



**Department of Mechanical Engineering  
RV College of Engineering®, Bengaluru-59**

**Materials Science for Engineers – ME232TB  
Unit IV**

**Prepared by**

**Dr. RATNA PAL** ( BE, ME(IISc.), PhD)  
Assistant Professor  
**Email:** ratnapal@rvce.edu.in

**Dr. ROOPA T.S.** ( BE, M.Tech, PhD)  
Assistant Professor  
**Email:** roopats@rvce.edu.in

Department of Mechanical Engineering,  
RV College of Engineering  
Bengaluru-560 059

## Heat Treatment

----7 Hours

**Post processing heat treatment of electronic devices: thermal oxidation, diffusion, rapid thermal processing.**  
**Heat treatment of ferrous materials: annealing, spheroidizing, normalizing, hardening, tempering.**  
**formation of austenite, construction of Time Temperature Transformation (TTT) curves.** Special heat treatment processes: carburizing, nitriding, cyaniding, flame, and induction hardening. Defects in heat treatment.



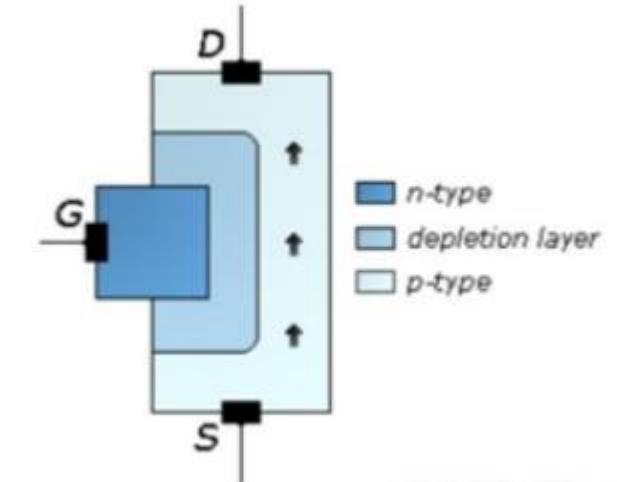
# Heat Treatment of Electronic Devices

Post-processing treatments for electronic devices (mainly semiconductor wafers) are carried out for the following **purposes**

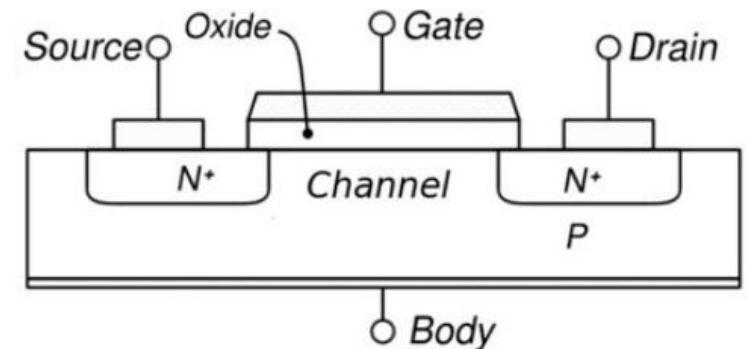
- Removing support structures
- Secondary curing
- Coating surfaces
- Polishing
- Improving surface roughness
- Changing material properties
- Changing structure shapes

Post-processing techniques can help address challenges like surface roughness, dimensional accuracy, mechanical properties, and surface finish.

Post processing techniques include **Annealing, baking, solder reflow, stress relieving, thermal oxidation** etc.



FET



MOSFET

**Annealing** is a thermal process used in semiconductor device fabrication to improve the material's electrical and structural properties:

**Reduces crystalline defects** - Heat pulses, usually around 800°C, restore the original crystalline structure.

**Removes impurities** - Impurities migrate to specific locations within the semiconductor, reducing their impact on the device.

**Repairs atomic level disorder** - Annealing can repair damage caused by ion implantation, where the crystal substrate is bombarded with high energy ions.

**Activates dopants** - Dopant atoms, such as boron, phosphorus, or arsenic, move into substitutional positions in the crystal lattice

There are several annealing methods, including:

**Furnace annealing**: Multiple semiconductor wafers , several hours to a day.

**Rapid thermal annealing (RTA)**: This method is increasingly replacing furnace annealing.

**Laser annealing**: Laser annealing is a cost-effective solution with superior performance compared to non-laser technologies.

**The annealing process depends on precise temperature control, time duration, and the use of specific atmospheres**

**Baking** is a process that removes moisture from semiconductor devices to prevent delamination and other issues:

**Purpose** - Baking evaporates moisture from the device, which can help prevent delamination, a condition where moisture escapes through copper holes and causes the board to separate.

**Process** - The device is baked in an oven at a temperature between 100°C and 130°C. The baking time and temperature depends on the device's type, size, and process requirements.

**Considerations** - The baking environment should be clean, dustproof, moisture-proof, and anti-static. The device should also be ventilated during baking.

**Risks** - Overheating or baking for too long can damage the device. If the baking temperature is too low, it can also affect the device's performance.

**Benefits** - Once baked, the device is less likely to have moisture sensitivity issues.

The semiconductor device is baked at a temperature within a range of 165° C. to 175° C. for 6 hours or more by using a hot plate. Thus, this baking under the reduced pressure prevents generation of blisters and cracks on the plastic package.

**Solder Reflow Process** Overview – it is a key technique in electronics assembly, where solder paste is used to attach electronic components to a printed circuit board (PCB), ensuring precise, high density and reliable connections between components and a PCB. Process steps are discussed below :

**Solder Paste Application** : Solder paste, a mix of solder powder and flux, is applied using stencil to the PCB's pads where components will be placed.

**Component Placement** : Electronic components are then carefully placed onto the PCB, aligning their leads or pads with the solder paste.

**Preheat Stage** : The PCB is gradually heated in a preheat zone of the reflow oven to bring it up to a temperature that helps to activate the flux in the solder paste, which helps to clean the surfaces and prepare them for soldering.

**Reflow Stage** : The PCB moves through the reflow oven's reflow zone, subjected to a carefully controlled temperature profile. This profile heats the solder paste to its melting point, allowing it to flow and create strong connections between the component leads and PCB pads. **Cooling**: After the reflow stage, the PCB moves into a cooling zone where it is gradually cooled down. This cooling process allows the solder to solidify, forming durable and reliable solder joints.

**Inspection and Testing** : Once cooled, the assembled PCB is inspected for soldering quality, checking for issues such as solder bridges or insufficient solder.

**Stress relief** treatments are used to reduce or remove residual stresses in materials caused by manufacturing processes. These treatments are often performed on ferrous and non-ferrous alloys, and are intended to prevent distortion and service problems.

Here are some ways to reduce stress in semiconductor devices:

**Use an asymmetrical structure** - An asymmetrical structure, like a three-phase current-source rectifier (CSR), can reduce stress by lowering the voltage and current load on the switches.

**Stress relieve annealing** - This is a special annealing process that reduces residual stresses without significantly changing the material's basic properties.

Stress can affect a device's performance, dependability, and efficiency. It can be caused by heat, high voltage, high current, or change frequency.

**High temperature electronic devices** are designed to operate in environments with temperatures beyond the standard range for silicon-based devices, which is typically below 125°C. The term "high temperature" often refers to operating temperatures in the range of 200–400°C.

High temperature electronics are used in a variety of applications, including:

- Geothermal
- Offshore safety control systems
- Pressure control sensors
- Motor controllers
- Engine ground-test instrumentation
- Control and condition monitoring systems in advanced supersonic aircraft

Here are some examples of high temperature electronic devices:

## **Ozark IC's XNode modules**

It operates in temperatures up to 800°C and are used in applications such as space exploration, jet engines, and geothermal exploration.

## **Honeywell's high temperature electronic components**

These components are designed to operate continuously at 225°C for at least five years and are used in applications such as data acquisition, sensor signal conditioning, and control.

## **SiC JFET-integrated circuits**

These circuits are the most advanced and mature high-temperature logic technology and can operate stably at elevated temperatures for up to a year.

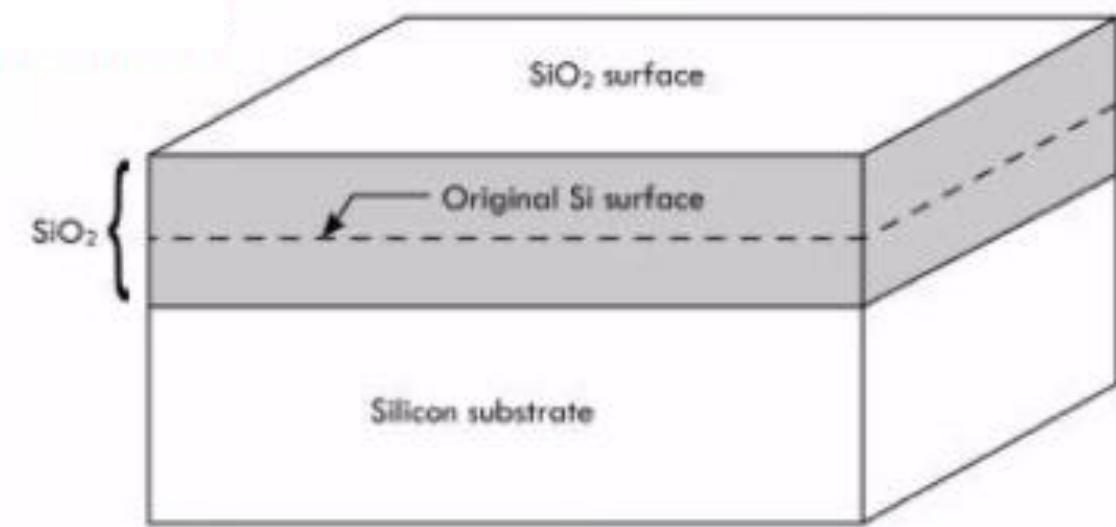
**Thermal oxidation** is a process used to grow a thin layer of silicon dioxide (**SiO<sub>2</sub>**) on the surface of silicon wafers by exposing them to an oxidizing atmosphere at elevated temperatures. This process is done using oxidation furnaces at elevated temperatures between 600°C to 1250°C to form silicon dioxide (SiO<sub>2</sub>) layer.

## Importance of Oxidation

It serves as an insulator on the wafer surface. Its high relative dielectric constant enables metal line to pass over the active silicon region.

SiO<sub>2</sub> acts as the active gate electrode in MOS device structure.

It is used to isolate one device from another. It provides electrical isolation of multilevel metallization used in VLSI.



## Importance of SiO<sub>2</sub> layer

It acts as a diffusion mask permitting selective diffusions into Si wafer through the window etched into oxide.

It is used for surface passivation which creates protective SiO<sub>2</sub> layer on the wafer surface. It protects the junction from moisture and other atmospheric contaminants.

## Steps in Thermal Oxidation Process

**Preparation of silicon wafers :** cleaned and free from any contamination or oxide layers

**Loading the furnace :** cleaned silicon wafers are loaded into a furnace chamber. Furnace chamber is sealed with controlled environment

**Purging and Heating :** The furnace chamber is purged with an inert gas (i.e N<sub>2</sub>) to remove any remaining traces of oxygen. Then the temperature of the furnace is raised to the desired level.

**Oxidation :** After reaching the desired temperature, an oxidizing atmosphere is created into the chamber by using oxidizing agent (such as dry oxygen, steam etc.). Silicon at the wafer's surface reacts with the oxygen to form silicon di oxide (SiO<sub>2</sub>). Slow and controlled process to grow an uniform layer of oxide on the silicon wafer. Duration of oxidation process controls the thickness of the oxide layer. Growth of oxide layer depend on the temperature, concentration of oxidizing agent and the ambient pressure.

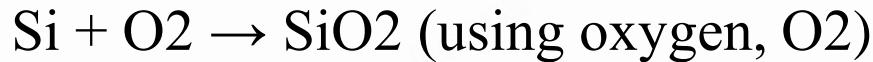
**Cooling and cleaning :** After achieving the desired thickness of oxide layer, the chamber is slowly cooled, oxidizing agents are removed and silicon wafer are unloaded and cleaned to remove any residual oxide and contaminant.

There are 2 methods of oxidation - **Dry and Wet oxidation**. In both cases Si is consumed from the surface of the substrate shown in figure.

