

General Information

Total Laboratory Turns: 12 [6 experiments in first session (before midsem) and 5 experiments in next session (after mid sem) + 1 turn project presentation]

Lab Viva/Lab Quiz will be either after both sets of experiments or after each set of experiment.

Each day, viva will be taken on the experiment.

Marks Distribution of Lab:

a) Daily Lab Job	15%
b) Lab Report	10%
c) Daily Lab Viva	10%
d) Project	25%
e) Final Lab Exam/ Lab Viva	40%

Safety:

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

Rules:

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

Guideline for the project:

- Each group will be assigned one project. Project allotted through lottery.
- Project will be completed using lab facilities and given time frame only.
- Special lab facilities such as furnace, XRD, DSC, Hardness Tester, etc.; you should book at least one week before.
- Prepare a project report contains Aim of project, Facilities/equipment used, Brief description of process, Result & Discussion, Utility & Limitation of process, Future scope, etc. Please do not include any net downloaded materials in project report.

Recommended Reading:

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

MSE 311 : Physical Metallurgy Lab

(SET-2)

After Mid Semester

	Experiment 1	Experiment 2	Experiment 3	Experiment 4	Experiment 5	Project Presentation	Remarks
Monday	21-Sep-15	28-Sep-15	5-Oct-15	12-Oct-15	26-Oct-15	2-Nov-15	Gr. A to Gr. D
Tuesday	22-Sep-15	29-Sep-15	6-Oct-15	13-Oct-15	27-Oct-15	3-Nov-15	Gr. E to Gr. H
Thursday	24-Sep-15	1-Oct-15	8-Oct-15	15-Oct-15	29-Oct-15	5-Nov-15	Gr. I to Gr. L
MONDAY							
Experiment Date↓	OM of ceramics	Nucleation & growth	Recovery	Carburization	DSC	Project Presentation	Remarks
21-Sep		all students will complete their 'OM of ceramics' experiment				For Project presentation necessary instructions will be given later	
28-Sep	All Students of Monday Batch (21/09/2015)	Group A	Group B	Group C	Group D		
05-Oct		Group D	Group A	Group B	Group C		
12-Oct		Group C	Group D	Group A	Group B		
26-Oct		Group B	Group C	Group D	Group A		
TUESDAY							
Experiment Date↓	OM of ceramics	Nucleation & growth	Recovery	Carburization	DSC	Project Presentation	Remarks
22-Sep		All students will complete their experiment of 'OM of Ceramics'				For Project presentation necessary instructions will be given later	
29-Sep	All Students of Tuesday Batch (22/09/2015)	Group E	Group F	Group G	Group H		
06-Oct		Group H	Group E	Group F	Group G		
13-Oct		Group G	Group H	Group E	Group F		
27-Oct		Group F	Group G	Group H	Group E		
THURSDAY							
Experiment Date↓	OM of ceramics	Nucleation & growth	Recovery	Carburization	DSC	Project Presentation	Remarks
24-Sep		All students will complete their experiment of 'OM of Ceramics'				For Project presentation necessary instructions will be given later	
01-Oct	All Students of Thursday Batch (24/09/2015)	Group I	Group J	Group G	Group L		
08-Oct		Group L	Group I	Group J	Group G		
15-Oct		Group G	Group L	Group I	Group J		
29-Oct		Group J	Group G	Group L	Group I		

Experiment 7

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.

Background:

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 is observed in most of the industrially important alloys, consists of the appearance of microscopic regions of the 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 (N) and the rate of growth (G). The rate of nucleation 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.

