HEAT TREATMENT LABORATORY MANUAL

Er. SUBRAT KUMAR BEHERA

Lecturer in Metallurgy

DIPLOMA

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Department of Metallurgical Engineering

ORISSA SCHOOL OF MINING ENGINEERING, KEONJHAR

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HEAT TREATMENT PROCESSES

In general, heat treatment can be defined as an operation, or the combination of operations that involve heating and cooling of a metal in solid phase to obtain certain required properties.

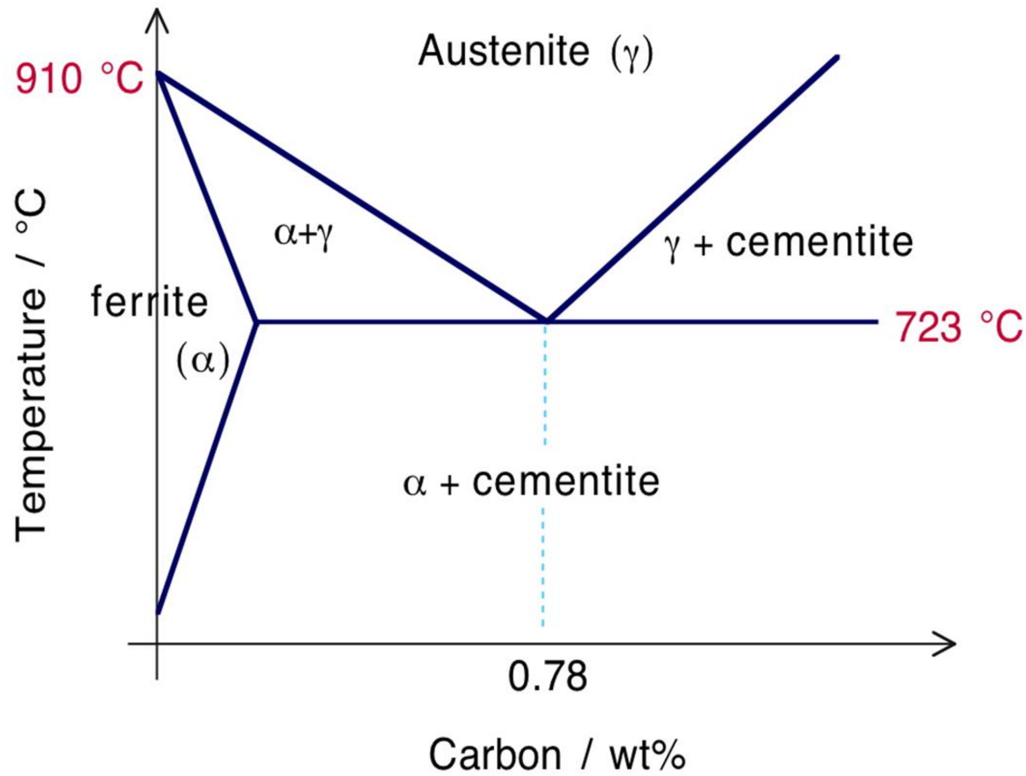
The ferrous materials can be heated to above transformation temperature and can be heat – treated to obtain different structure.

The different heat treatment processes are based on heating the material to certain temperature and employing different cooling rates.

In this process, heating temperature and rate of cooling adopted plays an important role.

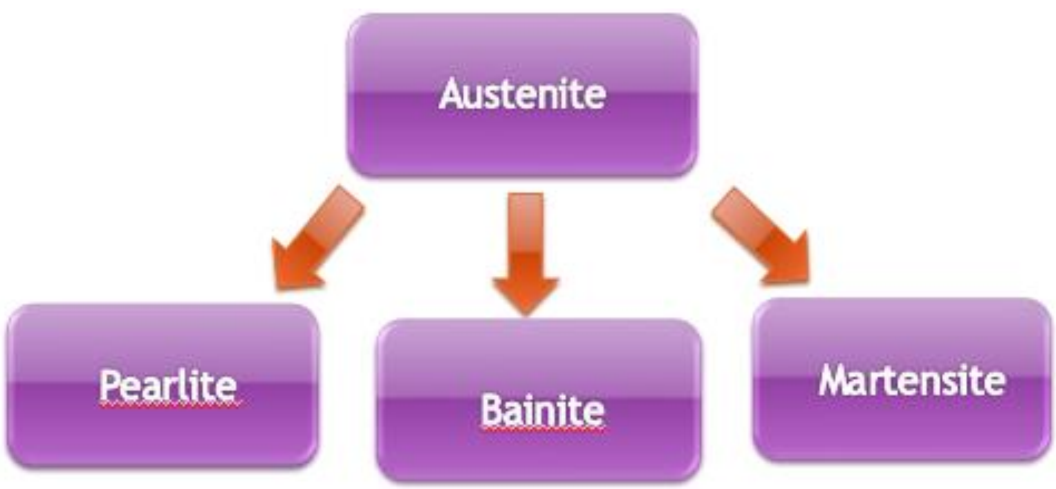
The different processes are:

