels

Draw CCT Diagram for Hypo-Eutectoid, Eutectoid, Hyper-eutectoid Steels

Draw a schematic view of a laboratory furnace

EXPERIMENT 2

ANNEALING & NORMALIZING

OBJECTIVE

To study the microstructure, grain size and hardness of Annealed and Normalized plain carbon steel having ≤0.2%C and ≤0.4%C.

EQUIPMENT / MATERIALS

Steel samples of 0.2%C, 0.4%C and 1.2%C, Electric furnace, SiC papers of various grit sizes, Polishing machine, Metallurgical microscope, Rockwell hardness tester.

THEORY

The heat treatment includes heating and cooling operations or the sequence of two or more such operations applied to any material in order to modify its metallurgical structure and alter its physical, mechanical and chemical properties. Usually it consists of heating the material to some specific temperature, holding at this temperature for a definite period and cooling to room temperature or below with a definite rate. *Annealing*, *Normalizing*, *Hardening* and *Tempering* are the four widely used heat treatment processes that affect the structure and properties, and are assigned to meet the specific requirements from the semi-fabricated and finished components. Steels being the most widely used materials in major engineering fabrications undergo various heat treatment cycles depending on the requirements.

Annealing refers to a wide group of heat treatment processes and is performed primarily for homogenization, recrystallization or relief of residual stress in typical cold worked or welded components. Depending upon the temperature conditions under which it is performed, annealing eliminates chemical or physical non-homogeneity produced of phase transformations. Few important variants of annealing are full annealing, isothermal annealing, spheroidise annealing, recrystallization annealing, and stress relief annealing. In this experiment we will do full annealing treatment of carbon steels.

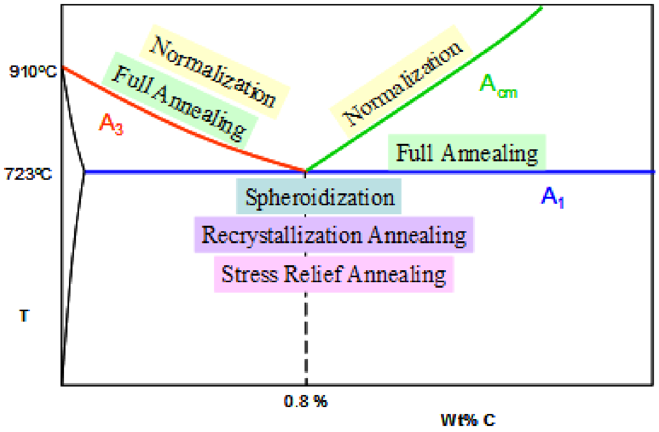
Full annealing

Full annealing process consists of three steps. First step is heating the steel component to above A_3 (upper critical temperature for ferrite) temperature for hypo-eutectoid steels and above A_1(lower critical temperature) temperature for hypereutectoid steels by 30-50^{o}C. The second step is holding the steel component at this temperature for a definite holding (soaking) period of at least 20 minutes per cm of the thick section to assure equalization of temperature throughout the cross-section of the component and complete austenization. Final step is to cool the hot steel component to room temperature slowly in the furnace, which is also called as *furnace cooling*. The *full* *annealing* is used to relieve the internal stresses induced due to cold working, welding, etc, to reduce hardness and increase ductility, to refine the grain structure, to make the material homogenous in respect of chemical composition, to increase uniformity of phase distribution, and to increase machinability.

Normalizing

Normalizing process consists of three steps. The *first step* involves heating the steel component above the A_3 temperature for hypo eutectoid steels and above A_{cm} (upper critical temperature for cementite) temperature for hypereutectoid steels by 30^0C to 50^0C (*Figure 4.7.5*). The *second step* involves holding the steel component long enough at this temperature for homogeneous austenization. The final step involves cooling the hot steel component to room temperature in still air. Due to air cooling, normalized components show slightly different structure and properties than annealed components.

The properties of normalized components are not much different from those of annealed components. However, normalizing takes less time and is more convenient and economical than annealing and hence is a more common heat treatment in industries. Normalizing is used for high-carbon (hypereutectoid) steels to eliminate the cementite network that may develop upon slow cooling in the temperature range from point A_{cm} to point $A_{1.}$ Normalizing is also used to relieve internal stresses induced by heat treating, welding, casting, forging, forming, or machining. Normalizing also improves the ductility without reducing the hardness and strength.



Schematic representation of annealing operation

PROCEDURE

OBSERVATIONS

Observe the microstructure and write down the phases present

Sample Identity:

Draw the microstructure in the circle given below and write down the phases

Magnification used _____________

The phases present in the microstructure and the approximate % of major phases are

Phases Present	Percentage
➢ --	------------
➢ --	-------------
➢ --	-------------

Etchant used _________________ Hardness _________________

From the observation of microstructure & Hardness of the given sample

……………………………………………………………………………………………..

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Observe the microstructure and write down the phases present

Sample Identity:

Draw the microstructure in the circle given below and write down the phases

Magnification used _____________

The phases present in the microstructure and the approximate % of major phases are

	Phases Present	Percentage
➢	--	------------
➢	--	-------------
➢	--	-------------

Etchant used _________________

Hardness _________________

From the observation of microstructure & Hardness of the given sample

……………………………………………………………………………………………..

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Answer the following Questions

1. Define the term “heat treatment”. Why are the steels heat treated?

2. If one of the specimens of the same kind of steel is normalized and the other is annealed, which will show more strength and why?

3. Out of the normalized and annealed samples, which one is more machinable and why?

4. How does the hardness vary with cooling rate?

5. How do your microstructures compare with standard microstructures?

6. What temperature will be used for annealing and normalizing the mild steel specimen with 0.25%C?

7. What is the nature of the microstructure in spheroidized high carbon steel, normalized high carbon steel, annealed high carbon steel?

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

9. Explain the process of annealing?

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

EXPERIMENT 3

SPHEROIDISING & HARDENING

OBJECTIVE

To study the microstructure and hardness of Hardened and Spheroidized plain carbon steel having $\leq$1.2%C steel.

EQUIPMENT / MATERIALS

Steel samples of 0.2%C, 0.4%C and 1.2%C, Electric furnace, SiC papers of various grit sizes, Polishing machine, Metallurgical microscope, Rockwell hardness tester.

THEORY

One of the techniques to improve the ductility of the high carbon steels are spheroidised annealing.

Spheroidized annealing

Spheroidise annealing is one of the variant of the annealing process that produces typical microstructure consisting of the globules (spheroid) of cementite or carbides in the matrix of ferrite. The following methods are used for spheroidise annealing.

Holding at just below A_1

Holding the steel component at just below the lower critical temperature (A_1) transforms the pearlite to globular cementite particles. But this process is very slow and requires more time for obtaining spheroidised structure.

Thermal cycling around A_1

In this method, the thermal cycling in the narrow temperature range around A_1 transforms cementite lamellae from pearlite to spheroidal. *Figure* depicts a typical heat treatment cycle to produce spheroidised structure. During heating above A_1, cementite or carbides try to dissolve and during cooling they try to re-form. This repeated action spheroidises the carbide particles. Spheroidised structures are softer than the fully annealed structures and have excellent machinability. This heat treatment is utilized to high carbon and air hardened alloy steels to soften them and to increase machinability, and to reduce the decarburization while hardening of thin sections such as safety razor blades and needles.

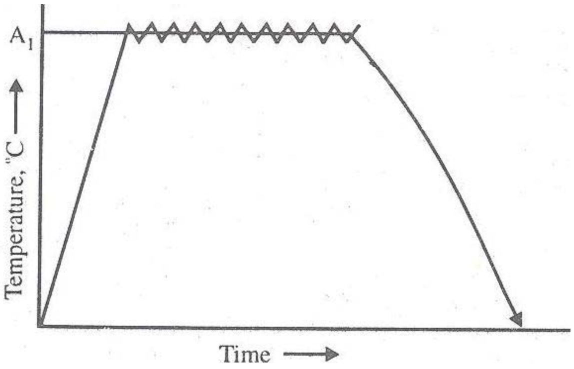

Figure 1: A typical heat treatment cycle to produce spheroidised structure

Different techniques to improve the hardness of the steels are conventional hardening, and tempering.

Conventional hardening

Conventional hardening process consists of four steps. The *first step* involves heating the steel to above A_3 temperature for hypo-eutectoid steels and above A_1 temperature for hypereutectoid steels by 50^0C. The *second step* involves holding the steel components for sufficient socking time for homogeneous austenization. The *third step* involves cooling of hot steel components at a rate just exceeding the critical cooling rate of the steel to room temperature or below room temperature. The *final step* involves the tempering of the martensite to achieve the desired hardness. Detailed explanation about tempering is given in the subsequent sections. In this conventional hardening process, the austenite transforms to martensite. This martensite structure improves the hardness.