**Factors of thermal Oxidation :** Temperature, time, oxidizing agent concentration and ambient pressure

## Characteristics of Oxidation

### Dry Oxidation



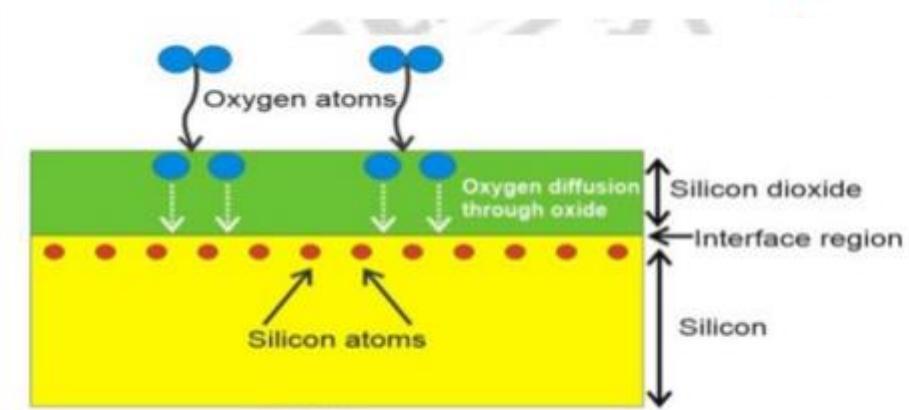
Uses pure oxygen to create a silicon dioxide layer

- Slow growth rate
- High density
- High breakdown voltage
- Produces thinner oxide (excellent electrical properties)
- Growth speed is slow

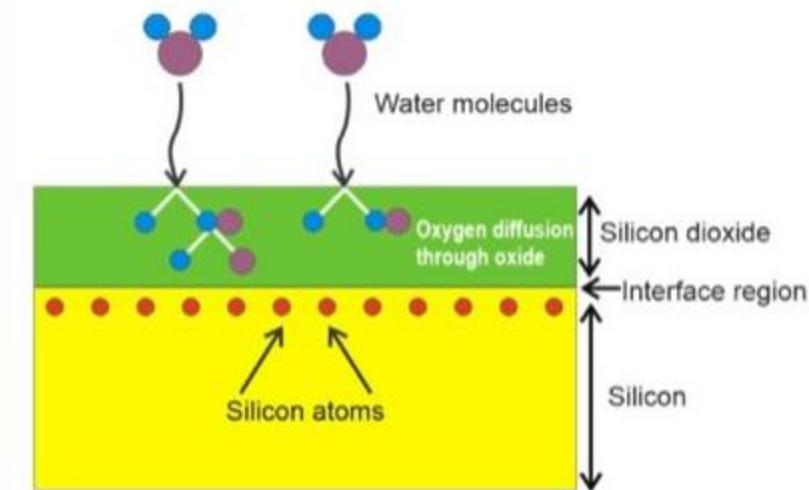
### Wet Oxidation

$\text{Si} + 2\text{H}_2\text{O} \rightarrow \text{SiO}_2 + 2\text{H}_2$  (using steam,  $\text{H}_2\text{O}$ ). Involves steam or water vapour

- Fast growth even on low temperature
- Low quality compared to dry oxidation
- Produces thicker oxide
- Low density
- Growth speed is fast



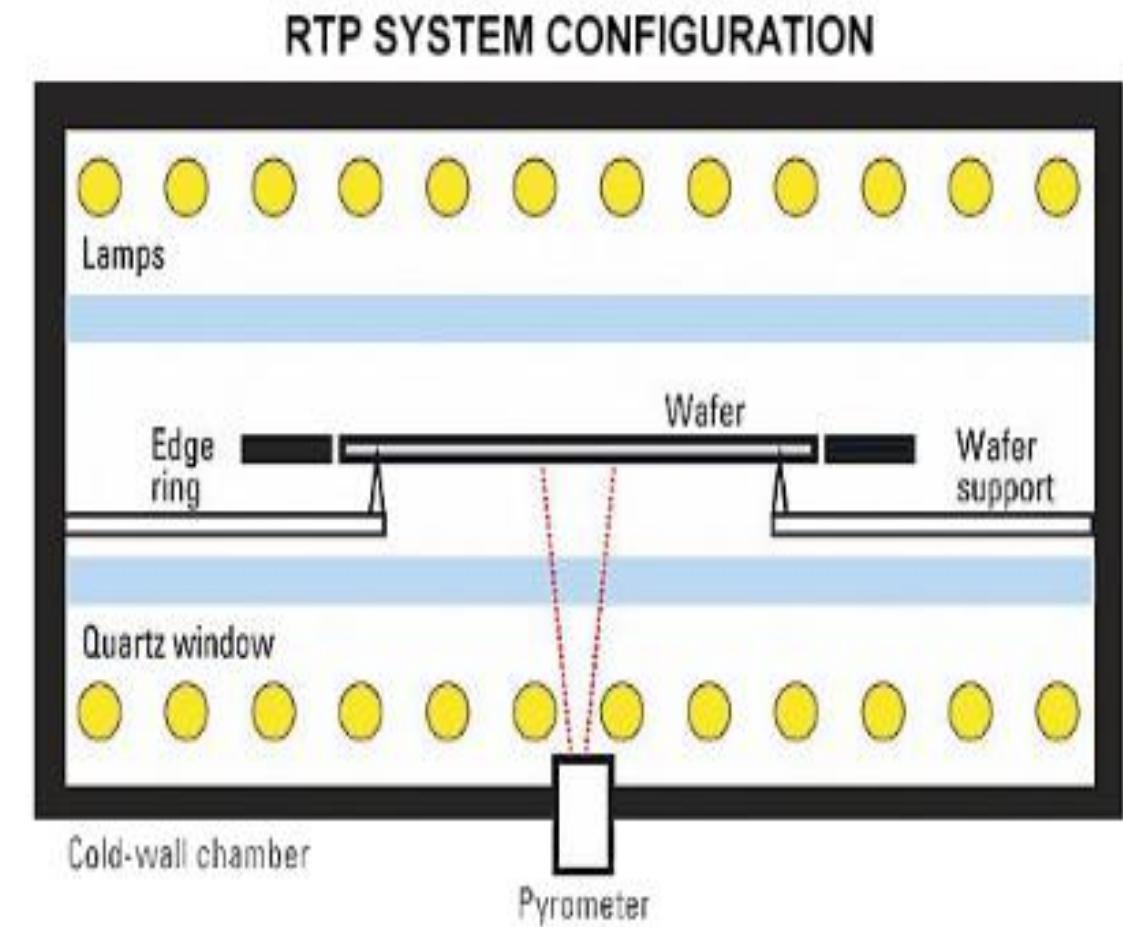
$\text{SiO}_2$  layer by Dry Oxidation



$\text{SiO}_2$  layer by wet Oxidation

**Rapid thermal processing (RTP)** is a semiconductor manufacturing process that involves heating silicon wafers to high temperatures for a short time, then cooling them slowly. The process is carried out in a **RTP chamber**

RTP is a key technology in semiconductor manufacturing because it **allows for the growth of very thin layers with extreme precision with very short period of time**. It also allows for the effective activation of implanted impurities with minimal diffusion.



RTP chamber

## RTP Chamber

- The RTP chamber is a thermally isolated process chamber. Cooling water lines are installed inside the chamber along with gas inlets. Ambient gases are used in the process . Lamps are installed on top, bottom and sides of the chamber and also cooled by air or nitrogen based cooling.
- Inside the process chamber and close to the lamps, several quartz plates or tubes (Quartzware) are installed to effectively isolate wafer from process chamber. Quartzware is cooled by CDA (Clean Dry Air cooling) or PN2 (Pure Nitrogen based cooling).
- Silicon wafer are placed on three to four quartz pins surrounded by Si edge guard ring to eliminate edge to centre temperature variation.
- Pyrometer assembly is mounted at the bottom of the chamber to control the process temperature and looks for the emitted radiation from the backside of the wafer.
- This process aims to restore or enhance the performance and functionality of semiconductor devices as well as their properties or structure. Rapid heating is achieved using high-intensity lamps, such as tungsten halogen lamps or lasers.
- Temperatures can range from 200 to over 1300°C, with ramp rates typically ranging from 20 to 200°C/sec.

## Steps of RTP Process

**Heating** - Silicon wafers are heated to temperatures of over 1,000°C (1832°F) for a few seconds or less using high-intensity lamps or lasers.

**Cooling** - The wafers are cooled gradually to prevent thermal shock and breakage.

**Monitoring** - Temperature control and measurement are critical to ensure the wafer reaches the correct temperature and doesn't overheat. Thermocouples and pyrometers are often used to measure and control the temperature.

**Environmental control** - A nitrogen curtain can be used to minimize unwanted oxygen. An oxygen analyzer can be used to monitor and control the oxygen concentration

## Advantages:

- Uniformity: Single wafer processing produces the best uniformity, especially for large wafers.
- Low thermal budget
- Short cycle time: decreases the amount of work in progress.
- Process flexibility: can run at atmospheric or reduced pressure, and can utilize a wide range of process gases.
- Ultra-clean process: RTP can be performed on a wafer-by-wafer basis within a cluster tool, making it an ultra-clean process

## Disadvantages :

- Temperature measurement and control is challenging, including the ramp-up, soak, and ramp-down steps.
- Absolute temperatures are almost never known .
- Nonthermal-equilibrium conditions make modeling and predicting difficult.
- Oxygen hazards: Oxygen can be used as a process gas in RTP systems, and it can create an explosive condition.

**Applications** - Dopant activation, thermal oxidation, metal reflow, chemical vapour deposition, silicide formation, and oxide growth.

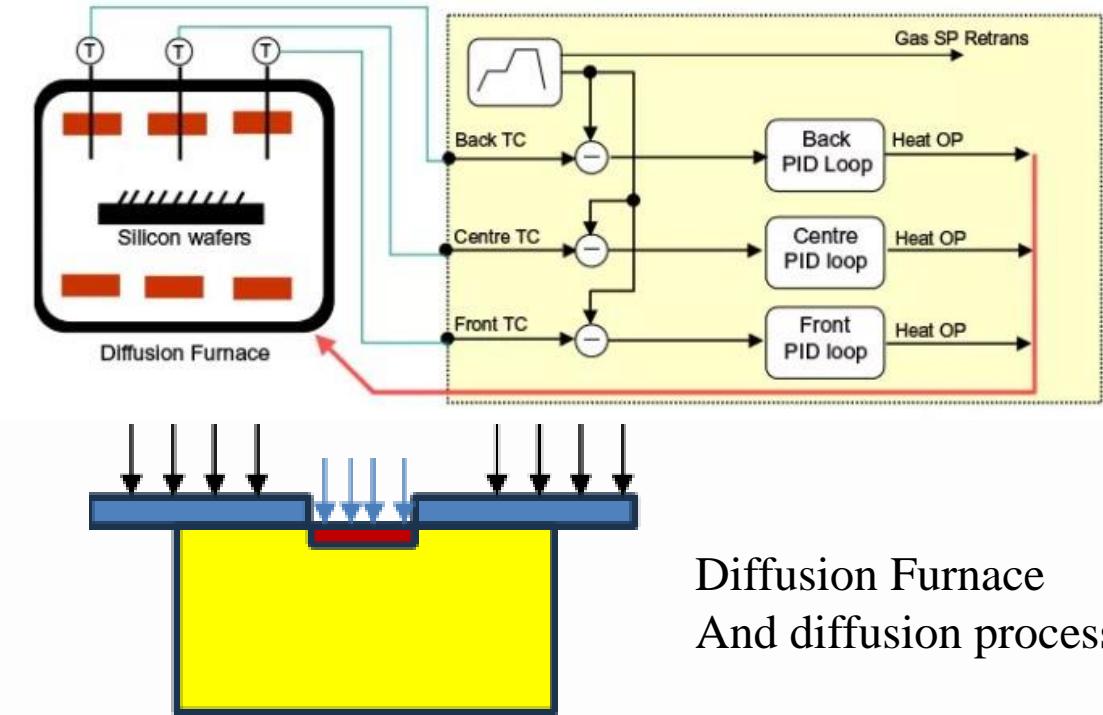
**Diffusion** is the movement of impurity atoms in a semiconductor material at high temperatures. **The driving force of diffusion is the concentration gradient.** Diffusion is applied to anneal the crystal defects after ion implantation or to introduce dopant atoms into silicon from a chemical vapour source. Diffusion time and temperature determine the depth of dopant penetration. Diffusion is used to form the source, drain, and channel regions in a MOS transistor.