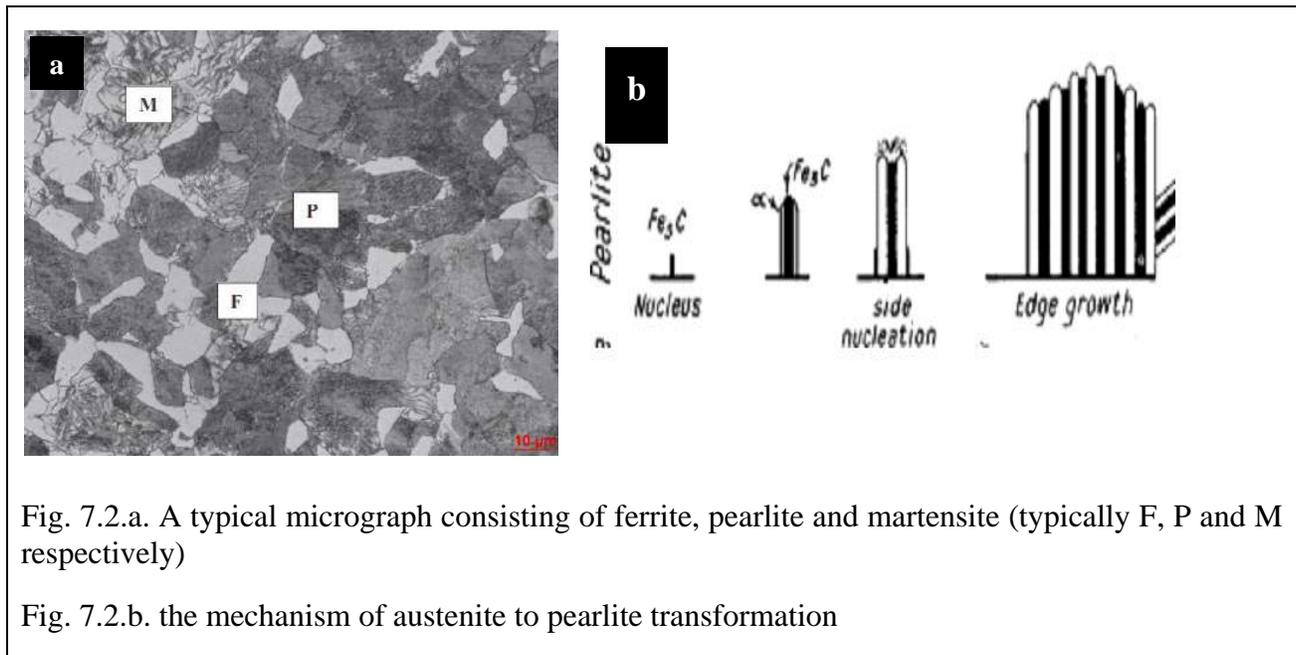
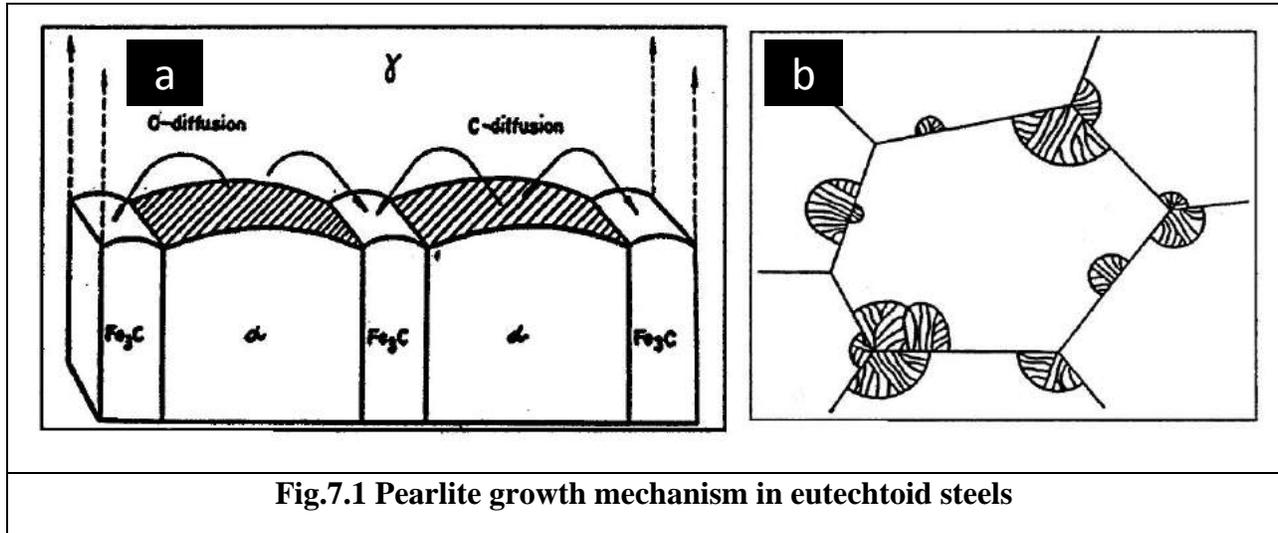
When the probability of the appearance of a nucleus in any small volume of the parent phase is same as that in any other small volume of the parent phase, nucleation occurs randomly all over the parent phase at the same rate (homogeneous nucleation). However, if high energy sites are present in the parent phase nucleation may occur preferably on these 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 austenitization to a temperature below the eutectoid temperature, and held for transformation to occur (Isothermal transformation - TTT diagram).

Eutectoid Steel:

0.8% C (wt %) is called eutectoid steel, where eutectoid reaction (γ to α 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 (7.1). The alternating layers of ferrite and cementite are collectively referred to as pearlite (Figure 7.1.a). The pearlite appears to grow into the adjoining grains as nodules as shown in Figure 7.1.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.

In this lab we will study the kinetics of the (\rightarrow 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.



Equipment and materials:

1. Samples of 0.7% carbon steel
2. Furnace with temperature controller, for temperature upto 1000°C.
3. Rockwell hardness tester.