Following are a few salient features in conventional hardening of steel.

1. Proper quenching medium should be used such that the component gets cooled at a rate just exceeding the critical cooling rate of that steel.
2. Alloy steels have less critical cooling rate and hence some of the alloy steels can be hardened by simple air cooling.
3. High carbon steels have slightly more critical cooling rate and has to be hardened by oil quenching.
4. Medium carbon steels have still higher critical cooling rates and hence water or brine quenching is necessary.

Figure 2 depicts the conventional hardening process which involves quenching and tempering. During quenching outer surface is cooled quicker than the center. Thinner parts are cooled faster than the parts with greater cross-sectional areas. In other words, the transformation of the austenite is proceeding at different rates. Hence there is a limit to the overall size of the part in this hardening process.

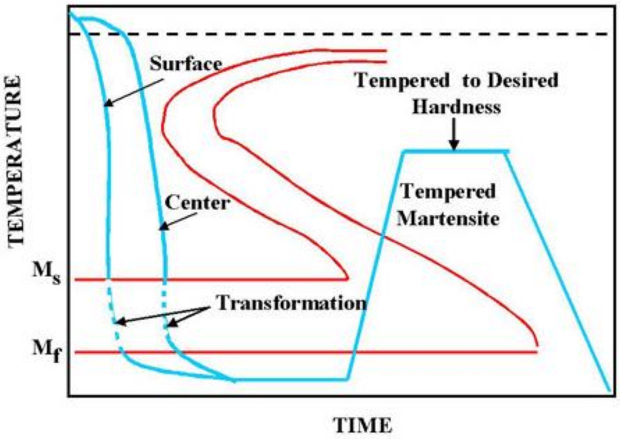

Figure 2: Heat treatment cycle for conventional hardening process

PROCEDURE

OBSERVATIONS

Observe the microstructure and write down the phases present

Sample Identity:

Draw the microstructure in the circle given below and write down the phases

Magnification used _____________

The phases present in the microstructure and the approximate % of major phases are

Phases Present	Percentage
➢ ---	------------
➢ ---	-------------
➢ ---	-------------

Etchant used _________________

Hardness _________________

From the observation of microstructure & Hardness of the given sample

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Observe the microstructure and write down the phases present

Sample Identity:

Draw the microstructure in the circle given below and write down the phases

Magnification used ____________

The phases present in the microstructure and the approximate % of major phases are

	Phases Present	Percentage
➢	--	------------
➢	--	-------------
➢	--	-------------

Etchant used ________________ Hardness ________________

From the observation of microstructure & Hardness of the given sample

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Answer the Following Questions

1. How do your tempered structures compare with standard microstructures of tempered martensite and spheroidised steel?

2. Give the physical and mechanical changes that occur during the above phase transformation

3. Explain the nature of pearlite and cementite phase in spheroidised steel?

4. Why is tempering preceded by converting the steel into martensite phase?

5. Which steel will be tougher, tempered high carbon steel or hardened medium carbon steel?

6. What is the nature of the microstructure in Spheroidized high carbon steel, normalized high carbon steel, annealed high carbon steel?

7. How does hardness vary with % carbon content in steels? Explain.

8. Why it is necessary to be really quick in transferring the specimen from the austeniting furnace to the water bath during quenching?

9. How does the hardness of specimen tempered at 700°C compare with that of the furnace cooled specimen? A Steel given such a tempering treatment is preferred to a furnace cooled specimen for good machinability. Can you explain this on the basis of the difference in microstructure?

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

EXPERIMENT 4

NUCLEATION OF ICE FROM WATER: A MODELLING APRROACH

OBJECTIVE

To study the transformation between Water to Ice nucleation.

To understand the relation between ΔG vs r, ΔG* vs ΔT, r* vs ΔT in Water to Ice transformation.

SOFTWARE/DATA

$K = 1.38 \times 10^{23} J/K, T_m = 273K, \Delta \mathrm{H_m} = 6.02 KJ/mol, \gamma_{\mathrm{SL}} = 0.033 J/m^2, \mathrm{V_m} = 1.90 \times 10^{-5} m^3$

MS-Excel software.

FORMULAE

$$\Delta G = -\frac{4}{3}\pi r^3 \left|\Delta G_V\right| + 4\pi r^2 \gamma_{SL}$$

$$r^* = \frac{2\gamma_{SL}}{\left|\Delta G_V\right|} = \frac{2\gamma_{SL} V_M T_M}{\left|\Delta H_m\right| \Delta T} \text{ and } \Delta G^* = \frac{16\pi}{3} \frac{\gamma_{SL}{}^3 V_M{}^2 T_M{}^2}{\Delta H_m{}^2 \Delta T^2}$$

$$\Delta G_V = \frac{\Delta H_m \Delta T}{T_m V_m} = \frac{\Delta H_m (T_m - T)}{T_m V_m}$$

THEORY

Phase changes occur in two ways, such as homogeneous transformation where every region of the parent phase undergoes change such that its free energy decreases and parent phase gradually approaches the product phase. Another mode of transformation, which observed in most of the industrially important systems, consists of the appearance of product phase at preferred discrete sites in the parent phase and growth of these regions by migrations of their interfaces. Such transformation is known as heterogeneous transformations. The initiation of the transformation at discrete sites is called nucleation and the propagation of the interface of the nucleated regions in the parent phase is called growth of the nucleated region. The rate of a transformation is determined by the rate of nucleation ($\dot{N}$) and the rate of growth ($\dot{G}$). The rate of nucleation $\dot{N}$ is usually expressed as the number of nuclei formed per unit time per unit volume. The rate of growth G is expressed in distance/time.

When the probability of the appearance of a nucleus in any small volume of the parent phase is same as that in any other small volume of the parent phase is same as that in any other small volume of the parent phase, nucleation occurs randomly all over the parent phase at the same rate (homogeneous nucleation). However, if high energy sites are present in the parent phase nucleation may occur preferably on this high energy sites (heterogeneous nucleation).