**Diffusion process** is carried out in diffusion furnaces.

They are tube furnaces used in the manufacturing process of semiconductor components. They are used to add doping impurities into high purity silicon wafers, thereby creating embedded semiconductor devices.

This process occurs at high temperatures and demands a high degree of measurement accuracy and control stability.

Most diffusion furnaces have 3 heating zones along with some gas flow control loops. Additionally some processes are conducted under vacuum.



Diffusion Furnace  
And diffusion process

## Steps in Diffusion process

**Dopant Selection** : Appropriate dopant material is selected depending on the desired electrical properties of the semiconductor. Common dopants are (B, P, As, Sb)

**Wafer Preparation** : Silicon wafer are the most semiconductor substrate are cleaned to removes native oxide and contaminants from wafer surface.

**Dopant Source** : Generally dopant materials are available in the solid form. The source is placed near the silicon wafers.

**Heating** : The wafer and the dopant source are heated together at high temperature in a controlled environment.

**Dopant Diffusion** : At high temperature under controlled environment, the dopant atoms from the source diffuses into silicon lattice in silicon wafers. Dopant concentration profile formed within the silicon wafer.

**Annealing** : After diffusion process annealing is performed to repair crystal damage and activate the dopants to achieve desired electrical properties.

**Etching and Clean-Up** : Any excess or unwanted dopant material is removed form the wafer's surface through chemical etching or other cleaning process.

**Parameters : Temperature, Time, Type of dopant and the environment**

## Advantages :

- High-quality junctions with minimal leakage current, and without damaging the crystal.
- Strong and durable bonds making the devices more reliable and durable.
- Protective layers - protects the wafer and insulates the circuits.
- Enhanced structural integrity of semiconductor materials.
- Precise control for ensuring high device performance and yield.

## Disadvantages :

- Diffusion in thermoelectric materials - When placed in a thermal gradient, thermoelectric materials can diffuse, resulting in inferior and non-homogeneous materials.
- Diffusion in micro-thermoelectric devices - Diffusion at the interfaces between the metallic electrodes, solder materials, and thermoelectric thin films can degrade the device's performance and reliability.
- Complex oxidation processes - When the interface between the substrate and oxide is not flat, it can be difficult to simulate experimental results.

# Heat Treatment of Ferrous Materials

**Heat Treatment** – it is a thermal process involving the operation of heating the material (usually above their critical temperature) and cooling under controlled condition to manipulate the microstructure and obtain desired mechanical properties.

Involves basically three basic operations

1. Heating the material above the critical temperature
2. Holding the material at that temperature to obtain uniform structure throughout (also termed as soaking)
3. Cooling the material under controlled condition to obtain the desired microstructural change and hence the mechanical properties.

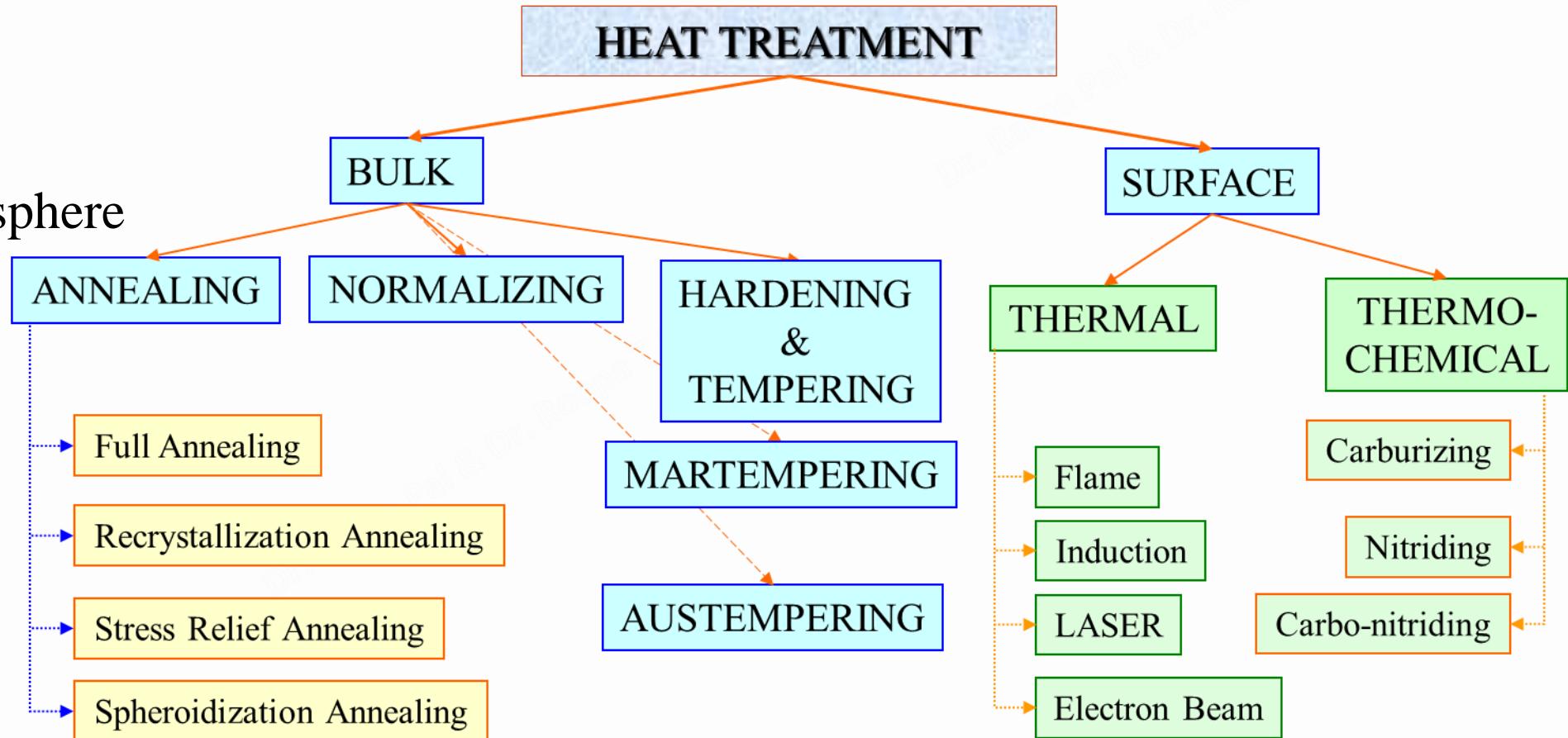
**Heating → Soaking → Cooling**

## Objectives of Heat Treatment

- To increase strength, hardness and wear resistance (*bulk hardening, surface hardening*)
- To increase ductility and softness (*Tempering, Recrystallization Annealing*)
- To increase toughness (*Tempering, Recrystallization annealing*)
- To obtain fine grain size (*Recrystallization annealing, Full annealing, Normalizing*)
- To remove internal stresses induced by differential deformation by cold working, non-uniform cooling from high temperature during casting and welding (*Stress relief annealing*)
- To improve machinability (*Full annealing and Normalizing*)
- To improve cutting properties of tool steels (*Hardening and Tempering*)
- To improve surface properties (*surface hardening, high temperature resistance-resistance precipitation hardening, surface treatment*)
- To improve electrical properties (*Recrystallization, Tempering, Age hardening*)
- To improve magnetic properties (*Hardening, Phase transformation*)

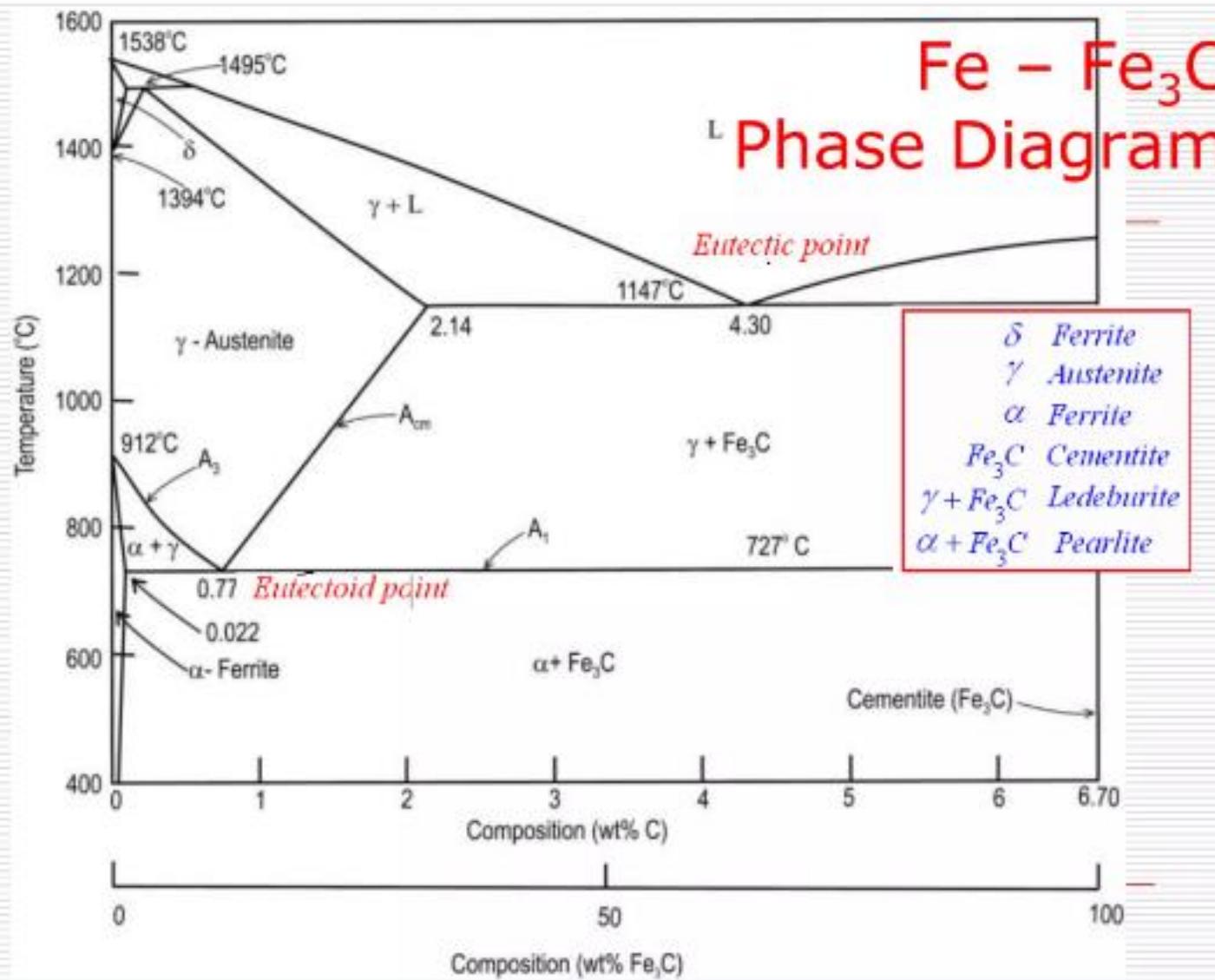
## Heat Treatment process variables

1. Temperature
2. Holding Time
3. Heating Rate
4. Cooling Rate
5. Furnace Atmosphere



# Phase Diagram of Fe-C

Go, change the world



## Types of ferrous materials

Steels – up to 2% carbon

Cast iron – 2-4.3 % carbon

Eutectoid steel – 0.8 % Carbon

Hypo-eutectoid steel < 0.8% C

Hyper-eutectoid steel > 0.8% C

## Important Invariant Reactions

Eutectic : at 1147°C with 4.3%C

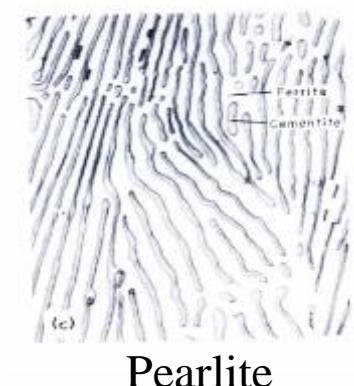
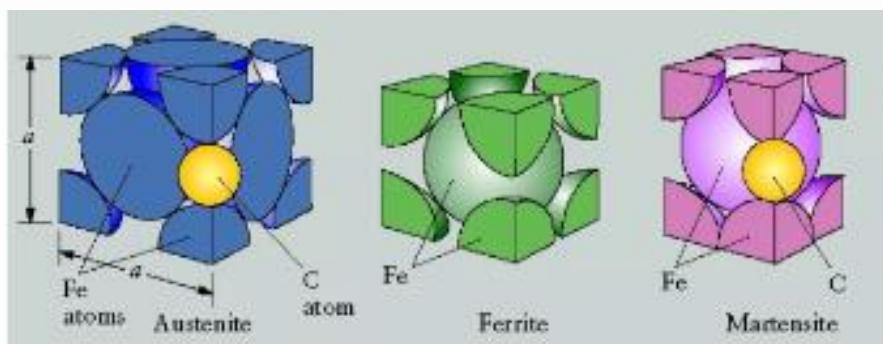
Liquid  $\longleftrightarrow$  Austenite + Cementite