4. Microscope.
5. Metallographic polishing equipment.
6. Belt grinder.
7. Etching agent

Procedure:

1. Pre-treatment:

3 samples of 0.7% carbon steel (nearly eutectoid composition) are austenitised at 1000°C for half an hour. These are then separately kept at 690°C and soak them for 3, 8 and 30 minutes. After soaking they are finally quenched in water. In this treatment, austenite (γ phase) at 1000°C when cooled to 690°C, pearlite will start nucleating on the grain boundaries. The nucleated pearlite then grows with time, till the complete austenite phase transforms into pearlite for 0.8% carbon steel till the complete austenite phase transforms into pearlite of 0.8% carbon steel. However, for 0.7% carbon steel alpha (α) ferrite will appear along the grain boundaries.

2. Polishing:

Polish the given samples with the appropriate emery papers and final polishing is done in velvet cloth using alumina suspension. The samples are then etched with nital (3% Nitric acid in alcohol)

3. Observation:

Observe the microstructure of the samples under the microscope (as in Figure 7.2.a) and measure the diameter or size of pearlite colonies in different region randomly in all the 3 samples. Take at least 5 readings for each sample.

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.

To observe austenite nucleation during heating (To do)

1. Low carbon steel (0.1-0.3%) with a microstructure ferrite + pearlite is heated to 1000°C
2. Holding the sample at 1000°C for 5, 10, 15 min
3. Then the sample is water quenched
4. Austenite will nucleate at 1000°C and with holding, the austenite will grow
5. The martensite fraction would also change as per the nucleation and growth of austenite during holding at 1000°C
6. Polish, etch and see the structure under optical microscope
7. Draw the schematic as a function of time
8. Is there any change in the hardness of your samples? Explain why. (Hint: relate this to the grain size)

Experiment 8

Recovery and Recrystallization

Objective:

1. To study the recrystallization behaviour of mild steel. Recrystallization behaviour includes,
2. To study the effect of temperature in microstructure of the cold worked materials
3. Measuring the percentage of recrystallization in each sample
4. To find out the time for 50% recrystallization in each sample

Background:

Plastically deforming a polycrystalline metal specimen at temperatures that are low relative to its absolute melting temperature produces microstructural and property changes that include

- (1) a change in grain shape
- (2) strain hardening and
- (3) an increase in dislocation density

Some fraction of the energy expended in deformation is stored in the metal as strain energy, which is associated with tensile, compressive, and shear zones around the newly created dislocations. Furthermore, other properties, such as electrical conductivity and corrosion resistance, may be modified as a consequence of plastic deformation. These properties and structures may revert back to the pre cold-worked states by appropriate heat treatment (sometimes termed an annealing treatment). Such restoration results from two different processes that occur at elevated temperatures: **recovery and recrystallization**, which may be followed by **grain growth**.

Recovery:

During recovery, some of the stored internal strain energy is relieved by virtue of dislocation motion (in the absence of an externally applied stress), as a result of enhanced atomic diffusion at the elevated temperature. There is some reduction in the number of dislocations, and dislocation configurations (Polygonization similar to that shown in Figure 8.1) are produced having low strain energies. In addition, physical properties such as electrical and thermal conductivities recover to their pre-cold-worked states.

Recrystallization:

Even after recovery is complete, the grains are still in a relatively high strain energy state. Recrystallization is the formation of a new set of strain-free and equi-axed grains (i.e., having approximately equal dimensions in all directions) that have low dislocation densities and are characteristic of the pre-cold-worked condition. The driving force to produce this new grain structure is the difference in internal energy between the strained and unstrained material. The new grains form as very small nuclei and grow until they completely consume the parent material

(Shown in Fig 8.1c). Several stages in the recrystallization process are represented in Figures 8.2a to 8.2d; in these photomicrographs. Thus, recrystallization of cold-worked metals may be used to refine the grain structure.

Also, during recrystallization, the mechanical properties that were changed as a result of cold working are restored to their pre-coldworked values; that is, the metal becomes softer and weaker, yet more ductile. Some heat treatments are designed to allow recrystallization to occur with these modifications in the mechanical characteristics.