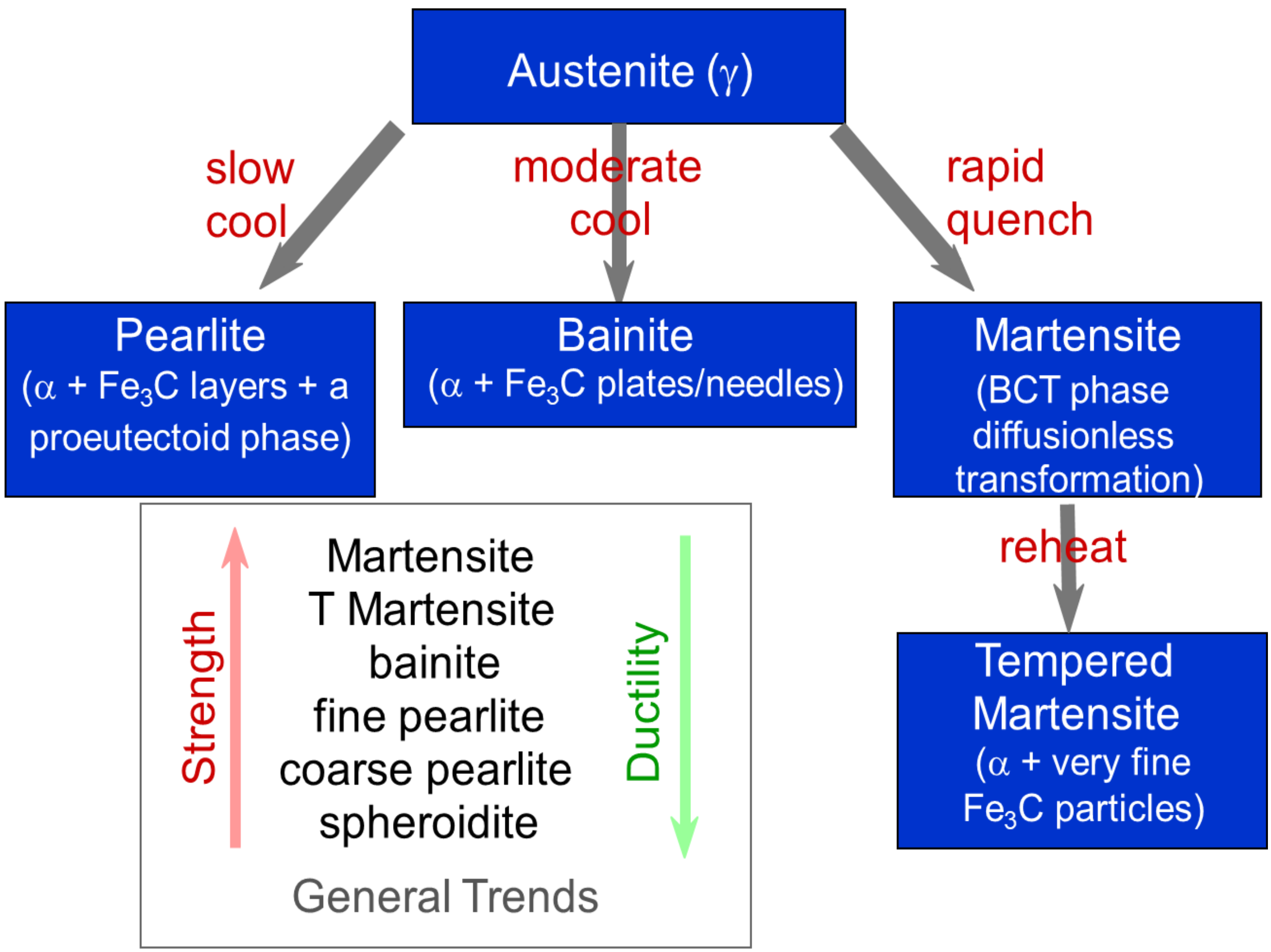
- **Annealing**
  - **Stress-relief annealing.**
  - **Process annealing.**
  - **Spheroidsing.**
  - **Full annealing.**
- **Normalizing**
- **Hardening(Quenching)**
- **Tempering**



AUSTENITE

- Austenite, also known as gamma phase iron (γ-Fe), is a metallic, non-magnetic allotrope of iron or a solid solution of carbon in gamma iron (γ-Fe).
- It forms above 723^0C.
- It has a FCC crystal structure.
- The maximum solubility of carbon in austenite is 2.13 % at 1147Oc





Austenite (γ)
slow cool
moderate cool
rapid quench
Pearlite
(α + Fe_3C layers + a proeutectoid phase)
Bainite
(α + Fe_3C plates/needles)
Martensite
(BCT phase diffusionless transformation)
reheat
Tempered Martensite
(α + very fine Fe_3C particles)
Strength
Martensite
T Martensite
bainite
fine pearlite
coarse pearlite
spheroidite
Ductility
General Trends