Eutectoid : at 727°C with 0.8%C

Austenite  $\longleftrightarrow$  Pearlite (mixture of ferrite and cementite)

## Important phases of the Iron-Carbon phase diagram

- **Ferrite** – Interstitial solid solution of carbon in  $\alpha$  iron (BCC). Max solubility 0.02 % at  $723^{\circ}\text{C}$  and 0.008% at room temperature. Very soft and ductile.
- **Austenite** - solid solution of carbon in  $\gamma$  iron (BCC). Max solubility 2.08 % at  $1148^{\circ}\text{C}$  and 0.8% at  $723^{\circ}\text{C}$ . High formability, useful for heat treatment.
- **Cementite** – Intermetallic compound of Fe and 6.67% C, Orthorhombic crystal structure, hard and brittle.
- **Pearlite** - Eutectoid Mixture of 0.8 % C and forms at  $723^{\circ}\text{C}$  on very slow cooling rate. It is very fine platelike or lamellar structure of ferrite and cementite
- **Martensite** – A supersaturated solid solution of carbon in ferrite. It is a non-equilibrium phase forms when steel is cooled very rapidly. The interstitial carbon atoms distort the BCC ferrite into a body centered tetragonal (BCT) structure. **Very hard and brittle.**

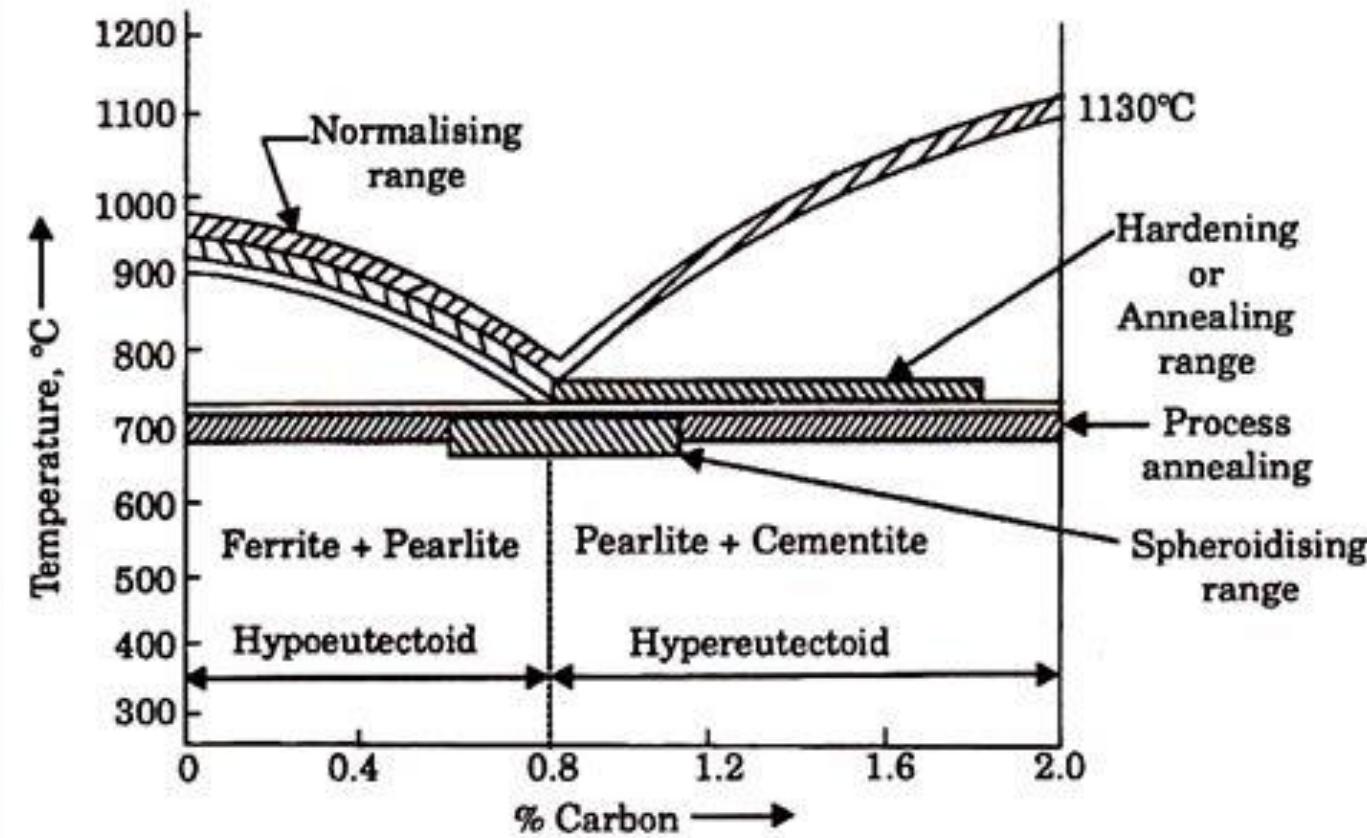


## Temperature range of Heat Treatment

**A1** – Lower critical temperature - below which there is no austenite present in the steel under equilibrium conditions. It is eutectoid temperature ( $727^{\circ}\text{C}$ )

**A3** - Upper critical Temperature - above which hypoeutectoid steels (< 0.8% C) are completely transformed to austenite.

**A<sub>cm</sub>** - Upper critical Temperature - above which hypereutectoid steels (> 0.8% C) are completely transformed to austenite.

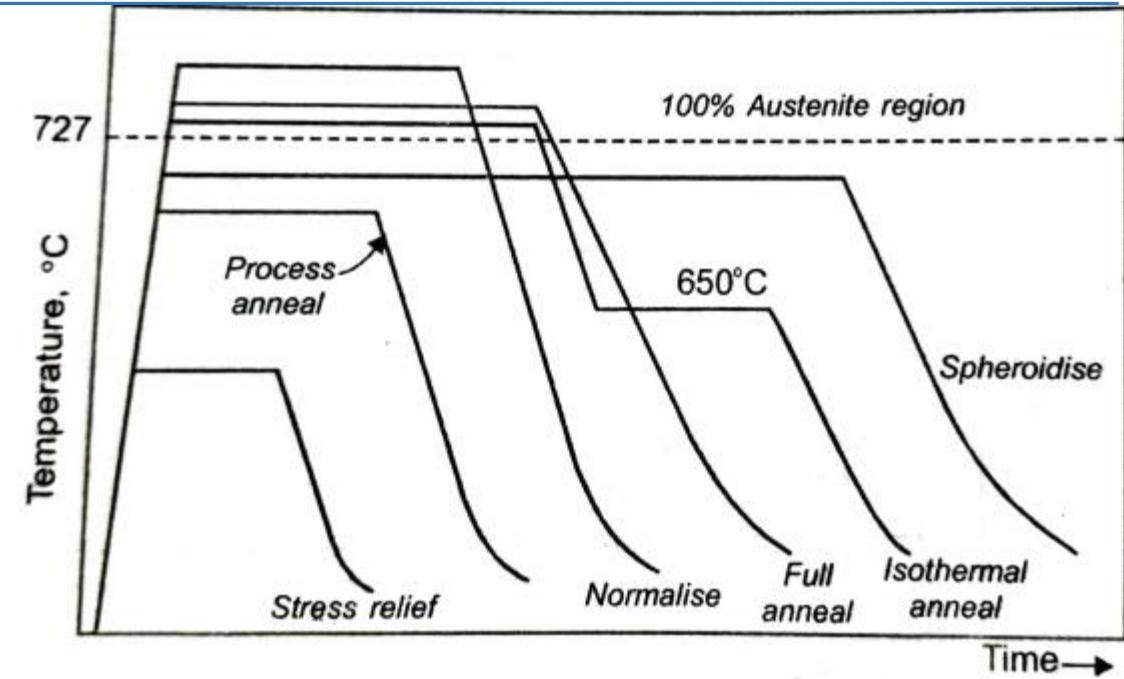


## Purpose of Annealing

- reduce hardness
- Improve machinability
- Relieve internal stress
- Improve mechanical properties like ductility, strength, toughness
- Refine the grain size
- Prepare steel for other heat treatments

## Types of Annealing

1. Full annealing
2. Sub critical/ Process annealing
3. Stress Relief annealing
4. Spheroidization Annealing
5. Homogenization Annealing
6. Isothermal Annealing



Temperature ranges for Annealing

## Full Annealing

**Heat above A<sub>3</sub> or A<sub>1</sub> → Hold → Cool slowly (Furnace cooling) (Coarse Pearlite)**

- The purpose of Full Annealing is to **obtain a material with high ductility**.
- A microstructure with **coarse pearlite** (i.e. pearlite having high interlamellar spacing) is endowed with such properties.
- Refer the range of temperatures used for different annealing processes.
- The steel is heated above A<sub>3</sub> (for hypo-eutectoid steels) and A<sub>1</sub> (for hyper-eutectoid steels) → (hold) → then the steel is furnace cooled to obtain Coarse Pearlite.
- Coarse Pearlite has low hardness but high ductility.
- For hyper-eutectoid steels the heating is not done above A<sub>cm</sub> to avoid a continuous network of pro-eutectoid cementite along prior Austenite grain boundaries (presence of cementite along grain boundaries provides easy path for crack propagation).

## Recrystallization Annealing

- During any cold working operation (say cold rolling), the material becomes harder (due to work hardening), but loses its ductility. This implies that to continue deformation the material needs to be recrystallized (wherein strain free grains replace the ‘cold worked grains’).
- Hence, recrystallization annealing is used as an intermediate step in (cold) deformation processing.
- To achieve this the sample is heated below  $A_1$  and held there for sufficient time for recrystallization to be completed.

**Heat below  $A_1$  → Sufficient time → Recrystallization**

## Stress Relief Annealing

- Due to various processes like quenching (differential cooling of surface and interior), machining, phase transformations (like martensitic transformation), welding, etc. the residual stresses develop in the sample. Residual stress can lead to undesirable effects like warpage of the component.
- The annealing is carried out just below  $A_1$ , wherein ‘recovery\*’ processes are active (Annihilation of dislocations, polygonization).

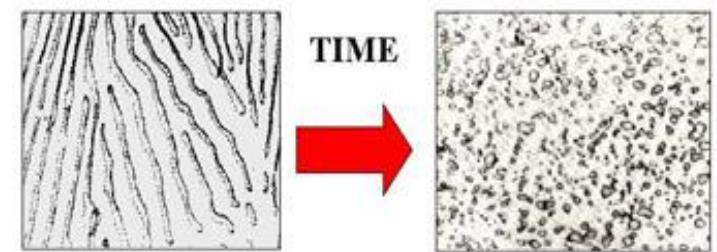
Residual stresses → Heat below  $A_1$  → Recovery

Annihilation of dislocations,  
polygonization

## Spheroidization Annealing

- This is a very specific heat treatment given to high carbon steel requiring extensive machining prior to final hardening and tempering.
- The main purpose of the treatment is to increase the ductility of the sample.
- Like stress relief annealing the treatment is done just below  $A_1$ .
- Long time heating leads cementite plates to form cementite spheroids. The driving force for this (microstructural) transformation is the reduction in interfacial energy.