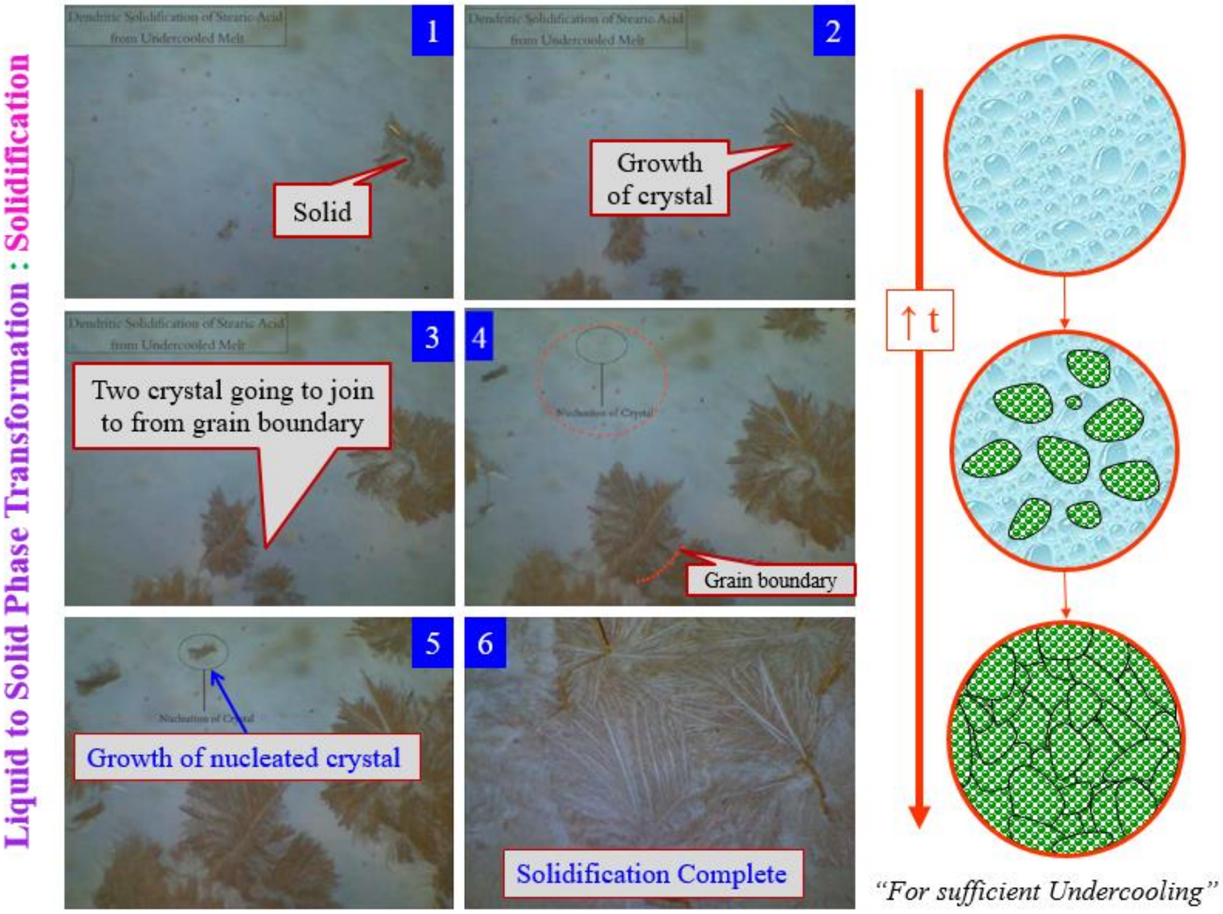
Homogeneous Nucleation

Let us consider L→S transformation taking place by homogenous nucleation. Let the system be undercooled to a fixed temperature ΔT. Let us consider the formation of a spherical crystal of radius ‘r’ from the melt. We can neglect the strain energy contribution.

Let the change in ‘G’ during the process be ΔG. This is equal to the decrease in bulk free energy + the increase in surface free energy. This can be computed for a spherical nucleus as below.

$$\Delta G = (\text{Volume}).(\Delta G) + (\text{Surface}).(\gamma)$$

$$\Delta G = \left(\frac{4}{3}\pi r^3\right).(\Delta G_v) + \left(4\pi r^2\right).(\gamma)$$



As we have noted previously ΔG vs r plot will go through a maximum (implying that as a small crystal forms ‘G’ will increase and hence it will tend to dissolve). The maximum of ΔG vs r plot is obtained by, by setting dΔG/dr = 0. The maximum value of ΔG corresponds to a value of ‘r’ called the critical radius (denoted by superscript *). If by some ‘accident’ (technically a ‘statistical random fluctuation’) a crystal (of ‘preferred’ crystal structure) size > r* (called supercritical nuclei) forms, then it can grow down-hill in ‘G’. Crystals smaller than r* (called embryos) will tend to shrink to reduce ‘G’. The critical value of ΔG at r* is called ΔG*.

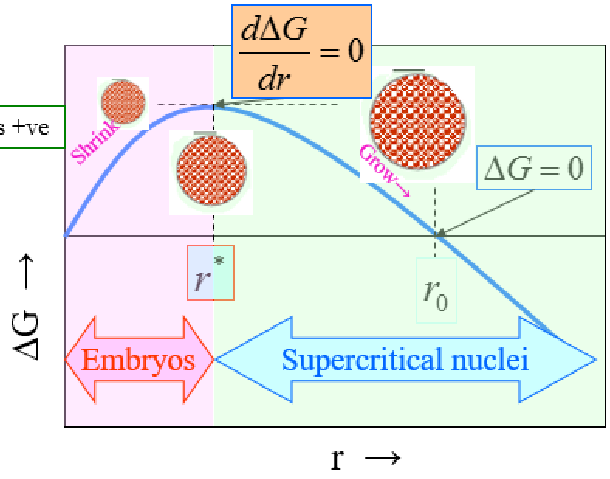
Reduction in G (below the liquid state) is obtained only after r_0 is obtained (which can be obtained by setting ΔG = 0).

$$\frac{d\Delta G}{dr} = 0 \Rightarrow \begin{cases} r_1^* = 0 & \text{Trivial} \\ r_2^* = -\dfrac{2\gamma}{\Delta G_v} & \end{cases}$$

As ΔG_v is −ve, r^* is +ve

$$r^* = -\frac{2\gamma}{\Delta G_v} \qquad \Delta G^* = \frac{16}{3}\pi\frac{\gamma^3}{\Delta G_v^2}$$

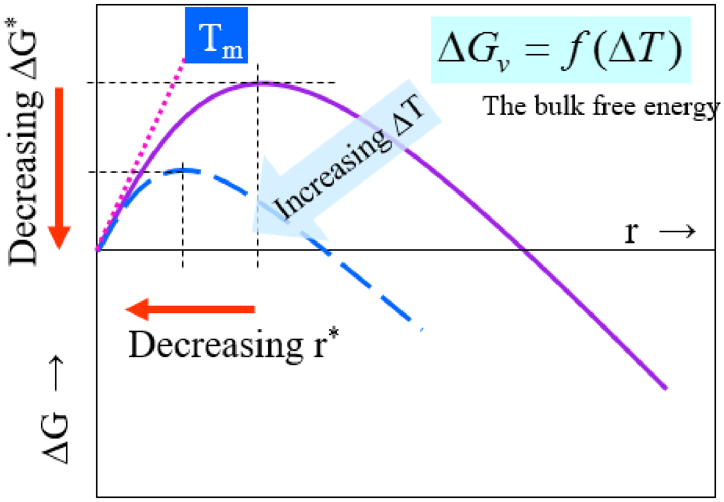
$$\Delta G = 0 \Rightarrow r_0 = -\frac{3\gamma}{\Delta G_v}$$



What is the effect of undercooling (ΔT) on r* and ΔG*?

We have noted that ΔG_V is a function of undercooling (ΔT). At larger undercooling ΔG_V increases and hence r* and ΔG* decrease. This is evident from the equations for r* and ΔG* as below

At T_m, ΔG_V is zero and r* is infinity! → That the melting point is not the same as the freezing point!! This energy (G) barrier to nucleation is called the *'nucleation barrier'*.



$$\Delta G_v = f(\Delta T)$$

The bulk free energy reduction is a function of undercooling

$$r^* = -\frac{2\gamma}{\Delta G_v} \qquad \Delta G^* = \frac{16}{3}\pi\frac{\gamma^3}{\Delta G_v^2}$$

Using the Turnbull approximation (linearizing the G-T curve close to T_m), we can get the value of ΔG interms of the enthalpy of solidification.

$$\Delta G^* = \frac{16}{3}\pi\gamma^3\frac{T_m^2}{\Delta T^2\,\Delta H^2}$$

OBSERVATIONS

Answer the Following Questions

1. Define Homogeneous nucleation & Heterogeneous nucleation.

2. Draw the cooling curves for Homogeneous and Heterogeneous nucleation.

3. Is this G* is same for both nucleation process? Give a reason.

4. What is the effect if interfacial energy on nucleation process?

5. Explain the variation of ΔT in homogeneous and heterogeneous nucleation.

6. Is this r* is same for both nucleation process? Give a reason.

7. When will the grain growth stop in a single phase polycrystalline material? Will the grain size continue to increase until the material is a single crystal?

8. What is the effect of grain growth on hardness and ductility?

9. Most of the materials that we encounter in our daily lives are polycrystalline. If the grain boundary energy does increase the free energy of the system, why do they not disappear leaving behind a single crystal?

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

EXPERIMENT 5

STUDY OF NUCLEATION AND GROWTH IN EUTECTOID STEEL

OBJECTIVE

To study the nucleation rate and growth rate of pearlite in eutectoid steel by austenitising, followed by isothermal holding for different time intervals below eutectoid temperature of the given samples, followed by final quenching.

EQUIPMENT / MATERIALS

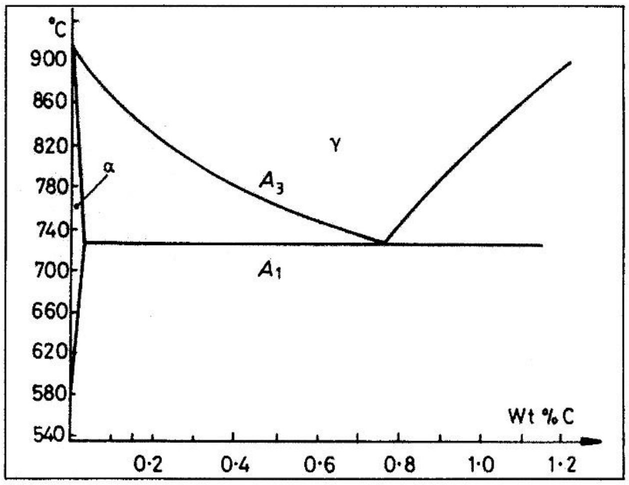
Samples of 0.8% carbon steel, Furnace with temperature controller, for temperature up to 1000°C. Rockwell hardness tester, Microscope, Metallographic polishing equipment, Belt grinder, Etching agent

THEORY

Part of the Fe-C phase diagram is shown in below figure. When the austenite phase is cooled below 727˚C (A1 temperature), it transforms into ferrite and cementite. The reaction may be written as:

$$\gamma \rightarrow \alpha + Fe_3C$$

This reaction is an example of a eutectoid transformation. In this lab we are interested in studying the nucleation and growth of the new-phases.