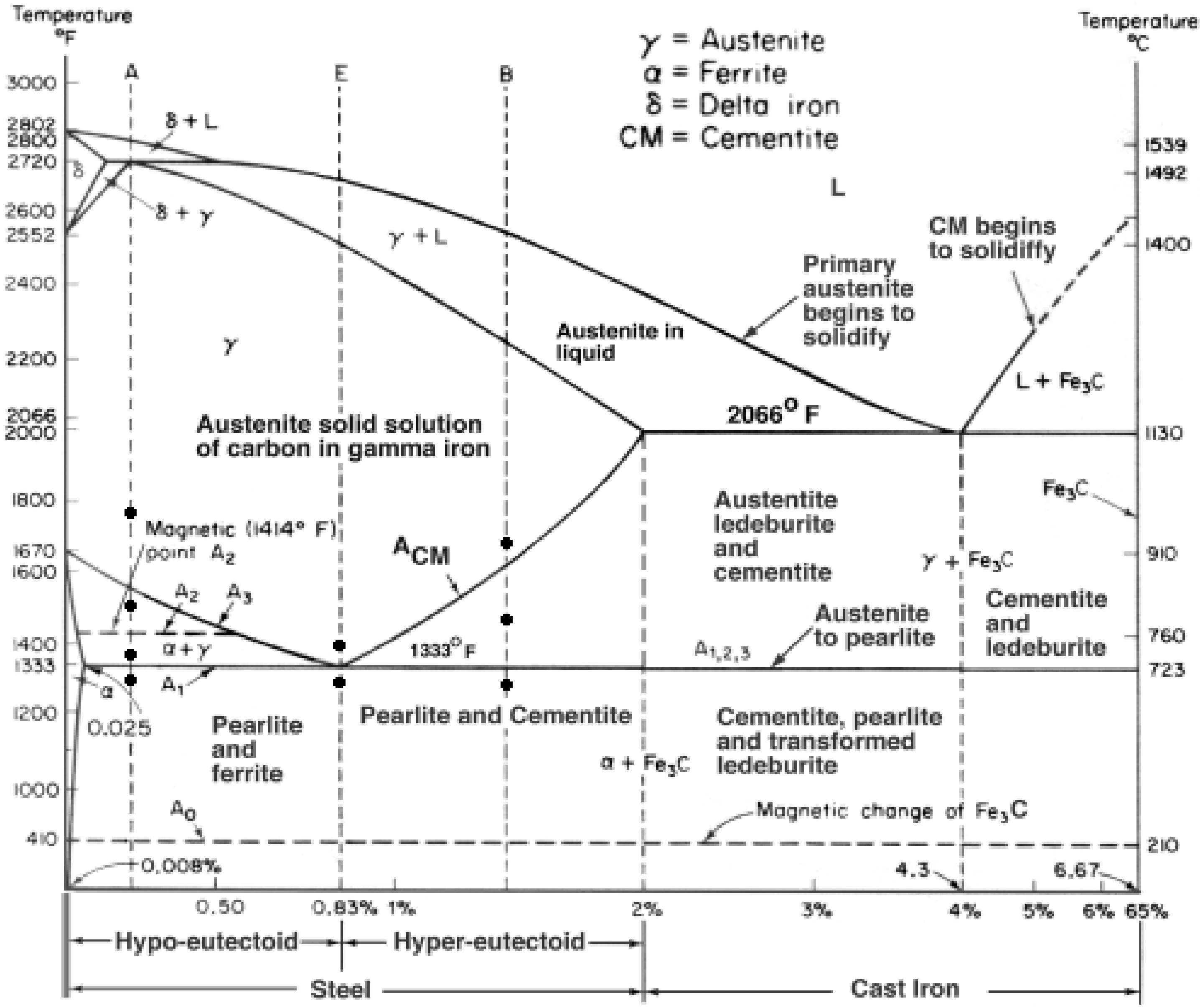


Fig: - Iron- Carbon Diagram

EXPERIMENT-1

Aim: - Annealing of plain carbon steel

Apparatus Required: -

- Hacksaw blade
- Plain Carbon Steel (Mild Steel) Sample
- Muffle Furnace
- File
- Emery Paper
- Double disc polisher
- Etchant (Nital) [98% C_2H_6OH + 2% HNO_3]
- Metallurgical Microscope

Theory: -

Annealing generally involves heating to a predetermined temperature, holding at this temperature and finally cooling at a very slow rate

The temperature and holding time depend on a variety of factors such as composition, size, shape and final properties desired

Annealing treatment can be classified into subdivisions based on temperature of treatment, phase transformation occuring during the treatment and the purpose of the treatment.

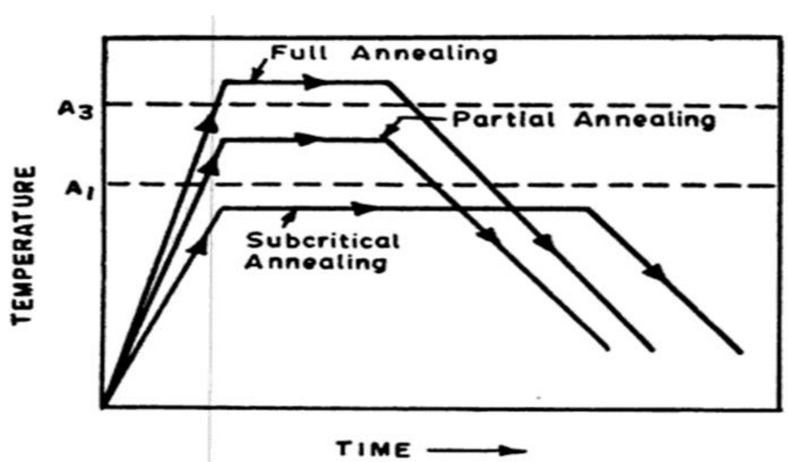
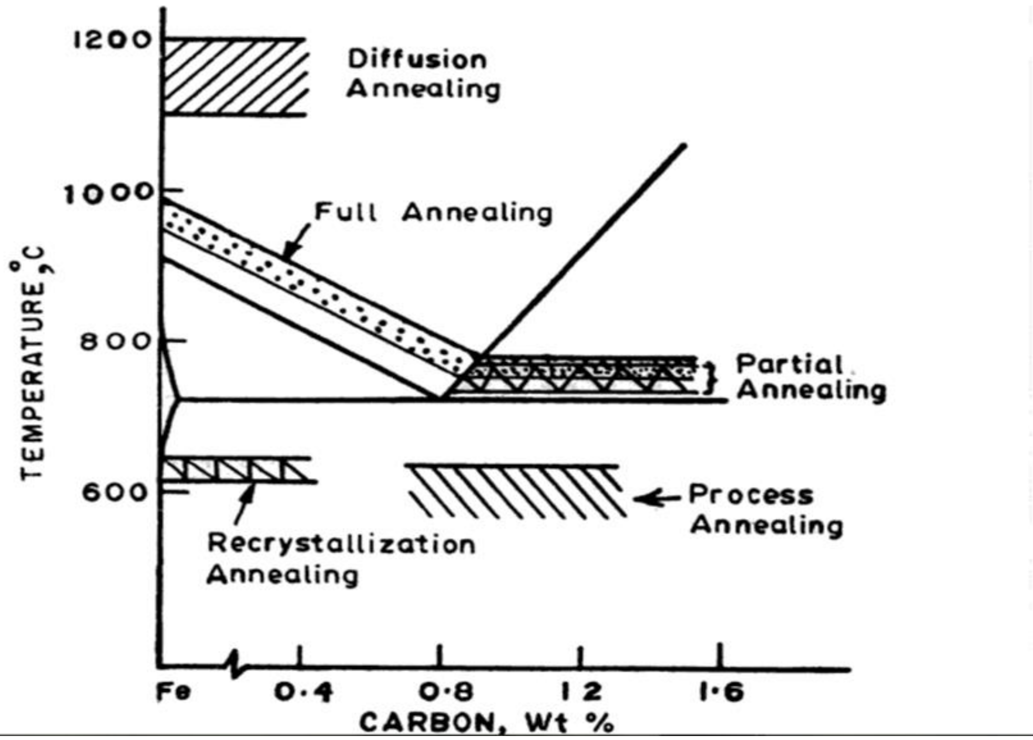


Fig. 5.1 Various types of annealing processes classified on the basis of annealing temperature.