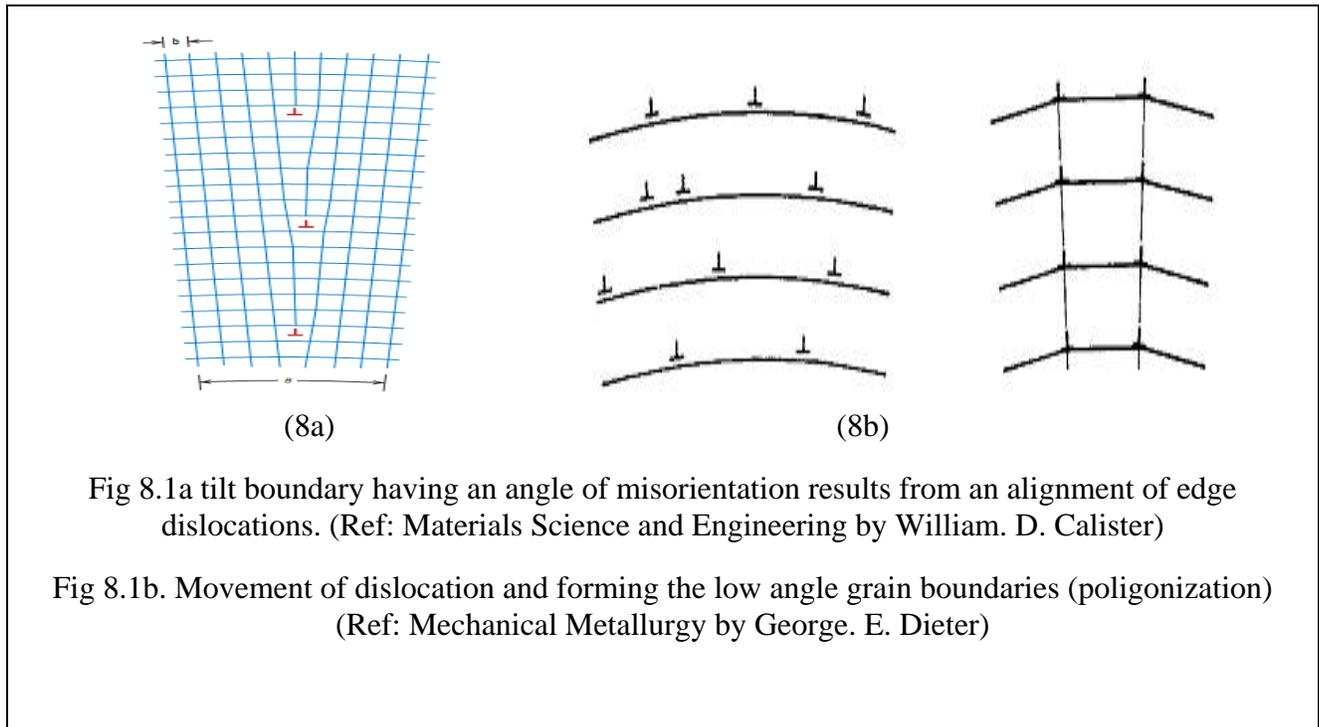


Fig 8.1a tilt boundary having an angle of misorientation results from an alignment of edge dislocations. (Ref: Materials Science and Engineering by William. D. Calister)

Fig 8.1b. Movement of dislocation and forming the low angle grain boundaries (polygonization) (Ref: Mechanical Metallurgy by George. E. Dieter)