When the probability of the appearance of a nucleus in any small volume of the parent phase is same as that in any other small volume of the parent phase is same as that in any other small volume of the parent phase, nucleation occurs randomly all over the parent phase at the same rate (homogeneous nucleation). However, if high energy sites are present in the parent phase nucleation may occur preferably on this high energy sites (heterogeneous nucleation). Grain boundaries, inclusion, free surface, dislocations etc. are some of the heterogeneous sites found in metals. The nucleation growth rates can be obtained if a sample of the coarse grained steel is cooled after austenization to a temperature below the eutectoid temperature, and held for transformation to occur (Isothermal transformation - TTT diagram).

0.8% C (wt %) is called eutectoid steel, where eutectoid reaction (gamma to alpha and cementite) occurs. Once a steel is just below eutectoid temperature (727˚C), eutectoid reaction leads to formation of pearlite by forming the first phase as ferrite or cementite. After the first phase formation, alternate lamellae form leading to the typical morphology of pearlite. The formation kinetics are quite complicated. However, the nucleation of pearlite occurs at the grain boundaries, and with time (isothermal) as well with temperature (non-isothermal), growth of the nucleated pearlite occurs. The schematic of the resulting microstructure is shown in the Figure (5.1). The alternating layers of ferrite and cementite are collectively referred to as pearlite (Figure 5.1.a). The pearlite appears to grow into the adjoining grains as nodules as shown in Figure 5.1.b. Each nodule contains one or more colonies: a colony refers to adjacent ferrite and cementite sheets having the same orientation and growing in the same direction.

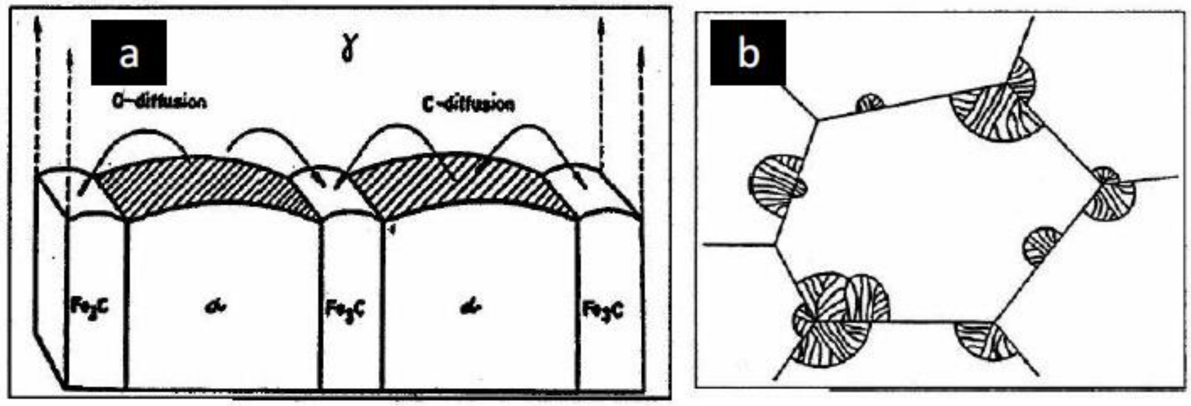
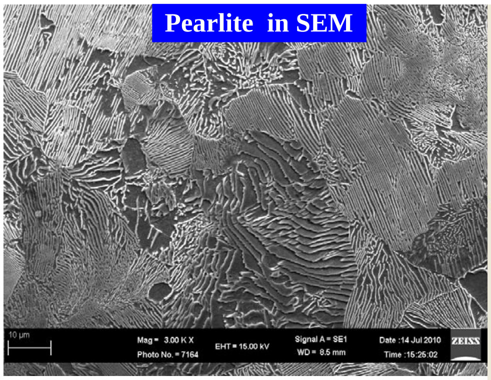
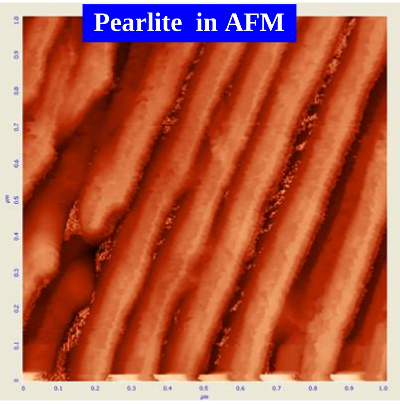
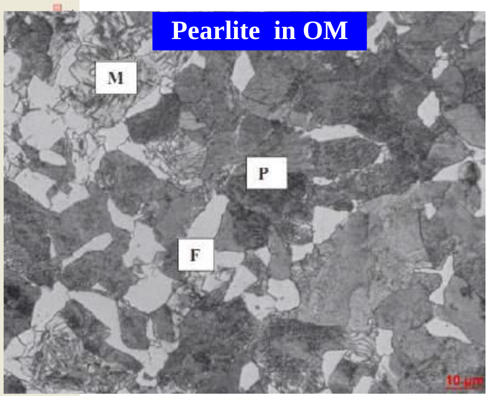


Fig.5.1 Pearlite growth mechanism in eutectoid steels


Pearlite in SEM


Pearlite in AFM


Pearlite in OM

In this lab we will study the kinetics of the (→ pearlite transformation). Therefore, the numbers of circular pearlite patches grow out of a single nucleus. Therefore, the numbers of circular pearlite patches give the number of nuclei. From these data, rate of nucleation can be calculated. Similarly, the radius or diameter of the circular patch (pearlite colony) is due to the growth of this patch between the time at which the corresponding nuclei appeared and the time of final quench. Therefore, the largest circular patch must represent the first nuclei and its radius or diameter can be used to calculate the growth rate.

Even austenite nucleation can also be done in the similar way where fully annealed low carbon steel can be heated to the austenitising temperature and holding it at that temperature with time and quenching. The quenched microstructure, the martensite zones have formed from the nucleated austenite from the interfaces of the ferrite-pearlite microstructure.

PROCEDURE

OBSERVATIONS

Observe the microstructure and write down the phases present

Sample Identity:

Draw the microstructure in the circle given below and write down the phases

Interlamellar spacing/Time (Hrs.)

Magnification used ____________

The phases present in the microstructure and the approximate % of major phases are

	Phases Present	Percentage
➢	---	------------
➢	---	-------------
➢	---	-------------

Etchant used ________________

From the observation of microstructure & Hardness of the given sample

……………………………………………………………………………………………..

……………………………………………………………………………………………..

…………………………………………………………………………………….....

....

Draw a plot between Interlamellar spacing vs. Time (t)

Draw a plot between Volume fraction of pearlite vs. Time (t)

Answer the Following Questions

1. Discuss the variation in colonies size as a function of soaking time.

2. Distinguish between eutectic and eutectoid steels.

3. Explain the differences between the TTT and CCT curves. Suppose you are concerned with the formation of martensite following welding. Which curve will you use to analyses the process?

4. How do you explain the "C" appearance of the TTT-diagram? Give an example of a phase transformation where C-Curve kinetics are not expected. Explain your example.

5. The nucleation of pearlite is known to occur on grain-boundaries. What effect would a coarser grain size result in? Do you expect the austenitising temperature to affect the C-curve kinetics? How?

6. Why is it necessary to use thin samples for this experiment?

7. The addition of solutes (e.g. Mn, Cr, Si.... etc) will affect the shape and position of the C-curve. Describe any two mechanisms by which the solutes are able to bring about such changes.

8. If a piece of steel having 0.8 % carbon has martensitic structure can it be converted to fully pearlite structure by holding it at 700° C?

9. Estimate the ratio of the widths of ferrite and cementite plates in lamellar pearlite.

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

EXPERIMENT 6

HARDENABILITY: JOMINY END QUENCH TEST

OBJECTIVE

To study hardness and microstructure as a function as a function of quench rate, and investigate the hardenability of steels by Jominy test.

EQUIPMENT / MATERIALS

Sample of medium or high carbon steel (the steel must have more than 0.35%C), or alloy steel, Quenching tank, Electric furnace, Special tongs, Rockwell hardness tester, metallographic polishing equipment, Metallurgical microscope.