**Heat below  $A_1$  → Hold for very long time → Cool slowly (Cementite spheroids)**



**Process :** The sample is heat above  $A_3$  /  $A_{cm}$  to complete Austenization. The sample is then air cooled to obtain **Fine pearlite**. Fine pearlite has a reasonably good hardness and ductility.

In hypo-eutectoid steels normalizing is done  $50^{\circ}\text{C}$  above the annealing temperature.

In hyper-eutectoid steels normalizing done above  $A_{cm}$  → due to faster cooling cementite does not form a continuous film along GB.

**Heat above  $A_3$  and  $A_{cm}$  → Hold → Cool in air (Fine Pearlite)**

**Purpose of Normalizing**

- Refine grain structure
- Increase the hardness
- Reduce segregation of alloying elements

# Annealing vs Normalizing

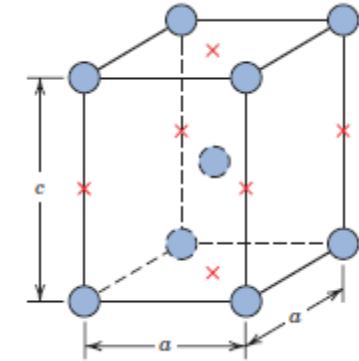
	Annealing	Normalizing
Austenising temperature	Above A3 or A1	Above A3 or Acm
Cooling rate / time	Very slow ( 10 – 100 <sup>0</sup> C/hr) Generally furnace cooled Takes several hours to few days	Air cooled or air quenched. Takes few minutes or hours. Cooling is faster.
Microstructure	Coarse grained pearlite	Fine grained pearlite
Mechanical properties	Very soft and ductile	Relatively less softer, less ductile but more tough
Economy	Expensive	Relatively less expensive
Applications	Used as pre treatment to obtain soft and ductile structure prior to machining or plastic deformation	Used as post treatment after plastic deformation to refine the grain structure and make the hard and tough structure

The sample is heated above  $A_3$  |  $A_{cm}$  to cause Austenization. The sample is then quenched at a cooling rate higher than the critical cooling rate (i.e. to avoid the nose of the TTT / CCT diagram).

The quenching process produces residual strains (thermal, phase transformation).

The transformation to Martensite is usually not complete and the sample will have some retained Austenite.

The Martensite produced is hard and brittle and tempering operation usually follows hardening. This gives a good combination of strength and toughness.



Martensite

**Heat above  $A_3$  |  $A_{cm}$  → Austenization → Quench (higher than critical cooling rate) (Martensite)**

**Martensite** – a microconstituent or phase called Martensite is formed when austenitized iron–carbon alloys are rapidly cooled (or quenched) to a relatively low temperature. Martensite is a nonequilibrium single-phase structure that results from a diffusionless transformation of austenite.

## Structure in Quenched state

- Highly supersaturated Martensite
- Retained austenite
- Undissolved carbides
- Rods, plates of carbide particles
- Segregation of carbons



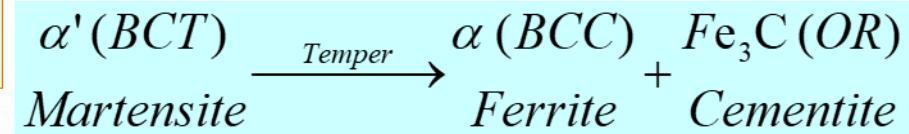
**Martensite**

Martensite grains take on a platelike or needlelike appearance, as indicated in figure. The white phase in the micrograph is austenite (retained austenite) that did not transform during the rapid quench. As already mentioned, martensite as well as other microconstituents (e.g., pearlite) can coexist.

**The martensitic transformation occurs when the quenching rate is rapid enough to prevent carbon diffusion** (any diffusion whatsoever will result in the formation of ferrite and cementite phases). This occurs in such a way that the FCC austenite experiences a polymorphic transformation to a body-centered tetragonal (BCT) martensite. A unit cell of this crystal structure is simply a body-centered cube that has been elongated along one of its dimensions. All the carbon atoms remain as interstitial impurities in martensite, as such, they constitute a supersaturated solid solution that is capable of rapidly transforming to other structures if heated to temperatures at which diffusion rates become appreciable by the diffusionless transformation. Because the martensitic transformation does not involve diffusion, it occurs almost instantaneously.

- A sample with martensitic microstructure (hardened steel) is hard but brittle. Hence after quenching the sample (or component) is tempered. Martensite being a metastable phase decomposes to ferrite and cementite (tempered Martensite) on heating (providing thermal activation).
- Tempering is carried out just **below the eutectoid temperature** (**heat → wait → slow cool**).
- The time temperature cycle for tempering is chosen so as to optimize strength and toughness. E.g. tool steel has a quenched hardness of  $R_c 65$ , which is tempered to get a hardness of  $R_c 45-55$ .

## Tempering



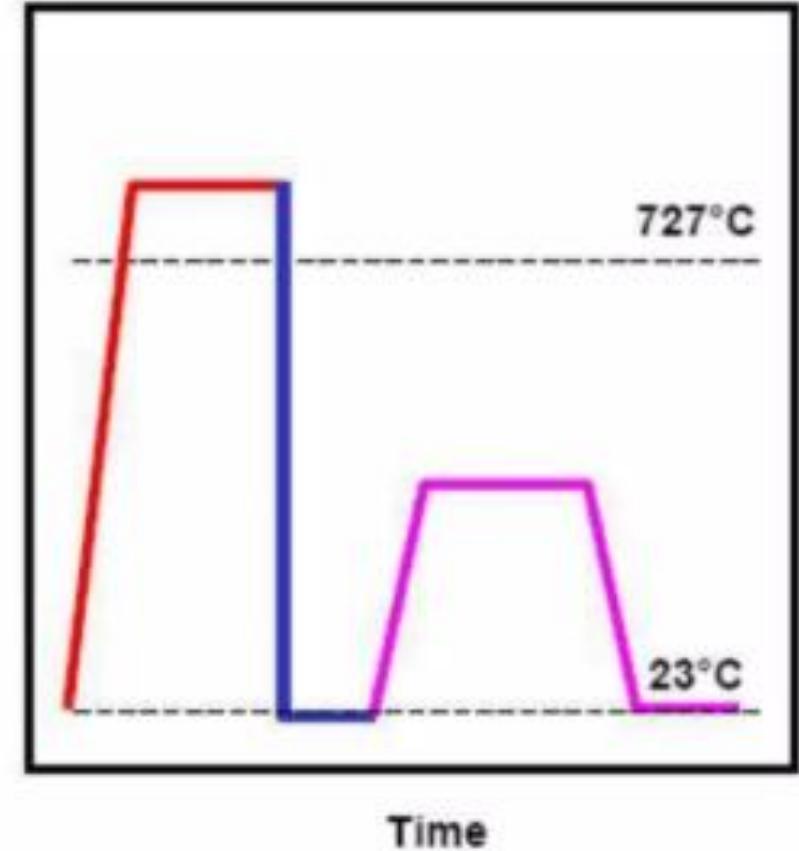
(Martensite) Heat below A<sub>1</sub> (650°C to 150°C) → Hold → Cool in the air (Tempered Martensite)

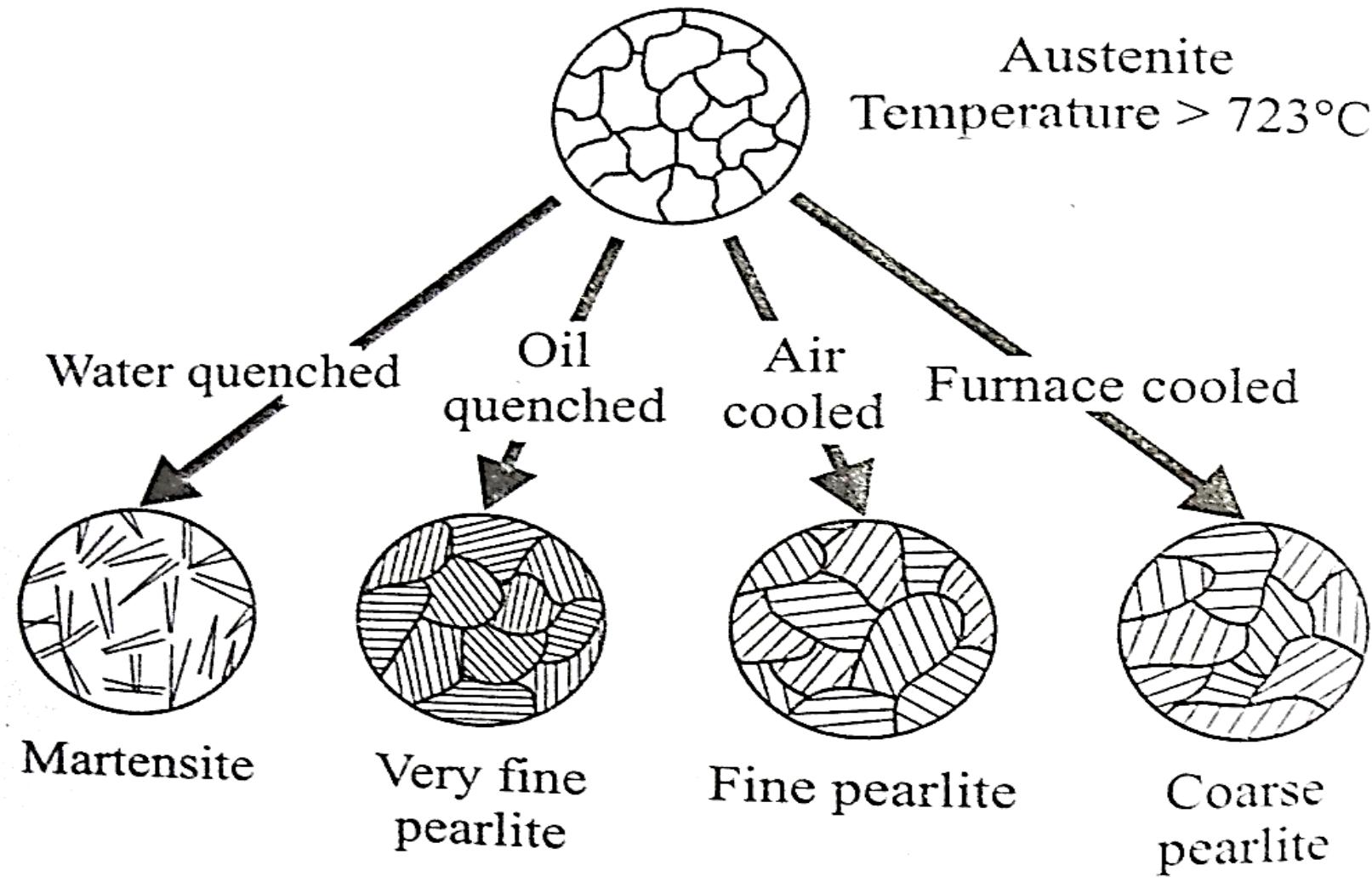
Depending on Temperatures, Tempering can be classified as

1. Low-Temperature Tempering ( 150 – 250<sup>0</sup>C)
2. Medium -Temperature Tempering ( 350 – 450<sup>0</sup>C)
3. High -Temperature Tempering ( 500 – 650<sup>0</sup>C)

## Objectives of Tempering

- Reduce brittleness
- Relieve internal stresses
- Restore ductility and toughness
- Improve dimensional stability
- Improve magnetic properties





Austenite forms in iron and steel alloys when heated above the eutectoid temperature of 723°C. The formation of austenite involves the diffusion of carbon atoms from cementite into ferrite. The process of transforming a metal's crystal structure into austenite is called austenitizing.

**Formation of austenite:**

**Nucleation** - Austenite nucleates at the interfaces of ferrite and carbide.

**Grain growth** - The rate of growth is the main factor in determining the size of austenite grains.

**Alloying elements** - Adding elements like nickel and manganese can stabilize austenite at room temperature. Elements like silicon, molybdenum, and chromium can make austenite unstable.