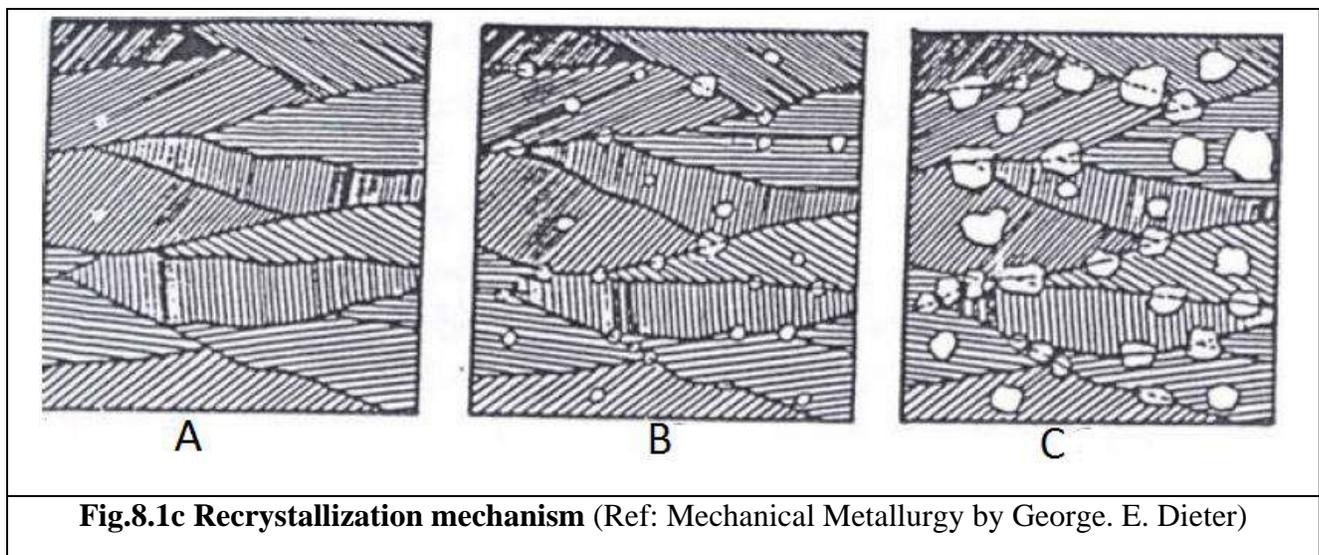
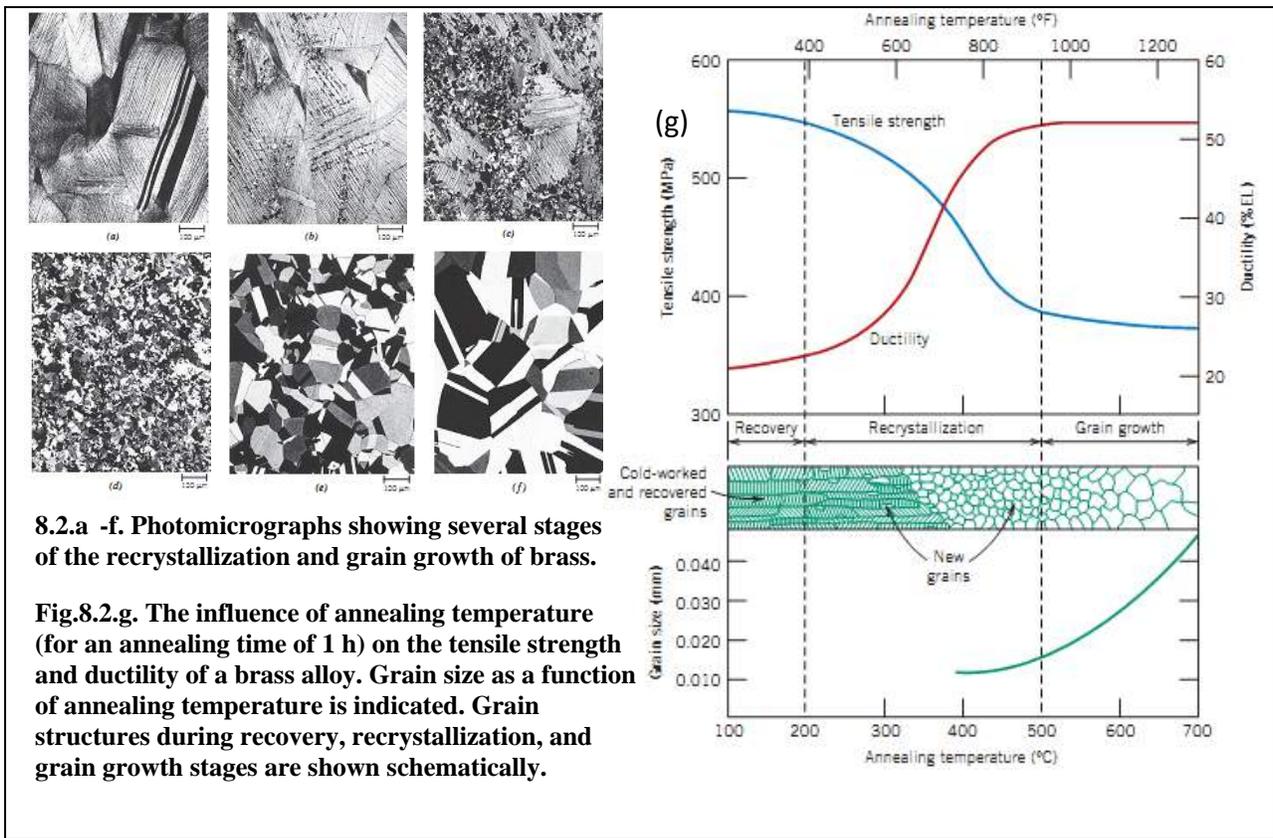


Fig.8.1c Recrystallization mechanism (Ref: Mechanical Metallurgy by George. E. Dieter)



The extent of recrystallization depends on both time and temperature.

The time required for recrystallization is calculated from the Johnson – Mehl equation. ‘X’ is volume fraction of the recrystallized region. This equation suggests the sigmoidal curve.

$$X = 1 - \exp(-kt^n)$$

n is time exponent

This equation can be rewritten as shown below

$$\ln k \dots \dots \dots + n \ln t = \ln \left[\ln \frac{1}{1-x} \right]$$

Plotting $\ln t$ vs $\ln \left[\ln \frac{1}{1-x} \right]$ the exponent ‘n’ can be calculated

The degree (or fraction) of recrystallization increases with time, as may be noted in the photomicrographs shown in Figures 8.2a to 8.2d. The recrystallization temperature can be defined as the temperature at which 50% recrystallization occur in one hour. Recrystallization process is an activated process. The time and temperature required to recrystallize the material can be given by the equation shown below

$$\frac{1}{t} = ke^{-Q/RT}$$

Plotting $\ln(1/t)$ vs $1/T$, the effective activation energy “Q” can be obtained by from the slope of the linear plot.