Annealing serves the following purposes:

- ❖ Relieve internal stresses developed during solidification, machining, forging, rolling and welding
- ❖ Improve or restore ductility and toughness
- ❖ Enhance machinability
- ❖ Eliminate chemical non uniformity
- ❖ Refine grain size
- ❖ Reduce gas content in steel



The three important parts of full annealing are:

1. Proper austenitising temperature
2. Soaking time
3. Very slow cooling through A1(critical temperature)

Proper Austenitising Temp: the austenitising temp varies with variation in carbon%. Proper austenitising temp is required to get fine grains of austenite

- ⦿ Soaking Time: soaking at the austenitising temp is of utmost importance as it leads to formation of homogeneous austenite

- Very Slow Cooling through A1: this is done so that austenite always transforms at temp just below A1 to obtain equiaxed and relatively coarse grained ferrite as well as pearlite with large interlamellar spacing to induce softness and ductility.

The formation of austenite destroys all structures that have existed before heating. Slow cooling yields the original phases of ferrite and pearlite in hypoeuetectoid steels and that of cementite and pearlite in hypereutectoid steels

The slow cooling of full annealing causes austenite transformation to ferrite and pearlite close to A3 and A1 temperatures, respectively, and ensures that coarse-grained equiaxed ferrite and pearlite with coarse interlamellar spacing will form, producing microstructures of high ductility and moderate strength

Once the austenite has fully transformed to ferrite and pearlite, the cooling rate can be increased to reduce processing time and thereby improve productivity

PROCEDURE:

- Keep the Specimen inside the furnace and close the door.
- Switch on the furnace, set the temperature control knob to given temperature.
- The specimen kept in the furnace allowed heat between 35 min to 1 Hr.
- The specimen is taken out and any of the following steps are adopted as required
- The furnace is switched off and the specimen is cooled slowly inside the furnace.
- After the specimen reaches the room temp, measure the Rockwell hardness and record it in the observation table.

CONCLUSION:-

Coarse Pearlite (Annealing) x 700

1. White patches indicate formation of ferrite
2. Black patches indicate pearlite formation.

EXPERIMENT-2

Aim: - Normalising of plain carbon steel

Apparatus Required: -

- Hacksaw blade
- Plain Carbon Steel (Mild Steel) Sample
- Muffle Furnace
- File
- Emery Paper
- Double disc polisher
- Etchant (Nital) [98% C_2H_6OH + 2% HNO_3]
- Metallurgical Microscope

Theory: -

Normalizing is a technique used to provide uniformity in grain size and composition throughout an alloy.

The term is often used for ferrous alloys that have been heated above the upper critical temperature and then cooled in open air.

It is a process of heating steel to about 40-50°C above upper critical temperature (A3 or Acm), holding for proper time, then cooling in still air or slightly agitated air to room temperature.

Objectives of Normalizing:-

1. To refine the coarse grains of steel castings, forgings, etc. which have not been worked under high temperatures.
2. To improve the mechanical properties of plain carbon steels particularly forged shafts ,rolled stocks and castings for moderate load conditions.
3. To eliminate , or reduce microstructural irregularities.
4. To incresase machinability of low carbon steels.
5. To eliminate, or break coarse cementite network in hypereutectoid steels.
6. General refinement of structure prior to hardening of steel.

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. Steel is heated to austenitic temperature and then cooled in air. Purpose is

- To refine grain structure
- To increase strength of steel
- To reduce segregation in castings or forgings

In special cases cooling rate is controlled either by air temperature or by changing air volume.

Normalizing process consists of three steps.

The first step involves heating the steel component above the A3 cm temperature for hypoeutectoid steels and above A(upper critical temperature for cementite) temperature for hypereutectoid steels by 300C to 500C.

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.

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 Acm to point A1.

During normalising we use grain refinement which is associated with allotropic transformation upon heating $\gamma \rightarrow \alpha$.

Parts that require maximum toughness and those subjected to impact are often normalized.

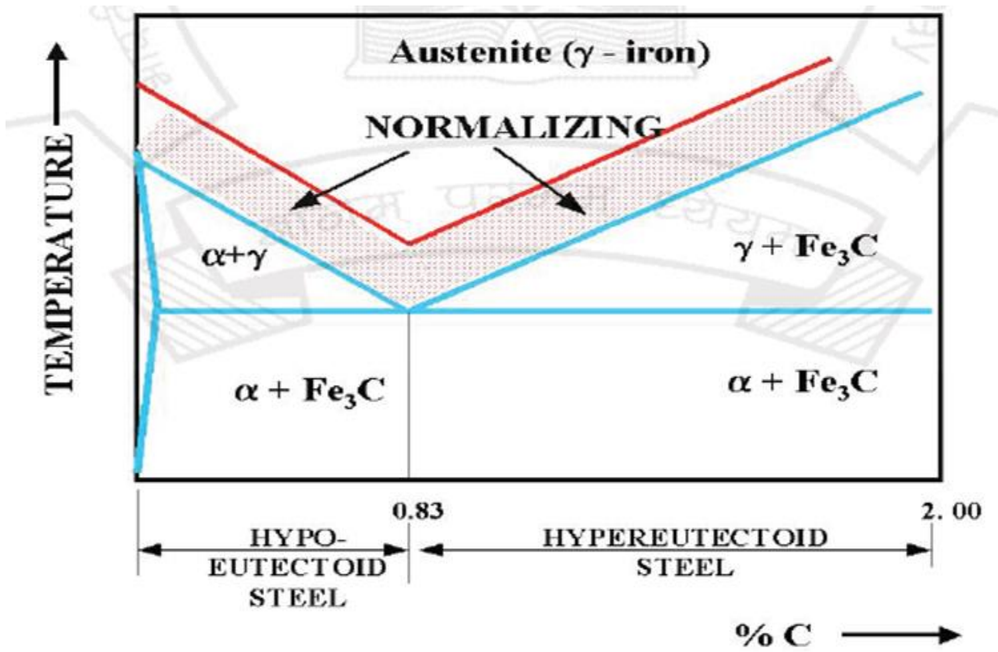
The microstructure obtained by normalizing depends on the composition of the castings (which dictates its hardenability) and the cooling rate.