**Heating rate** - The change from carbon diffusion control to interface mobility control is only noticeable above certain heating rates.

**Austenitizing** - Austenitizing is important in manufacturing because it improves the strength, ductility, and corrosion resistance of steel products

**Grain size** – Initial grain size of the steel determines the grain size of austenite.

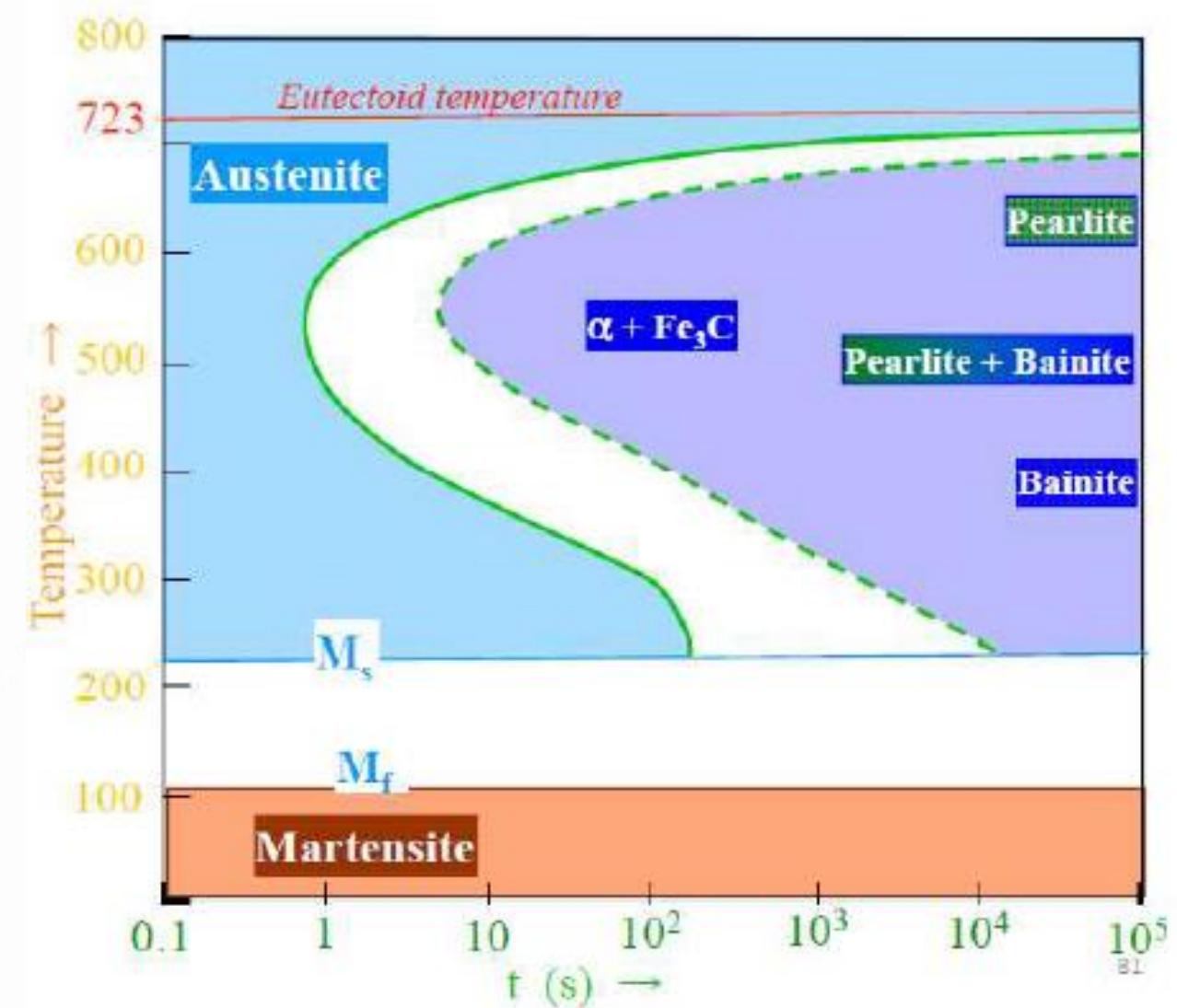
**Homogeneity** – Homogeneous austenite grains determine the structure of steel after heat treatment

Time temperature transformation (TTT) diagram controls the nature of transformed product of austenite which in turn decides the resultant properties of heat treated steels.

Transformation of austenite occur at constant temperature, so, the diagram also referred as Isothermal transformation diagram

There are many methods to determine TTT diagram. Here we will follow **salt bath technique**.

For every composition of steel, there is a different TTT diagram. Here we will exercise for 0.8% eutectoid steel.



## Determination of TTT Diagram of 0.8% (eutectoid) steel, using salt bath technique

- Two salt bath and one water bath are used
- Salt bath 1 is maintained at  $780^{\circ}\text{C}$ .
- Salt bath 2 is maintained at specified temperature at which transformation is to be determined (below A<sub>1</sub>, generally  $700 - 250^{\circ}\text{C}$ ).
- Bath 3 is a cold water bath maintained at room temperature.
- In bath 1 number of samples are kept for sufficient time (about an hour, to convert fully to austenite)
- Then samples are removed from bath 1 and transferred to bath 2 (each one is kept at different temperature for different specified time (ex. T<sub>1</sub>, T<sub>2</sub>, T<sub>3</sub> .... for t<sub>1</sub>, t<sub>2</sub>, t<sub>3</sub>.... )) and bath 3.
- The microstructure of each sample is studied using metallographic technique (determines type and quantity of phases).
- At a given temperature (ex. T<sub>1</sub>) start and finish time to convert to pearlite or martensite is noted from a transformation curve.
- The start and finish time is extended for different temperature and TTT diagram is constructed.

## Determination of TTT Diagram of 0.8% (eutectoid) steel

**X- axis** – Time in log scale

**Y- axis** – Temperature in  $^{\circ}\text{C}$

**Incubation period** - The minimum time required at a given temperature for the transformation of austenite to pearlite.

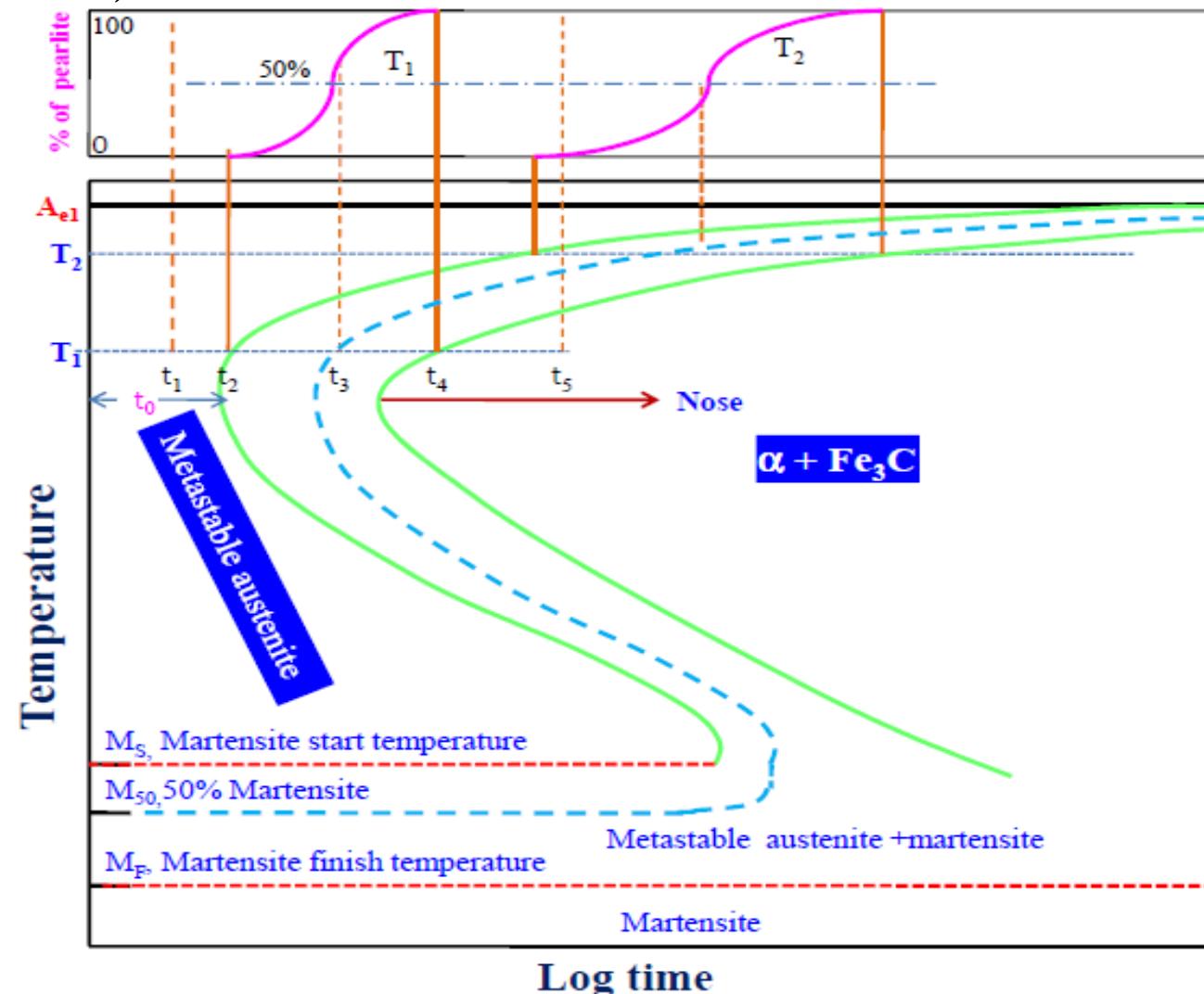
**Nose** – The temperature at which incubation period is minimum (in sec).

**C- curve** – Refers to the transformation of austenite to pearlite

**Horizontal diagram** - Refers to the transformation of austenite to martensite. If we quench below  $\text{Ms}$  ( $225^{\circ}\text{C}$ ), austenite transforms through a diffusionless transformation to a very hard phase known as martensite.