THEORY

In a practical sense it is not possible to heat-treat all parts to the same degree. The difference is due to the thickness or volume effect. Basically, when a part is quenched in water or some other fluid, the heat must be conducted out through the surface. This leads to a temperature gradient dt/dx between the surface and the center of the part being heat-treated. The temperature gradient varies with time.

The temperature gradient is less steep between the center and the edge at later times. Therefore, the temperature of the center lags in time behind the temperature of the surface. If we were to plot a time profile of the center and the edge temperatures as shown in Figure 1, the time to reach a given temperature T_2 is definitely longer in the center than at the edge. This means that cooling rate varies as a function of depth. The greater the depth the slower the cooling rate.

The situation with respect to the cooling rate can lead to a different hardness in the center than at the edge. The edge could transform to martensite and the center to pearlite or bainite.

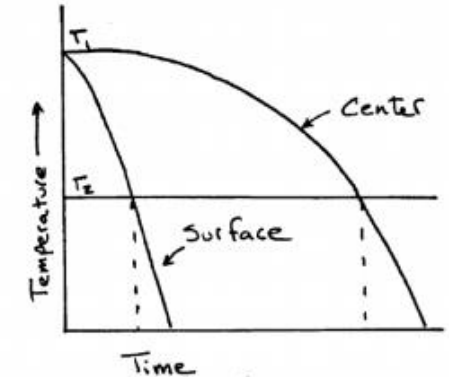


Figure 1: Cooling curves for the surface and center of a quenched specimen

In selecting a steel, the ability to cool the center depends upon the thickness of the part. The thicker the part, the slower the cooling rate at the center. For a given thickness, one must select a steel that can be hardened in the center if that is desired. The cooling rate in this case is fixed. The center part of steel can be hardened by shifting the time-temperature transformation diagram through alloying. Figure 2 shows that alloying elements added to plain carbon steel can shift the nose of the TTT curve to longer times and raise the M_s temperature. This means a slower cooling rate can be used to reach the martensitic state. A slower cooling rate means a thicker part can be heat-treated.

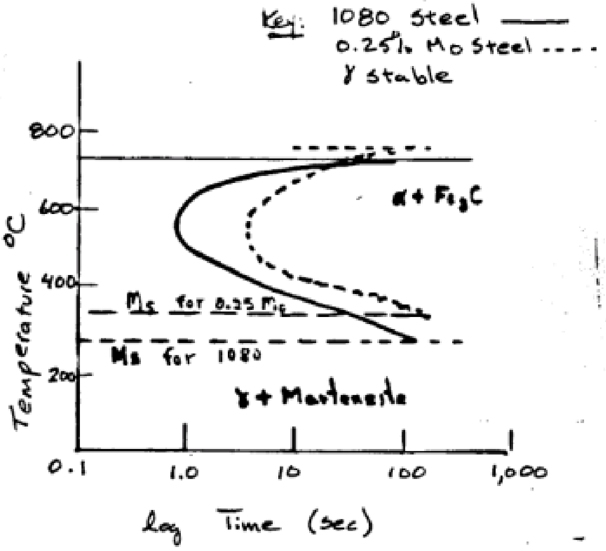
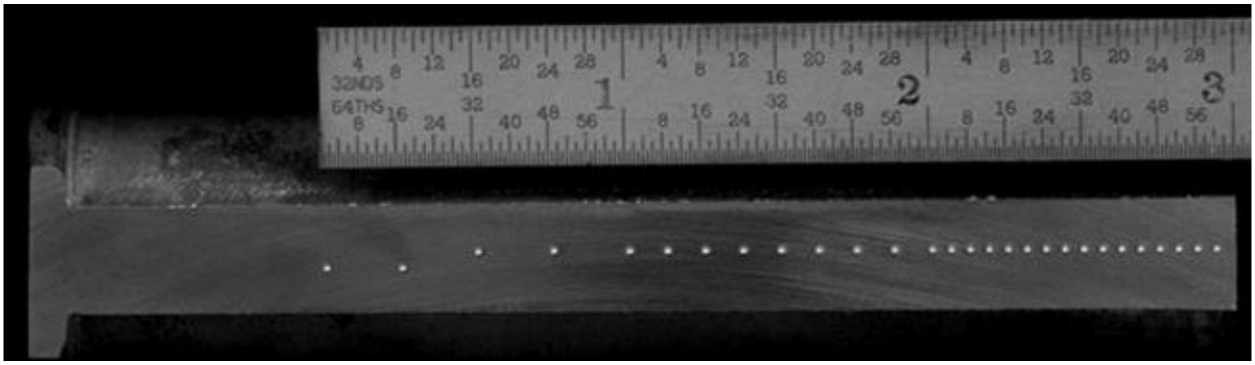


Figure 2: TTT diagram for two different steels

To obtain standardized data on the hardness of steels as functions of cooling rates, the Jominy End Quench test was developed. In the test, water is sprayed on one end of a bar of steel while it is hot. This leads to a one dimensional heat transfer cooling. Except near the surface of the bar the temperature is controlled by heat flow along the length of the bar (like thickness in the part). Moving axially away from the quenched end of the bar, the temperature and the rate of change of temperature are changing. The temperature is higher and the cooling rate is lower. If surface hardness is measured as a function of distance from the end, a hardness profile can be obtained which applies to any part made from the same steel, as shown in Figure 3.



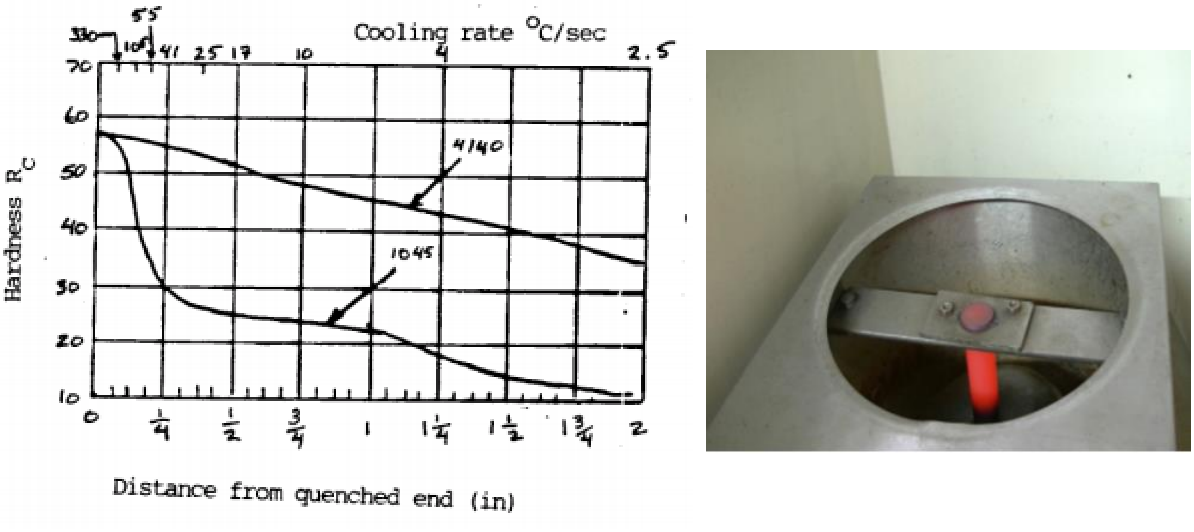


Figure 3: Quenched Tank and Hardness as a function of distance from quenched end for two steels

PROCEDURE

OBSERVATIONS

1. **Measure hardness as a function of distance from the rapidly quenched end. Make a hardness reading every $\frac{1}{8}$ in. in the most rapidly quenched one inch, every $\frac{1}{4}$ in**

Distance (inches)	**Hardness Measurements**			**Hardness ( )**
	Trail 1	**Trail 2**	**Trail 3**	

2. **Draw the plot between distance vs. hardness of given sample**

Answer the Following Questions

1. How do your hardness measurements correlate with your microstructural observations?

2. Give five applications for quench-hardened steels.

3. What will be the difference in hardness profile of a steel part quenched in water and in oil at the same temperature?

4. Steel gears are being quenched by dropping into a tank of room temperature oil. A competitor makes gears of the same size and shape, apparently from the same raw material, but his gears last longer than yours. What can you do to improve the quality of your product and cut down wear? What is the danger in this course of action if you overdo it?