The influence of temperature is demonstrated in Figure 8.2.g, which plots tensile strength and ductility (at room temperature) of a brass alloy as a function of the temperature and for a constant heat treatment time of 1 h. The grain structures found at the various stages of the process are also presented schematically. The recrystallization behaviour of a particular metal alloy is sometimes specified in terms of a recrystallization temperature, at which recrystallization just reaches completion in 1 h. Thus, the recrystallization temperature for the brass alloy of Figure 8.2.g is about 450° C. Typically, it is between one-third and one-half of the absolute melting temperature of a metal or alloy and depends on several factors, including the amount of prior cold work and the purity of the alloy.

Recrystallization proceeds more rapidly in pure metals than in alloys. During recrystallization, grain-boundary motion occurs as the new grain nuclei form and then grow. It is believed that impurity atoms preferentially segregate and interact with these recrystallized grain boundaries so as to diminish their (i.e., grain boundary) mobility. This results in a decrease of the recrystallization rate and raises the recrystallization temperature, sometimes quite substantially. For pure metals, the recrystallization temperature is normally $0.4T_m$, where T_m is the absolute melting temperature; for some commercial alloys it may run as high as $0.7T_m$.

Procedure:

Pre-treatment:

Three sets of samples of Metal annealed at 800°C for 0.5 hour and deformed by 50% reduction. These samples are then recrystallized at 600°C, 650°C, and 700°C for different times at each temperature.

Polishing:

Polish the given samples with the appropriate emery papers and final polishing is done in velvet cloth using alumina suspension. The samples are then etched with nital (3% Nitric acid in alcohol)

Observation:

Observe the microstructure of the samples under the microscope and note the following:

- a. Draw the microstructure of the each samples in order with increasing temperature and time with all the details like magnification, temperature and time
- b. Measure percentage of the recrystallization in each sample by quantitative metallography (systematic point counting technique)
Plot $\ln t$ vs $\ln [\ln (1/(1-x))]$ and find the exponent n from the slope.
- c. Find the time (t) for 50 % recrystallization in each case and then by plotting $\ln(1/t_{0.5})$ vs $1/T$, calculate effective activation energy Q from the slope.

To do:

1. Cold rolled mild steel sample is given at different cuts at different planes.
2. Observed the microstructures.
3. The cold rolled samples to be heat treated at 680°C for 30 min and 60 min.
4. Observe the microstructure on LT surface and report.

Experiment 9

Carburization of Steel

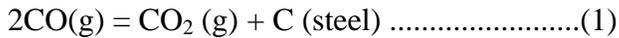
Objective:

To carburize the carbon steel and measure the diffusion coefficient

Background:

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.



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,

$$\frac{dc}{dt} = \frac{d}{dx} \left[D \frac{dc}{dx} \right] \dots\dots\dots(2)$$

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 $D \neq D(c)$

$$dc/dt = D(d^2c/dx^2) \dots\dots\dots(3)$$

The solution to this equation (3) is of the general form in case of the diffusion couple consisting of a constant concentration on the surface (C_s i.e. for carburizing process) is given by

$$\frac{C_s - C(x,t)}{C_s - C_0} = \text{erf} \frac{x}{2\sqrt{Dt}} \dots\dots\dots(4)$$

Where C_s 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.

Procedure:**Carburizing:****Pre-treatment:**

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°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.

Polishing:

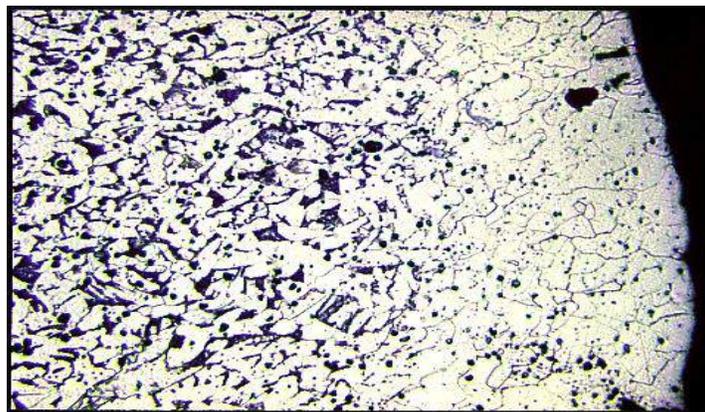
Polish the given samples with the appropriate emery papers and final polishing is done in velvet cloth using alumina suspension. The samples are then etched with nital (3% Nitric acid in alcohol)

Observation:

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

To do

1. Two samples (one carburized and another decarburized low –medium carbon steel) are given to you.
2. Decarburization will also be carried out in the laboratory by keeping the medium carbon steel at 1000°C for 1hrs followed by air cooling.
3. Observe the microstructure.
4. Report the changes of microstructures from surface to bulk for both the samples.