**Ms** – Start temperature of martensite

**Mf** – Finish temperature of martensite



## Phases

**Coarse Pearlite** – Upper part of the right side of the C curve above nose

**Fine Pearlite** - Lower part of the right side of the C curve above nose

**Austenite** – Left of the C curve – Untransformed austenite

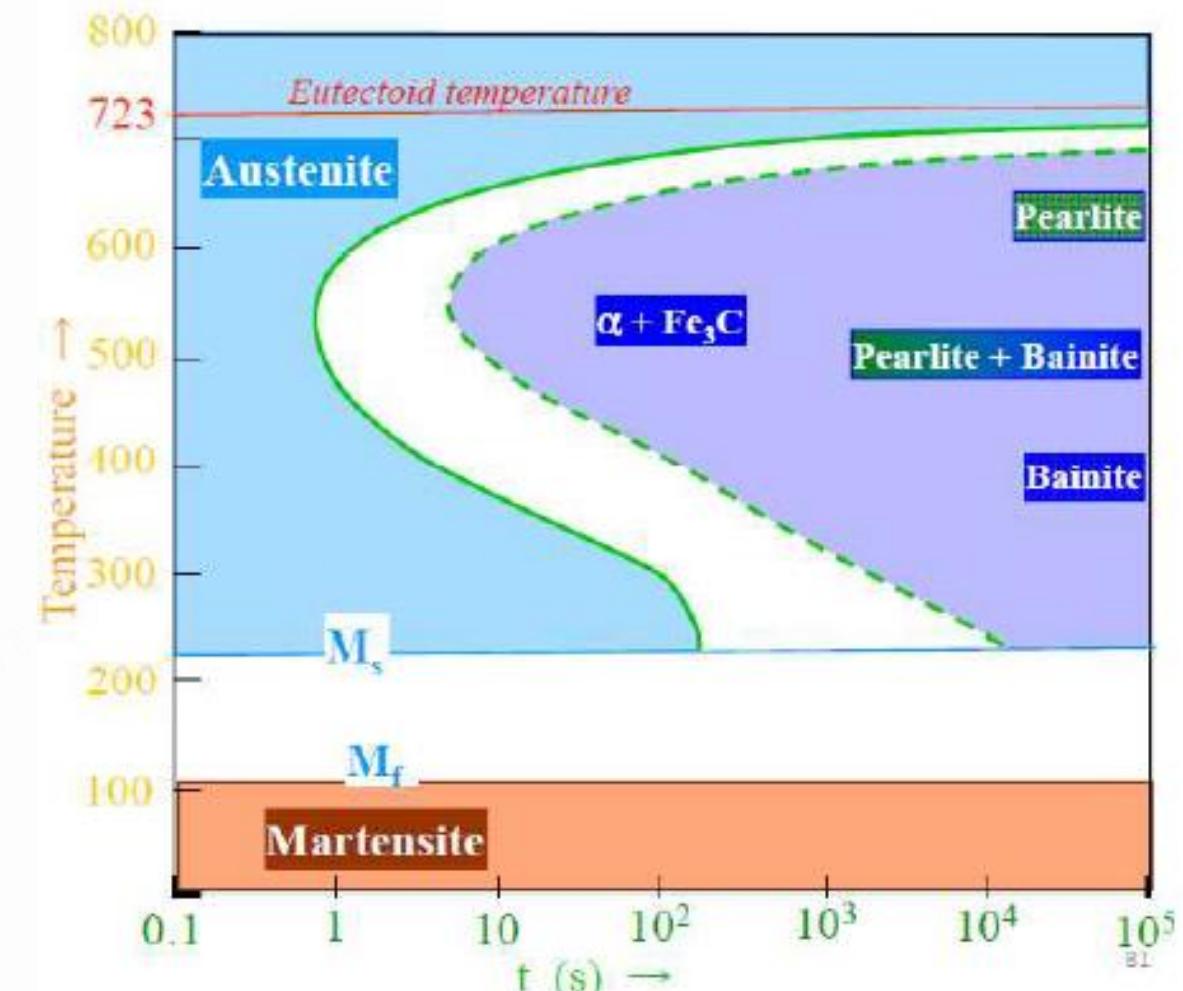
**Bainite** - Upper part of the right side of the C curve below nose

**Sorbite** (Upper bainite) - Upper part of the right side of the C curve below nose. Fine Bainite

**Troostite** (lower bainite) - Upper part of the right side of the C curve below nose. Very Fine Bainite

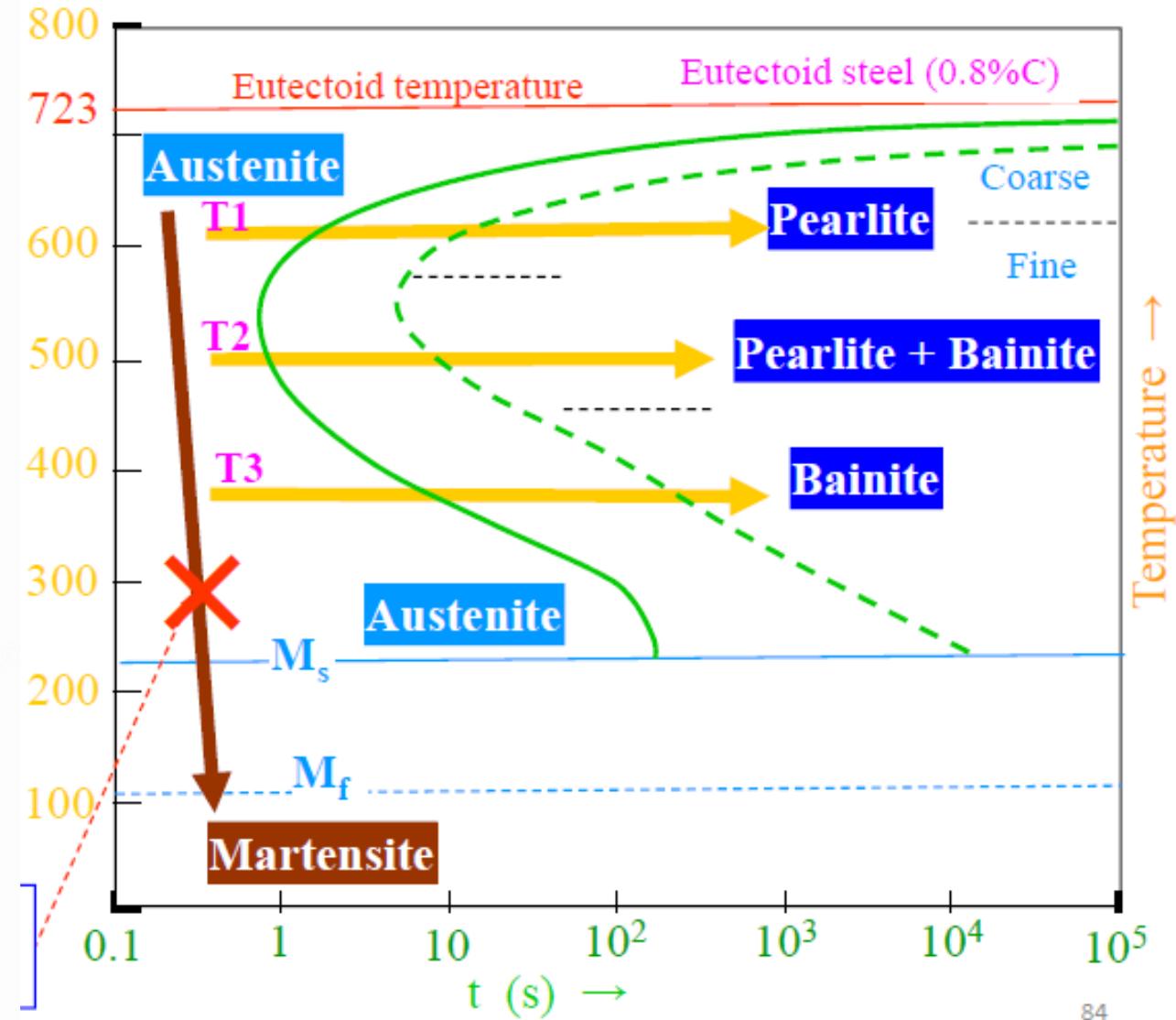
**Martensite** – In between Ms and Mf

**Retained Austenite** – Below Mf



## Isothermal transformation

- ❑ Isothermal hold at high temperature above nose (ex. T1) produces **coarse pearlite**.
- ❑ Isothermal hold at slightly low temperature above nose produces **fine pearlite**.
- ❑ Isothermal hold at low temperature (ex. T2) below nose produces **pearlite and Bainite**.
- ❑ Isothermal hold at low temperature (ex. T3) below nose produces **Bainite**.
- ❑ Transformation with cooling rate faster than critical cooling rate (quenching) produces **Martensite and retained austenite**. It is not an isothermal transformation.



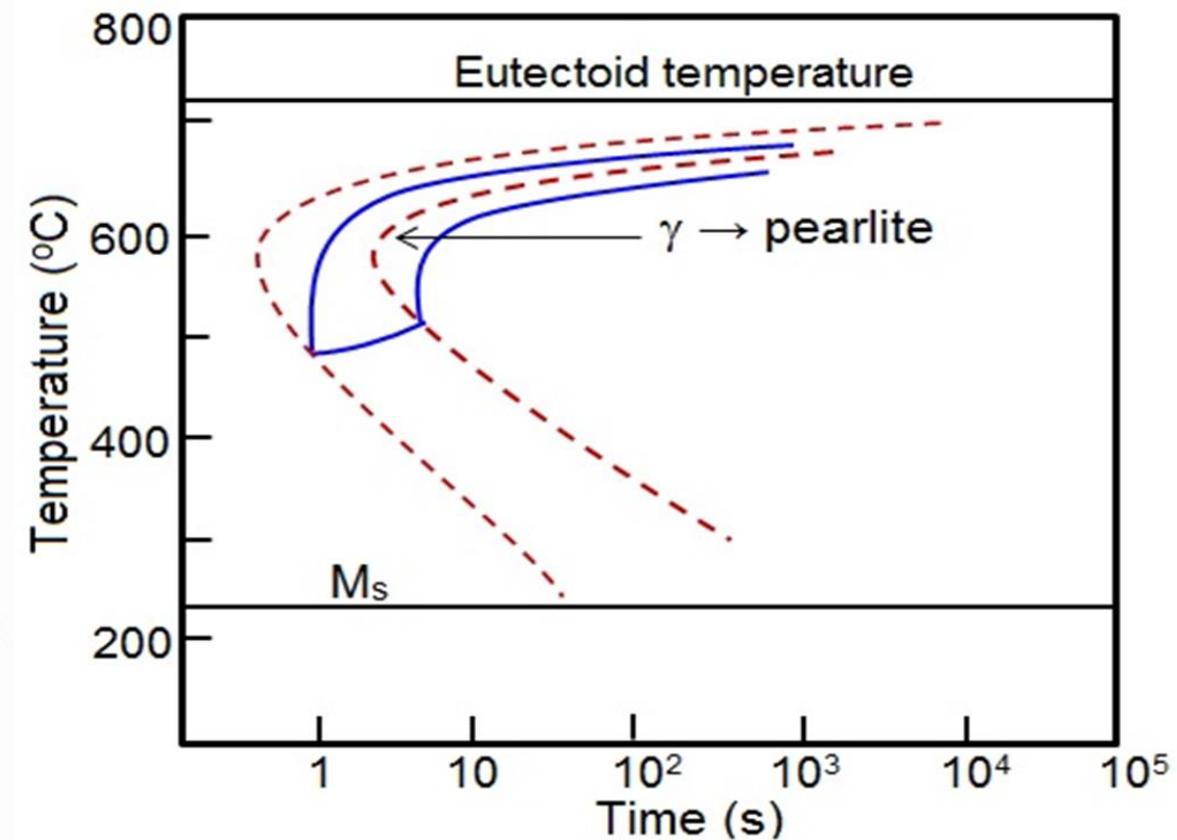
## Factors Affecting TTT diagram

- **Composition –**
  - Almost all alloying elements (except Al, Si, Co) increases the incubation period and moves the C curve to the right.
  - Elements (Ni, Mn, Ru, Rh, Pd, Os, Ir, Pt, Cu, Zn, Au) stabilizes austenite and depress or move the curve downward.
  - Elements (Be, P, Ti, V, Mo, Cr, Ta, Nb, Zr) stabilizes ferrite phase and move the curve upward.
  - Al, Si, Co shift TTT diagram to the left
  - C – In hypo and hyper eutectoid steel one more C curve is attached above the C curve of TTT diagram for ferrite and cementite phase respectively. It also pushes lower part of the curves downward.
- **Grain size** - Grain boundary facilitates nucleation sites for different phases like ferrite, pearlite, cementite, bainite etc. (not martensite because the transformation is diffusionless). Fine grain boundary reduces the incubation period and moves TTT curve to the left.
- **Heterogeneity of austenite** - Heterogeneous austenite increases incubation period, and favours martensitic transformation and bainite transformation

## Continuous Cooling Transformation curve (CCT Curves)

The TTT diagram show the structure produced by isothermal transformation i.e at a constant temperature. In practical situation heat treatment processes involves continuous cooling over a range of time.

We need to draw separate diagram which will show the transformation under continuous cooling. Such a diagram is termed as **Continuous Cooling Transformation (CCT)** diagram.