5. What is the ideal critical diameter and can it be determined with a Jominy test?

6. How is the role of carbon and various alloy elements on the hardenability of steels?

7. What effect does surface scale have on hardness measurements?

8. You have three steels. Select the most appropriate steel to achieve the necessary levels of mechanical properties, residual stress and distortion in a 1mm diameter wood-working drill. Why? 1% C, 0.4% Si, 1% Mn, 5% Cr, 1% Mo and 0.4% C, 0.4% Mn, 0.3% Si, and 0.5% C, 4% Cr, 6% Mo

9. As grain size increases, effect boron on hardenability decreases. Why?

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

EXPERIMENT 7

CARBURIZATION OF STEEL

OBJECTIVE

Observe the carburized and decarburized layer (selective etching will be done due to gradual carbon increment from surface to center.

To carburize the carbon steel and measure the diffusion coefficient

EQUIPMENT / MATERIALS

Samples of 0.2% Carbon steel alloy, 30-32% coke, 53-55% charcoal and 2-3% Na2Co3, Ba2Co3, and 3-4% CaCo3, Stainless steel box, Electric furnace, SiC papers of various grit sizes, Polishing machine, Metallurgical microscope, Rockwell hardness tester.

THEORY

Carburizing

If a steel is exposed to carbonaceous atmosphere (CO gas) at an elevated temperature, carbon dissolves in the surface of the specimen building up the carbon concentrations at the surface and diffuses inside under the influence of carbon concentration gradient (between the surface and inside). Such a process is known as carburization. Here the following reaction takes place at the surface.

$$2CO(g) = CO_2(g) + C\,(\text{steel})$$

If the carbon content of the carburizing atmosphere remains constant, it would give rise to a constant carbon concentration C_s (Solubility limit of carbon in steel at the carburizing temperature) at the surface of the steel. If we assume that the diffusion is unidirectional, the process of carbon diffusion can be described by Fick's second law,

$$\frac{dc}{dt} = \frac{d}{dx}\left[D\frac{dc}{dx}\right]$$

Where D is the coefficient of diffusion of the diffusing element, dc/dt and dc/dx are concentration gradients with time and distance, respectively. If $D \neq D(c)$

$$\frac{dc}{dt} = D\frac{d^2c}{dx^2}$$

The solution to this equation is of the general form in case of the diffusion couple consisting of a constant concentration on the surface (Cs i.e. for carburizing process) is given by

$$\frac{C_s - C(x,t)}{C_s - C_o} = erf\ \frac{x}{2\sqrt{Dt}}$$

Where Cs is surface carbon concentration, C(x,t) is carbon concentration at depth "x" after time "t", C_0 is initial carbon concentration of the given steel sample. Thus knowing $C_0 = 0.2$, $C(x,t) = 0.4$, $C_s = 0.8$, erf (y) can be obtained by [$y=x/2\sqrt{Dt}$]. Hence knowing x & t from the experiment D can be calculated.

0.2% Carbon steel is packed carburized in carburizing mixture (30-32% coke, 53-55% charcoal and 2-3% Na_2Co_3, Ba_2Co_3, and 3-4% $CaCo_3$) at 930^{o}C for 4hrs, 6hrs, 8hrs, 10hrs, and 12hrs. For this purpose, a stainless steel box is filled with the carburizing mixture and the samples are kept inside the mixture. The box then is sealed with high temperature cement to prevent the exposure of carburizing mixture and the samples to the atmosphere to prevent possible oxidation.

PROCEDURE

Answer the Following Questions

1. Explain why the surface hardness of quenched high-carbon steel may be less than the hardness under the surface.

2. Cite three main reasons for surface hardening of steel.

3. Explain why core refining heat treatment may not be required for case carburized aluminium killed steel.

4. Can steel having 0.1% carbon be case carburized at 850° C?

5. Show that the inter-lamellar spacing of pearlite is inversely proportional to the degree of under cooling.

6. List the factors that determine the strength of properly hardened steel.

7. Explain why thicker sections are more susceptible to cracking during hardening heat treatment.

8. A hardened steel has become embrittled on tempering. Can this be de-embrittled?

9. Microstructure of isothermally transformed pearlite should have identical spacing in all colonies. However often its microstructure often shows that lamellar spacing varies from colony to colony. Why is it so?

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

EXPERIMENT 8

PRECIPITATION HARDENING

OBJECTIVE

To study the effect of precipitation hardening treatment on Al-4% Cu alloy on Isothermal ageing.

EQUIPMENT / MATERIALS

Samples of Al-4%Cu alloy, Electric furnace, SiC papers of various grit sizes, Polishing machine, Metallurgical microscope, Rockwell hardness tester.

THEORY

Materials can be hardened by inhibiting the motion of crystal defects called dislocations. In pure metals, the presence of defects (such as vacancies, interstitials, dislocations and grain boundaries) can enhance the strength. In single phase alloys, additional resistance to deformation may arise from the presence of foreign atoms. In two-phase alloys, additional stress is needed to enable the dislocation to intersect the second-phase particles. A finely dispersed precipitate may, therefore, strengthen the material. This phenomenon is termed **precipitation hardening.**

The thermodynamics of precipitation in 2014 Al can best be understood by referring to the binary phase diagram of Aluminum-Copper in the aluminum-rich region in *Figure 1*.

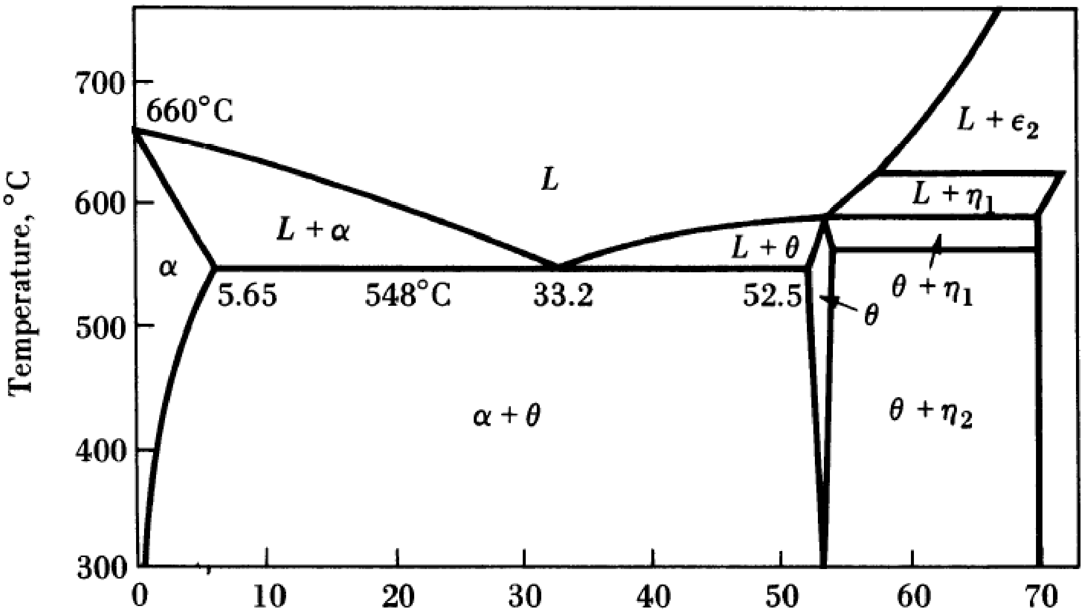


Figure 1. Binary Phase Diagram for Al-Cu

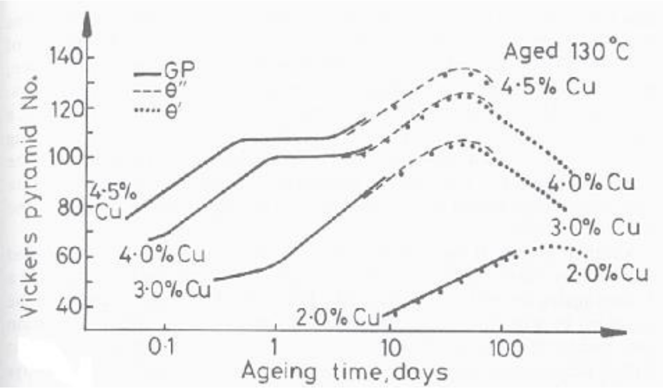
When the aluminum-copper alloy of less than 5 wt% copper is heated to a temperature just above the solvus line, only one phase (alpha, α) is thermodynamically stable. Other solid phases dissolve (disappear). This process is called solution treatment. The only requirement is that the specimen must be kept at this temperature for a long enough time. To solution treat a sample of 2014 Al (4 wt% Cu), the sample should be heated to 930^0F (500^0C) and held for 30 minutes.