Decarburization of steel sample

[<http://www.georgesbasement.com/Microstructures/LowAlloySteels/Lesson-1/Specimen02.htm>]

Experiment 10

Optical Microscopy of Ceramic samples Colour Metallography and phase contrast microscopy of non-metallic materials

Objective:

To study and observe the microstructure of ceramic samples such as graphite, SiC, ZrO₂ and Hydroxyapatite.

Background:

Ceramics are composed of at least two elements and often more. Their crystal structures are generally more complex than those of metals. The atomic bonding in these materials ranges from purely ionic to totally covalent. Many ceramics exhibit a combination of these two bonding types. The properties of ceramics are

- High Young modulus and high melting points : strong bonds (covalent and/or ionic)
- Limited electrical and thermal conductivity : Absence of electronic cloud (directional bond)
- Low thermal shock resistance : coefficients of thermal expansion and thermal conductivity are low
- Refractory : Stability at high temperature (less creep)
- Resistance to oxidation/corrosion : chemical stability and inertness

Some of the major engineering use of ceramics:

Ceramic samples are generally sintered at high temperature either by solid state or liquid-state sintering routes. Therefore, you would be able to see porosity as well as neck formation.

Experiment 11

Thermal Analysis using DSC to study phase transformations

Objective:

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

Background:

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 are 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.

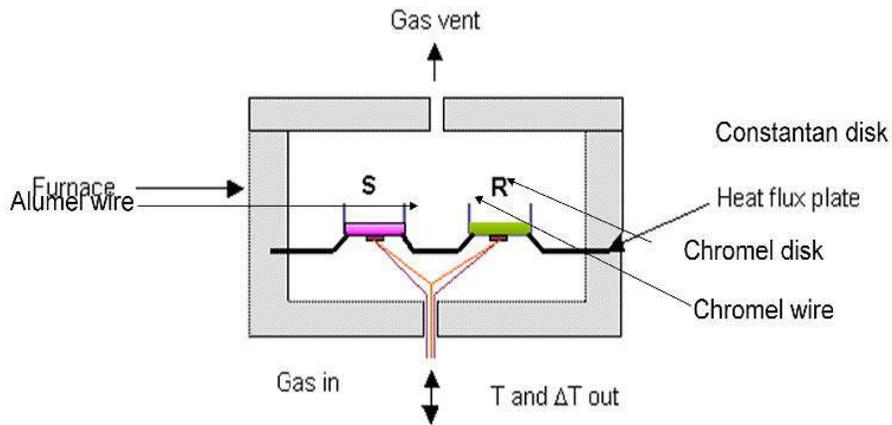


Fig 11.1 Heat flux in DSC

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.

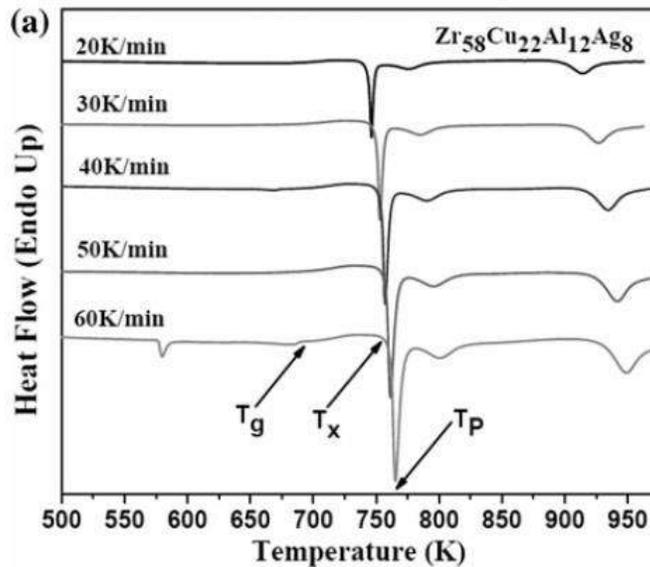


Fig 11.2 shows the typical DSC diagrams

Enthalpy changes:

The DSC curve of typical metallic glass shows (Fig.11.2) glass transition and crystallization. The area under the peak is the enthalpy for crystallization.

PerkinElmer STA 6000

In our laboratory, we are using the model PerkinElmer STA 6000 for DSC analysis.

Some possible processes giving enthalpy peaks:

Process	Exotherm	Endotherm
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	*	

Description:

The STA 6000 features the innovative SaTurnA Sensor for high quality, simultaneous TG and DTA/DSC measurements. This advanced sensor is optimized to achieve flat DTA baselines and high sensitivity. Because both sample and reference are measured simultaneously, you can be sure of the integrity of your analysis. The corrosion-resistant, pure platinum pan holder and reference ring make the instrument suitable for a wide variety of samples and applications. While many methods call for specific gas flow rates, others may require a gas switching during the analysis. Either way, the STA 6000 with its built-in mass flow controller both monitors and controls the purge flow rates.