By normalizing , an optimum combination of strength and softness is achieved , which results in satisfactory level of machinability in steels.

This method of improving machinability is specially applicable to hypoeutectoid steels.

Normalizing is the very effective process to eliminate the carbide network form during annealing of hypereutectoid steels.

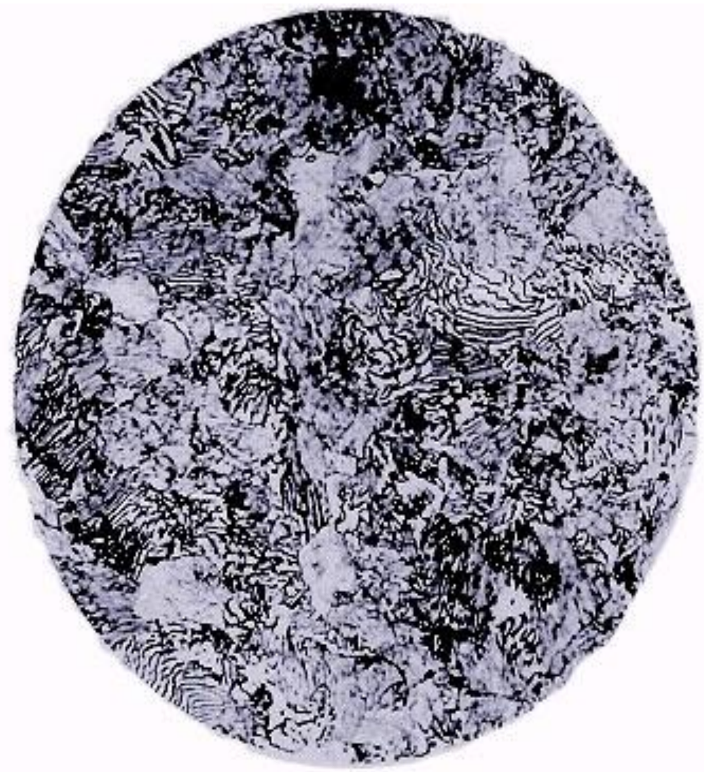
Due to shorter time available during cooling, this network does not appear in normalized structure.



PROCEDURE:

- ❖ Keep the Specimen inside the furnace and close the door.
- ❖ Switch on the furnace, set the temperature control knob to given temperature.
- ❖ The specimen kept in the furnace allowed heat between 35 min to 1 Hr.
- ❖ The specimen is taken out and any of the following steps are adopted as required
- ❖ The specimen is cooled against the air blower or fan.
- ❖ After the specimen reaches the room temp, clean with an abrasive paper.
- ❖ Measure the hardness and record it in the observation table.

Conclusion: -



Fine Pearlite (Normalizing) X 700

It is evident from figure that normalized microstructure is almost fully pearlite, as indicated by the black regions. The difference in microstructure is attributed to a higher rate of cooling in normalizing, as compared to annealing.

EXPERIMENT-3

Aim: - Hardening (Quenching) of plain carbon steel

Apparatus Required: -

- Hacksaw blade
- Plain Carbon Steel (Mild Steel) Sample
- Muffle Furnace
- File
- Emery Paper
- Double disc polisher
- Etchant (Nital) [98% C_2H_6OH + 2% HNO_3]
- Metallurgical Microscope

Theory: -

Quench hardening is a mechanical process in which steel and cast iron alloys are strengthened and hardened

The different stages of quenching are as follows:

STAGE 1: VAPOUR BLANKET STAGE.

Immediately on quenching, coolant gets vaporized as the steel part is at high temperature, and thus, a continuous vapour- blanket envelope the steel part.

Heat escapes from the hot surface very slowly by radiation and conduction through the blanket of water vapour. Since the vapour-film is a poor heat conductor, the cooling rate is relatively low (stage A in fig). This long stage is undesirable in most quenching operations.

STAGE 2: INTERMITTENT CONTACT STAGE.

Heat is removed in the form of heat of vaporization in this stage as is indicated by the steep slope of the cooling curve. During this stage, the vapour-blanket is broken intermittently allowing the coolant to come in contact with the hot surface at one instant, but soon being pushed away by violent boiling action of vapour bubble. The rapid cooling in this stage soon brings the metal surface below the boiling point of the coolant. The vaporization then stops. Second stage corresponds to temperature range of 500◦ to 100◦C , and this refers to nose of the CCT curve of the steel , when the steel transforms very rapidly (to non martensite product).