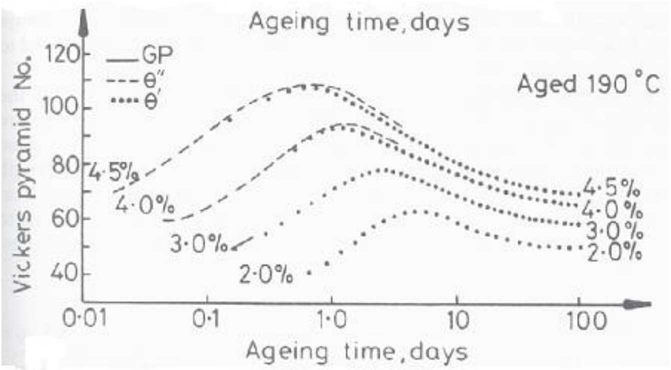
When a solution treated sample is rapidly cooled (quenched) to below the solvus line (Figure 1), two phases are thermodynamically stable (alpha and theta). These phases are two different solids, physically distinct, and separated by a phase boundary. The process is similar to precipitation of salt in supersaturated brine.

The process of precipitation is not instantaneous, as is often the case in liquid-solid precipitation. The process involves the formation of embryos of theta through thermal fluctuations and their subsequent growth, once they achieve stability. With time, more and more precipitates form. This process is called aging. Once the solution achieves an equilibrium composition given by the solvus line for the aging temperature, precipitation stops. For example, the precipitation of the copper-rich theta phase depletes the kappa phase of copper to approximately 1-1/2 wt% Cu at 715°F (380^0C).

The distribution of precipitates affects the hardness and yield strength. The hardness and yield strength are greater when the precipitates are small and finely dispersed in the kappa matrix than when the precipitates are large and not finely scattered. Therefore, to gain hardness in 2014 Al, the specimen should be heat treated to produce a fine dispersion of small precipitates. Unfortunately, there is a tendency when thermodynamic equilibrium is reached for large precipitates to grow and small precipitates to shrink. This will lower the surface to volume ratio of the precipitates, the surface energy, and therefore the energy of the system. As a result, at some point in the aging of 2014 Al, the precipitates begin to coarsen and (on average) the spacing between them gradually increases. At this point, the hardness and the yield point will begin to decrease with time of aging.

The process of aging is a function of temperature; the higher the temperature, the wider the spacing of the precipitates. They form initially on cooling from the solution treatment. Also, because coarsening is dependent upon the movement of copper atoms in kappa, the maximum point is generally reached sooner at a higher temperature than at a lower temperature, as shown in figure.





PROCEDURE

OBSERVATIONS

1. Record the date in following table

   Solution treatment temperature:

   Hardness of Quenched specimen:

   Aging temperature:

Sl.No	Ageing Time	Hardness

2. Plot Hardness vs ageing time curve

Answer the Following Questions

1. Discuss the variation in hardness as a function of ageing time.

2. How will the peak hardness vary with ageing temperature?

3. How will the hardness vs ageing time curve shift for an alloy with 5 percent copper?

4. Aluminum alloy rivets can be deformed easily when put in place but age-harden at room temperature. How could one salvage a batch of rivets which had age-hardened before they had been used?

5. What methods other than hardness measurement could be used to study the precipitation phenomenon? Discuss any of them.

6. What are the stages of precipitate formation in precipitation strengthened alloys?

7. What is the essential condition for age hardening?

8. What is the structural difference between GP zones and θ

9. What’s the difference between a Coherent and a Non-Coherent Precipitate?

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

EXPERIMENT 9

DIFFERENTIAL SCANNING CALORIMETRY

OBJECTIVE

To study the phase transformation behavior of an unknown sample using DSC (Differential Scanning Calorimetry). Though it is unknown, rough idea about the sample character can be utilized in analyzing the data.

EQUIPMENT / MATERIALS

Differential thermal analyzer, potentiometer for temperature measurement, micro-voltmeter for differential EMF measurement, Pb-Sn eutectic alloy.

THEORY

Principle

DSC measures the differences in heat flow into a substance and a reference as a function of sample temperature while both are subjected to a controlled temperature program. DSC provides access to accurate thermodynamic data as well as information regarding reactivity and phase transformations**.**

DSC – types

DSC is the most sophisticated and advanced of the thermal methods. There are two principal types:

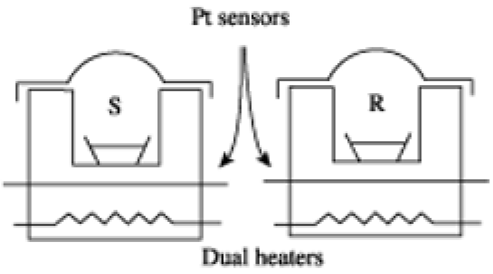
- Power compensated DSC
- Heat-flux DSC

Power Compensated DSC-principles

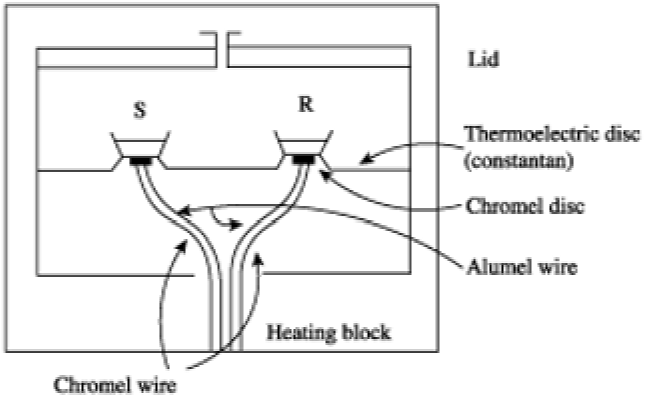
Temperature difference is maintained zero, i.e., $\Delta T = 0$, by supplying heat into the sample or reference according to heat emission or absorption Electrical power is proportional to heat change in the sample

Working principle

Samples are contained in shallow pans, with the aim of making a good thermal contact between sample, pan and heat flux plate. Symmetrical heating of the cell is needed, (Sample(S) and Reference(R)), and this can be achieved by constructing the furnace from a metal of high thermal conductivity – for example, silver. Sample and reference material are heated by separate heaters in two independent furnaces (Fig. 11.1). The furnaces are imbedded in a large temperature - controlled heat sink. Sample holders are above the furnaces. Pt resistance thermometers are imbedded in the furnaces to monitor the temperatures of sample and reference continuously. There is provision for establishing gas flow through the cell, to sweep away volatiles, provide the required atmosphere, and to assist in heat transfer.



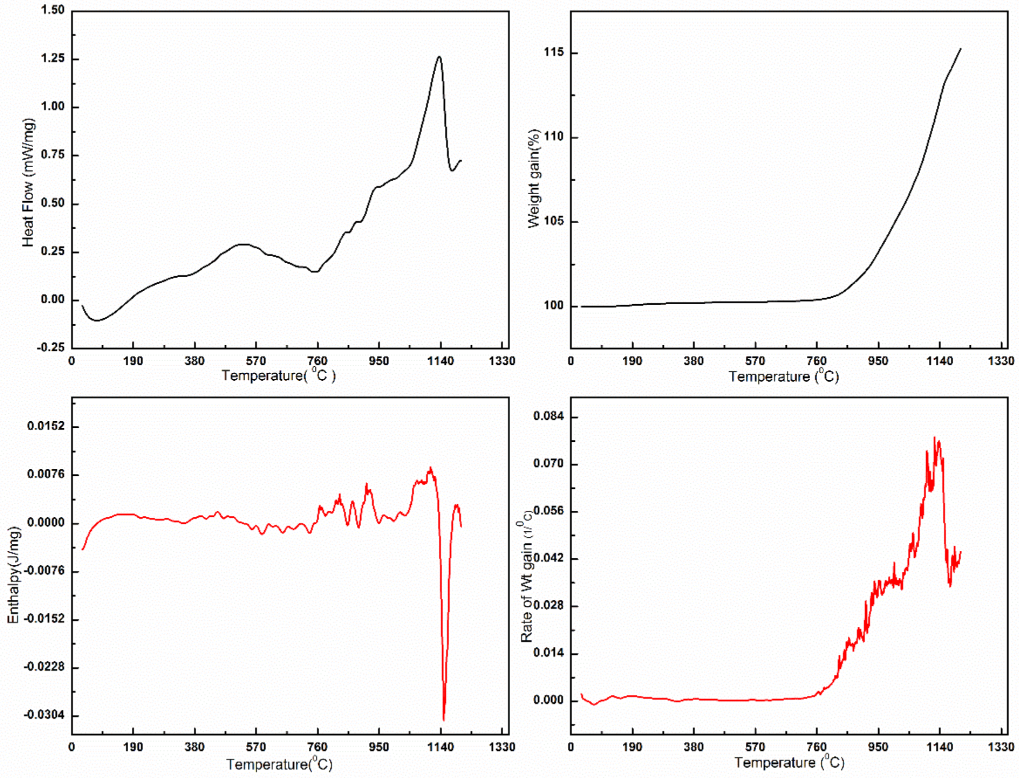
Power compensation DSC



Heat flux DSC

Sample and Reference are heated by a single heater. Differential heat flow into the S and R pans is monitored by thermocouple. Gas purging is done to avoid oxidation of the sample.