An inside look at the STA 6000:

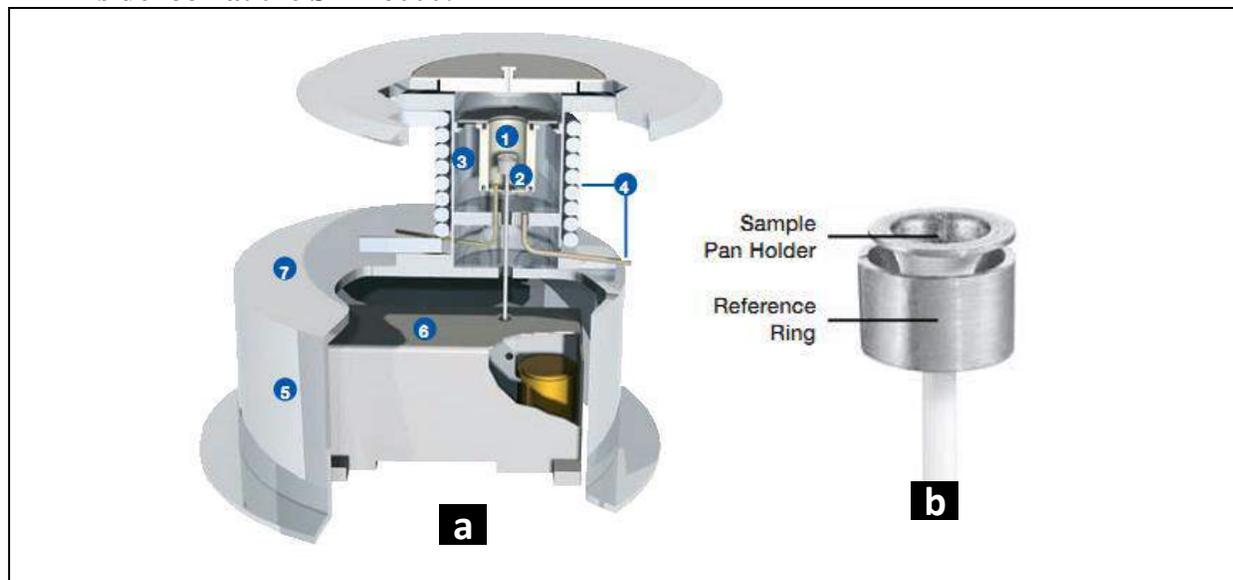
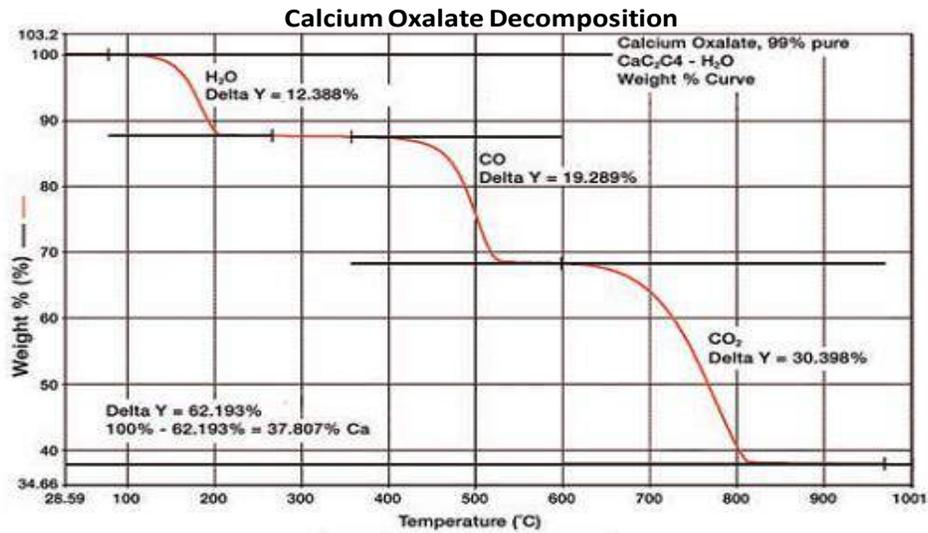


Fig.11.3. Parts of perkinelmer STA 6000, b. Sample Holder and Reference ring

http://www.perkinelmer.com/Content/RelatedMaterials/Brochures/BRO_STA-6000.pdf

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. For unattended operation, equip your STA 6000 with an optional 45 position autosampler.
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.

Applications of Thermal analysis (one such example):



Observation and Calculation:

- Plot ΔH Vs T for each sample from given data
- Calculate the peak area (mJ) during heating for each sample