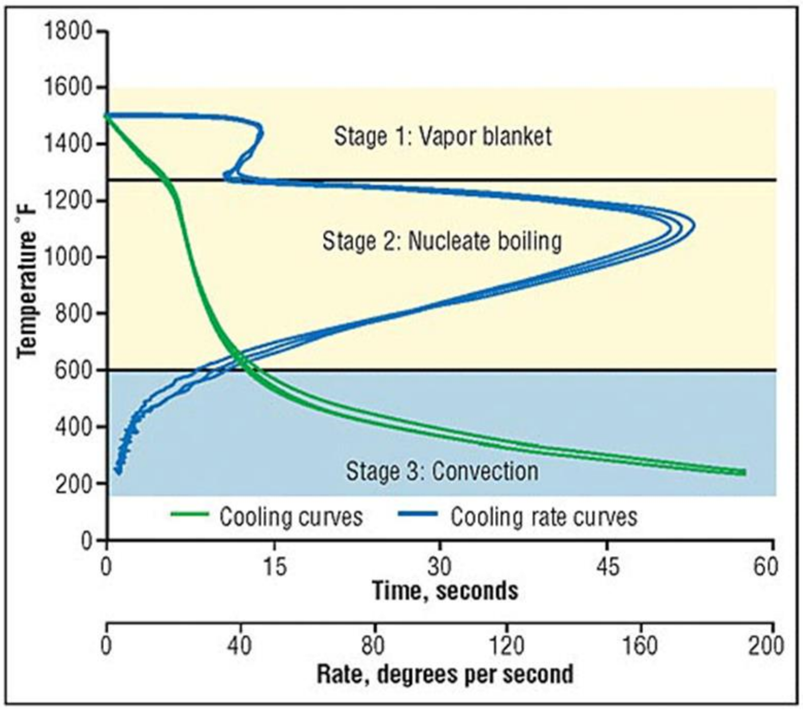
Thus, the rate of cooling in this stage is of great importance in hardening of steels

STAGE 3 : DIRECT CONTACT STAGE

This stage begins when the temperature of steel surface Is below the boiling point of coolant. Vapours do not form. The cooling is due to convection and conduction through the liquid. Cooling is slowest here.

- Some of the common quenching mediums are as follows:

1. -water
2. -brine
3. -oils
4. -polymer quenchants



In quenching of steels ,this may be caused by thermal stresses, structural stresses, or both, or even premature failure of part in service.

Cooling during quenching lakes place non-uniformly, i.e., causes temperature gradient across the section. Surface layers contract more than the central portion.

Contraction of surface is resisted by the central portion, and this puts the central portion under the compressive stresses, and the surface layers in tension . If the magnitude of stress becomes more than the yield stress of steel (at that deformation occurs.

These stresses that develop in a quenched part as a result of unequal cooling are called thermal stresses.Structural stresses are the stresses which develop due to due to phase change (mainly austenite to martensite), and at different times.

Structural stresses are developed due to two main reasons:

1. Austenite and its transformation products have unequal specific volume i.e. change in volume occurs when transformation occurs.

2. Phase changes occur at different times in the surface and in centre.

Under right conditions, both types of stresses get superimposed to become larger than the yield strength to cause warping. but when the tensile internal stresses become larger than the tensile strength cracks appear. If an austenitised steel is quenched, it contracts thermally till Ms temperature is attained .As surface cools faster than centre, i.e., contracts more than centre distribution of stresses across the section is illustrated in fig (b), i.e, the surface is under tensile nature of stress, while centre is under compressive stresses. Only thermal Stresses are produced in stage 2 , surface having attained Ms temperature, transforms to martensite, and thus expands, while the centre is still contracting as it is getting cooled.

In stage Il, centre may get plastically deformed ,as it is still ductile austenite.

In stage 3, martensite of surface and austenite of centre continue contracting leading to slight increase in stress levels.

PROCEDURE:

- Keep the Specimen inside the furnace and close the door.
- Switch on the furnace, set the temperature control knob to given temperature.
- The specimen kept in the furnace allowed heat between 35 min to 1 Hr.
- The specimen is taken out and any of the following steps are adopted as required
- The specimen is allowed to cool suddenly by quenching in cold water or oil bath or salt bath.
- Dry the specimen, with the help of a piece of cloth and an abrasive paper is used to remove the fins or scales adhere to the surface.
- Measure the hardness and record it in the observation table.

Conclusion: -

Martensite (Quenching) X 700

Microstructure is seen to consist of huge amount of fine, needle like structures. This structure is called martensite, which is hard, but brittle too. In quenching, the steel specimen experienced a very rapid rate of cooling. As such, the austenitic structure transformed into martensite.

EXPERIMENT-4

Aim: - Tempering of plain carbon steel

Apparatus Required: -

- Hacksaw blade
- Plain Carbon Steel (Mild Steel) Sample
- Muffle Furnace
- File
- Emery Paper
- Double disc polisher
- Etchant (Nital) [98% C_2H_6OH + 2% HNO_3]
- Metallurgical Microscope

Theory: -

Hardening of metal produces Martensite structure with some retained austenite. The martensite structure makes the metal very hard and brittle. The retained austenite is unstable and it will change with time. This transformation of retained austenite even at room temperature leads to distortion of metal. Due to these factors the hardened metal cannot be used as it is. Hence tempering is carried out on the metals.

Tempering treatment involves:

Heating the metal just above Martensite structure temperature (50 O C),

Holding it at that temperature for some time and then cooling either rapidly or slowly. The purpose of tempering is to remove brittleness and improve ductility in the material.

The Properties obtained after Tempering are:

- Improvement in ductility and toughness.
- Slight reduction in hardness.
- Increase in tensile strength.
- Reduction in internal stress.