Enthalpy changes



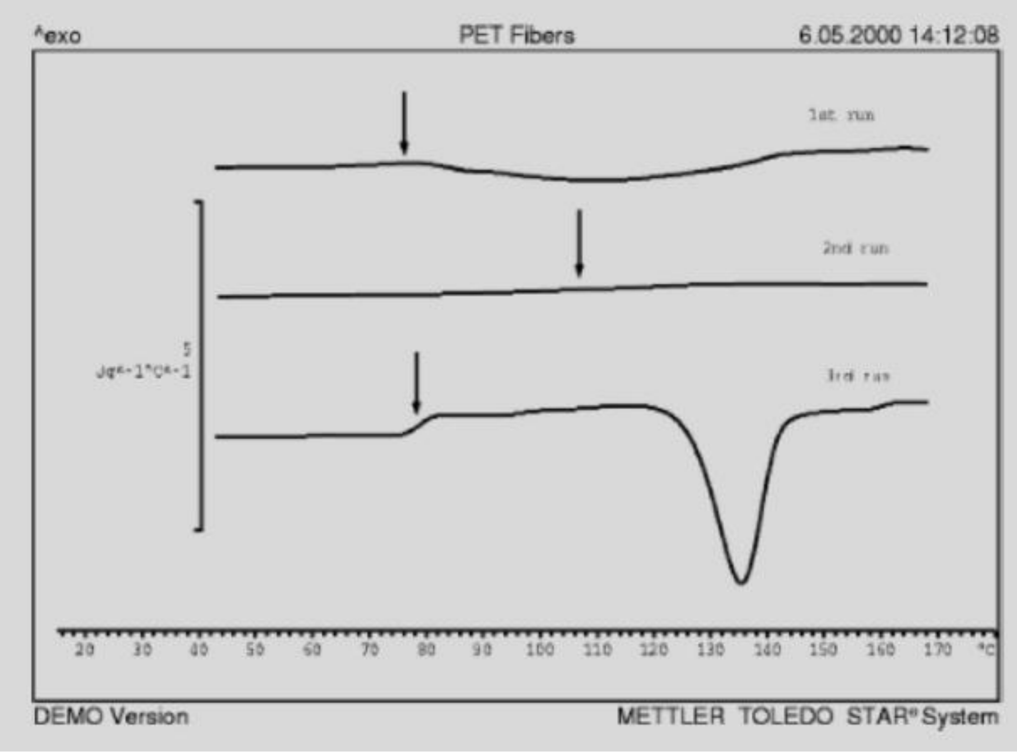
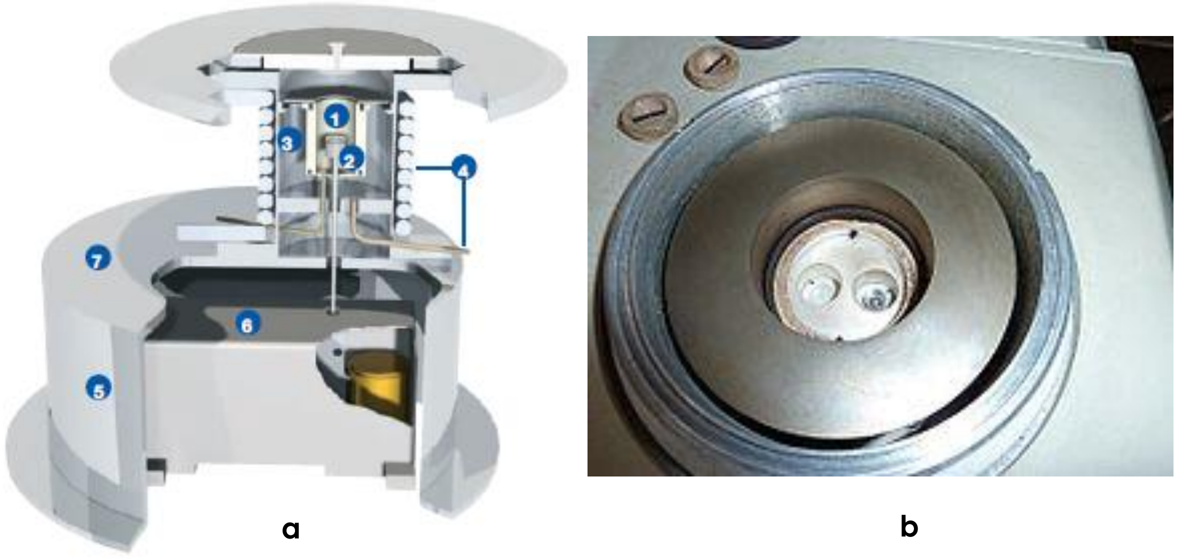


Figure: Glass transition of stretched PET fibers. The arrows mark the glass transition

Some possible processes giving enthalpy peaks

Process	Exothermic	Endothermic
Solid-solid transition	✓	✓
Crystallization	✓	
Melting		✓
Vaporisation		✓
Sublimation		✓
Adsorption	✓	
Desorption		✓
Desolvation (drying)		✓
Decomposition	✓	✓
Solid-solid reaction	✓	✓
Solid-liquid reaction	✓	✓
Solid-gas reaction	✓	✓
Curing	✓	
Polymerization	✓	
Catalytic reactions	✓	

EQUIPMENT



(a) Parts of perkinelmer STA 6000, (b) Sample Holder and Reference ring

1. The small furnace enables accurate temperature control, minimizes the time to achieve a pure gas environment and allows for the fastest cool down/turnaround times.
2. The SaTurnA sensor measures both the sample and reference temperature. The vertical loading balance allows for easy operation and sample loading.
3. The rugged alumina furnace is corrosion-resistant allowing for a wide variety of reactive gases to be used. Its large isothermal zone provides excellent temperature reproducibility.
4. Experience unsurpassed furnace cool-down speed with the chiller and integral forced air features and allow your laboratory to process more samples in less time.
5. The balance housing’s thick, stainless steel walls act as a large heat sink, thermally isolating the balance from the furnace.
6. Balance purge gas maintains a constant environment for the balance and protects it from the reactive sample purge gas as well as materials evolving from the sample.
7. Sample purge gas: the integrated mass flow controller provides accurate environmental control and meets the needs of methods that call for specific gas flow rates, or for gas switching during the analysis.

OBSERVATIONS

Answer the Following Questions

1. What is the transition temperature?

2. Describe briefly how the DSA technique is performed and sketch a typical DSA plot for the melting of a pure metal.

3. What are the advantages and disadvantages of the DSA?

4. Describe briefly how DSC differs from DTA and sketch a typical DSC plot for the melting of a pure metal.

5. What are the advantages and disadvantages of the DSC?

6. What is the effect on the shape of the free-energy curve for a solution if its interaction parameter is positive?

7. What is glass transition temperature (T_g)? Draw a typical plot for showing the Tg of a material.

8. What are the factors that govern the choice of chromel-alumel or Pt-Pt 10% Rh wires for differential thermocouple?

9. What is meant by reference standard? How one can select reference standard for different materials?

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

EXPERIMENT 10

CASE STUDY

1. Post weld treatment
2. Surface Hardening of steels
3. Zone refining in electronic materials
4. Development of Bainitic steels
5. Heat treatment of Titanium & alloys
6. Heat treatment of Superalloys (Ni-base)
7. Shape memory alloys
8. Development of Peak aging condition in Al-alloys
9. Dual Phase steels
10. Bearing steels
11. Gears
12. Spring steels
13. Boiler steels (P 91 or 92)
14. Rail steels
15. Austenitic stainless steels
16. Martensitic stainless steels
17. Duplex stainless steels
18. Sintering of powder metallurgy components
19. Development of Intermetallic
20. Choice from your side (if it related to this lab)

Select any topic above given, and study the heat treatment cycles for above materials with required properties and applications.

PRESENTATION FORMAT

OBJECTIVE

COMPOSITION

ROLE OF ALLOYING ELEMENTS

HEAT TREATMENT CYCLES

PROPERIES

Microstructural characteristics

Mechanical properties

APPLICATIONS

PRESENT STATUS

REFERENCES