STAGES OF TEMPERING

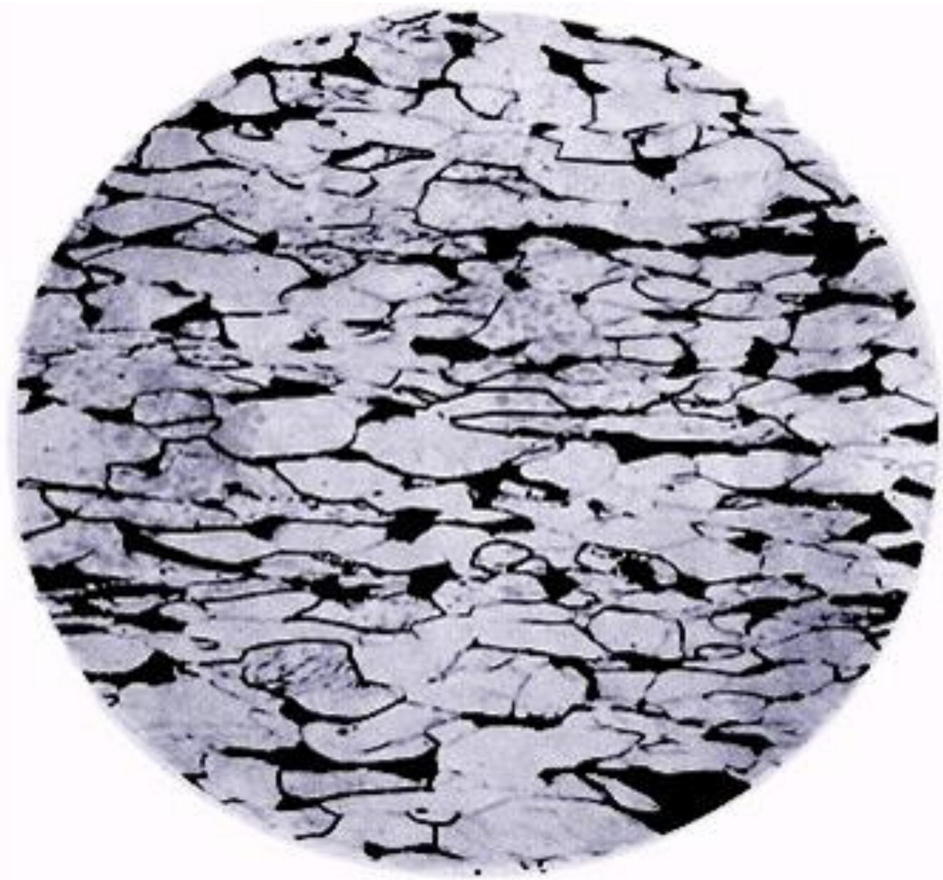
On reheating as-quenched martensite, the tempering takes place in four distinct but overlapping stages:

- Stage 1, up to 250°C — precipitation of E-iron carbide; partial loss of tetragonality in martensite.
- Stage 2, between 200 and 300°C — decomposition of retained austenite .
- Stage 3, between 200 and 350°C — replacement of &iron carbide by cementite; martensite loses tetragonality.
- Stage 4, above 350°C — cementite coarsens and spheroidizes; recrystallization of ferrite.

PROCEDURE:

- Keep the Specimen inside the furnace and close the door.
- Switch on the furnace, set the temperature control knob to given temperature.
- The specimen kept in the furnace allowed heat between 35 min to 1 Hr.
- The specimen is taken out and any of the following steps are adopted as required
- The specimen is allowed to cool suddenly by quenching in cold water or oil bath or salt bath.
- After the normalization, the specimen is subjected for reheating between 150 to
- 4000C.
- Now remove it from the furnace and quench it in the quenching bath.
- Clean it, measure its hardness and record it in the observation table

Conclusion: -



Carbide/Ferrite (Tempering) X 700

When martensite is reheated during tempering, it transforms into sorbite or troostite resulting in reduced hardness levels and increased ductility. It is seen that tempering leads to a decrease in hardness. This is actually desirable, as low hardness and good toughness will be beneficial for machining purposes, as cutting forces and specific energy required will be less.The microstructure consists of finer grains and hardness decreases, but not by much, which ensures good machinability.

EXPERIMENT-5

AIM: To determine the hardenability of a given steel by Jominy End Quench method

APPARATUS

- ✓ Jominy test apparatus
- ✓ furnace,
- ✓ Rockwell hardness tester
- ✓ grinder.

THEORY:

Jominy end quench test is used to determine harden ability of steels .The process of increasing the hardness of steel is known as Hardening .Specific specimenwith standard dimensions, used for the test is given in fig.7.1.The hardness of hardened bar is measured along its length.

Harden ability: The depth up to which steel can be hardened is defined as harden ability. A steel having high hardness need not have high harden ability. Harden ability may be defined as susceptibility to hardening by quenching. A material that has high harden ability is said to be hardened more uniformly throughout the section than one that has lower harden ability.

M.A Gross man devised a method to decide harden ability.

Critical diameter: The size of the bar in which the zone of 50% martensite occurs at center is taken as critical diameter. This is a measure of harden ability of steel for a particular quenching medium employed.

Severity of Quench:

The severity of quench is indicated by heat transfer equivalent

H=f/k

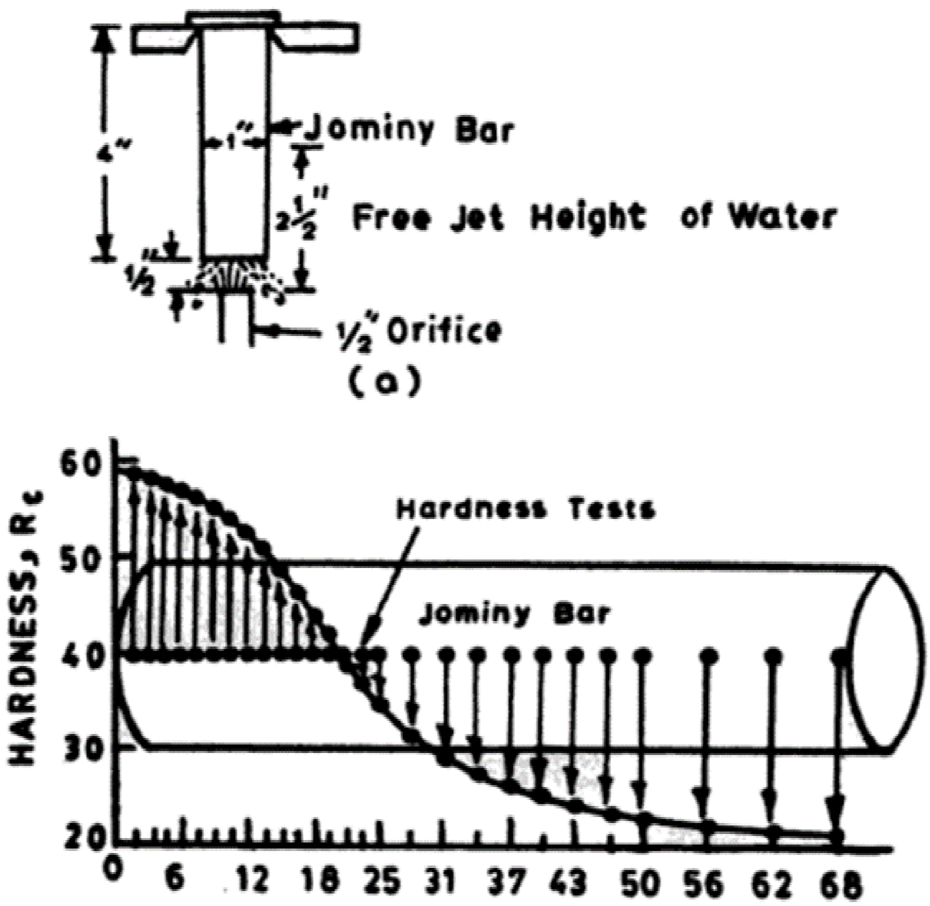
f = Heat transfer factor of quenching medium and the turbulence of the bath. K=Thermal conductivity of bar material.

The most rapid cooling is possible with severity of as infinity

Ideal Critical Diameter:

The harden ability of steel can be expressed as the diameter of bar that will form structure composed of 50% marten site at the center when quenched with H=infinity. This diameter is defined as critical diameter.

Description of Apparatus:



The apparatus consists of a cylindrical drum. At the top of the drum provision is made for fixing the test specimen. A pipe line is connected for water flow, which can be controlled by means of a stop cock.

PROCEDURE:

Out of the given steel bar, the standard sample is to be prepared as per the dimensions shown in the above figure.

a. The austenitising temperature and time for the given steel is to be determined depending on its chemical composition.

b. The furnace is setup on the required temperature and sample is kept in the furnace.

c.The sample is to be kept in the furnace for a predetermined time (based on chemical composition of steel) then it is taken out of the furnace and is kept fixed in the test apparatus.

d. The water flow is directed onto the bottom end of the sample. The water flow is adjusted such that it obtains shape of umbrella over bottom of sample.

e. The quenching is to be continued for approximately 15 minutes.

f. A flat near about 0.4 mm deep is grounded on the specimen.

g. The hardness of the sample can be determined at various points starting from the quenched end and the results are tabulated.

h. The graph is plotted with hardness versus distance from quenched end. From the results and graph plotted the depth of hardening of the given steel sample can be determined.

The harden ability of the specimen is foundry by observing the structure under themicroscope. As detailed above the diameter at which the percentage of martensite is 50 indicates harden ability of material. More this diameter high will be the harden ability. Now the important factor is the relationship between size are diameter of a steel bar quenched in an ideal quenching medium which has the same cooling rate at it centre as a given position along the fact that if position on the jominy bar where the structures is 50% martensite is known then the shown fig 8.4 makes it possible to determined ideal critical diameter

EXPERIMENT-6

Aim: -To study the carburising of mild steel

Apparatus Required: -

- Plain Carbon Steel (Mild Steel) Sample
- Muffle Furnace
- File
- Emery Paper
- Double disc polisher
- Etchant
- Metallurgical Microscope
- Barium Carbonate ($BaCO_3$) (5%)
- Charcoal (85%)
- Box
- Power Hacksaw
- Rockwell hardness testing machine

Theory: -

Carburization is a technique used to harden the surface of steels by diffusing into the crystal lattice. The carbon enters the interstitial spaces between the iron atoms. It strengthens the metal by distorting the crystal lattice, thus making it difficult for dislocations to move. Carburization is a surface technique because, even at high temperatures, diffusion is a slow process. The source of carbon can either be a gas or solid carbon.

The practical aims to familiarize you with a solution of Fick's Second Law of diffusion by studying the diffusion of carbon into iron.

You will gain experience of heat treatment in furnaces, preparation of metallographic sections and etching.The microscopy will require interpretation of more complicated microstructures.

Procedure: -

You are provided with two small pieces of Mild steel; this has a low C content.
One will be as blank (for comparison) and the other is required for carburizing at
950 ^{o}C

• Heat the samples in the oven at 950 ^{0}C for one hour. At the end turn off the oven, and let the samples cool in air to room temperature. This process is caused normalization, and can be done several days or weeks before the experiment; however, the sample should be at room temperature for at least 24 hours before proceeding to the next step.

To carburize your sample:

- Clean the surface of the specimen by polishing up to 9□ m.

- The samples is packed in a ceramic crucible in a mixture of powdered charcoal and sodium carbonate activator (10 % w/w). The carbonate releases carbon dioxide that reacts with C to give carbon monoxide and form a carburizing gas. A lid is needed to exclude air.

- Place your crucible in the furnace for 2 hours at 1000°C.

- Remove from the oven and quench the sample (hot) in water.

- Perform a Vickers hardness test, and examine the surface under the optical microscopy.

- Compare the data before and after carburizing and describe the microstructure.

Note: after carburizing the sample need to be polished; in particular it is important that the rusted (oxide) surface is removed to expose a bulk cross-section. After grinding and polishing, the specimens need to be etched, in 2% Nital. Check that the etch time is sufficient to show the structure clearly across the whole carbon profile.