The CCT curve (**Blue**) is shifted to the right of the T-T-T (dashed) curve as continuous cooling transformation occurs at lower temperature and longer time compared isothermal holding

## Different cooling rates for eutectoid steel

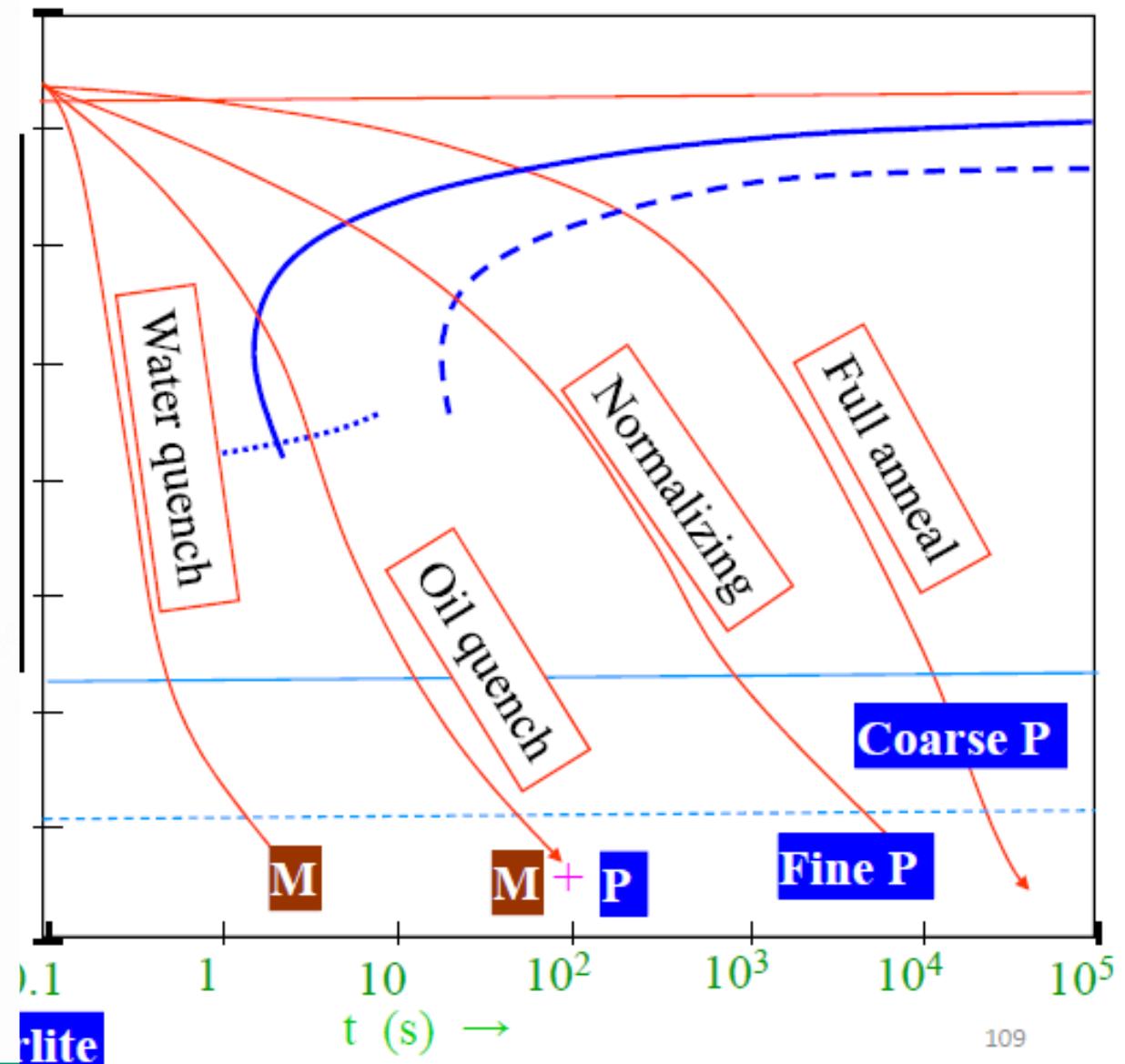
Figure shows continuous cooling diagram for eutectoid steel superimposed on the isothermal diagram from which it was derived.

On this diagram the superimposed curves represent different rates of cooling, and are continuous cooling curves.

### Critical cooling rate :

The minimum rate of cooling (quenching) at which the complete austenite transformed totally into martensitic structure.

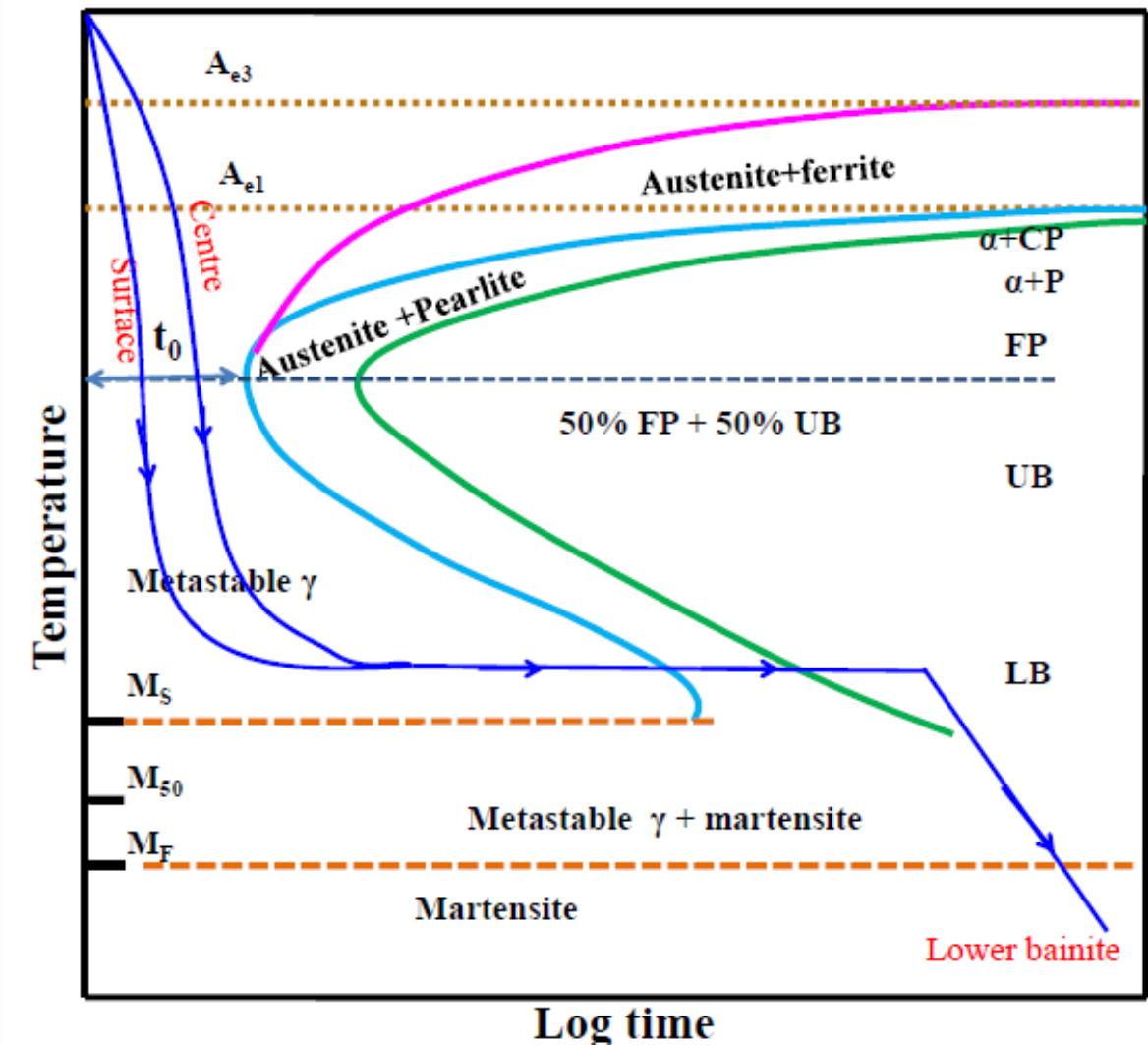
Carbon content and alloying elements affects the critical cooling rate



Austempering heat treatment is carried out to produce lower bainite in high carbon steel without any distortion or cracking to the specimen.

Austenite will be cooled rapidly in a bath maintained at lower bainitic temperature(above Ms, avoiding the nose of the TTT diagram) and holding it here to equalize surface and centre temperature till bainitic transformation finish time and it will be air cooled.

The microstructure contain fully **lower bainite**. The hardness and strength are comparable to hardened and tempered martensite with improved ductility and toughness and uniform mechanical properties. The final product do not required to be tempered.

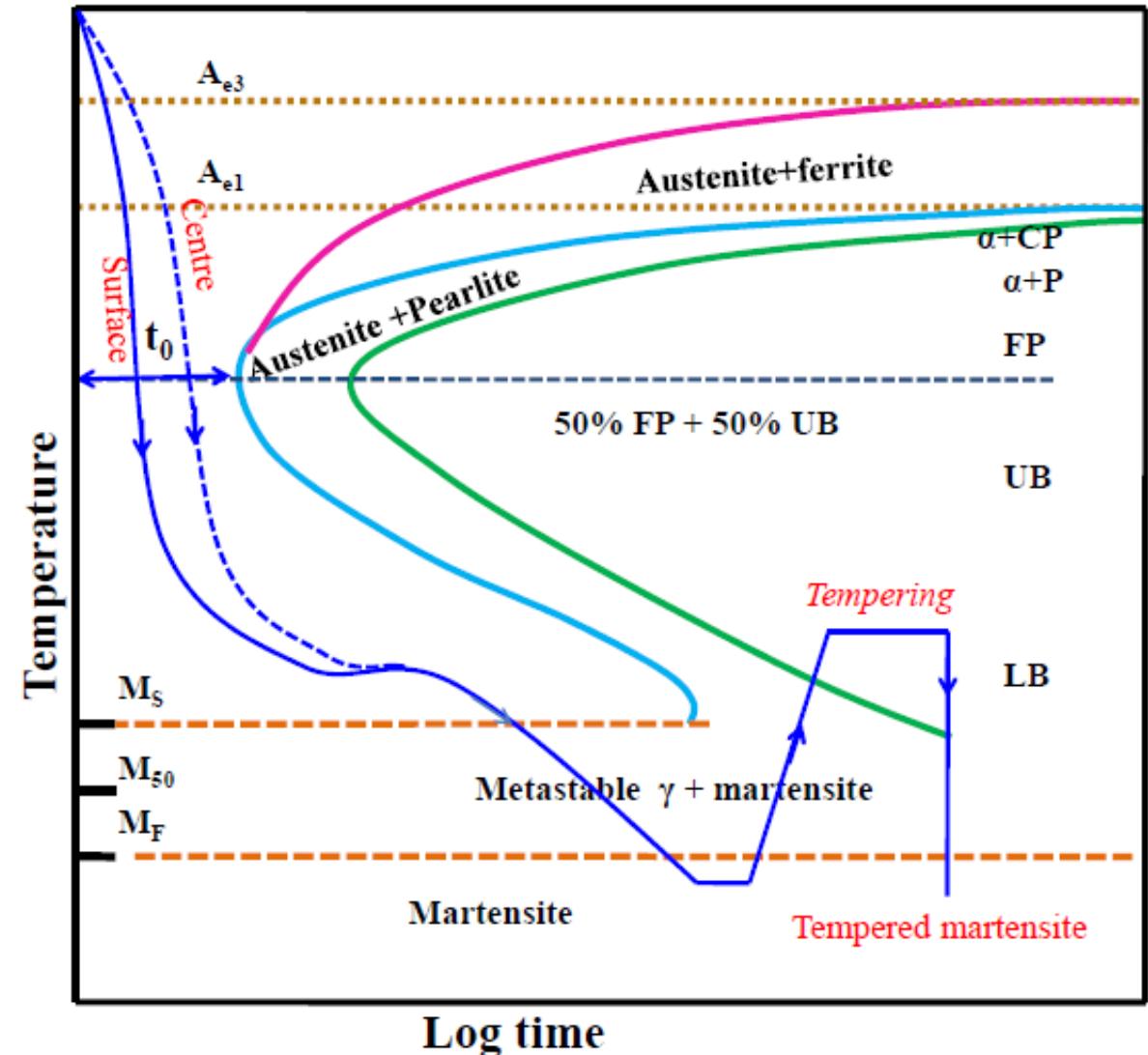


# Martempering

Go, change the world

**Martempering** heat treatment is performed to steel samples to produce martensite with minimal differential thermal and transformational stress to avoid distortion and cracking.

The steel should have reasonable incubation period at the nose of its TTT diagram and long bainitic bay.



## Martempering

The sample is quenched above Ms temperature in a salt bath to reduce thermal stress (instead of cooling below Mf directly) surface cooling rate is greater than at the centre.

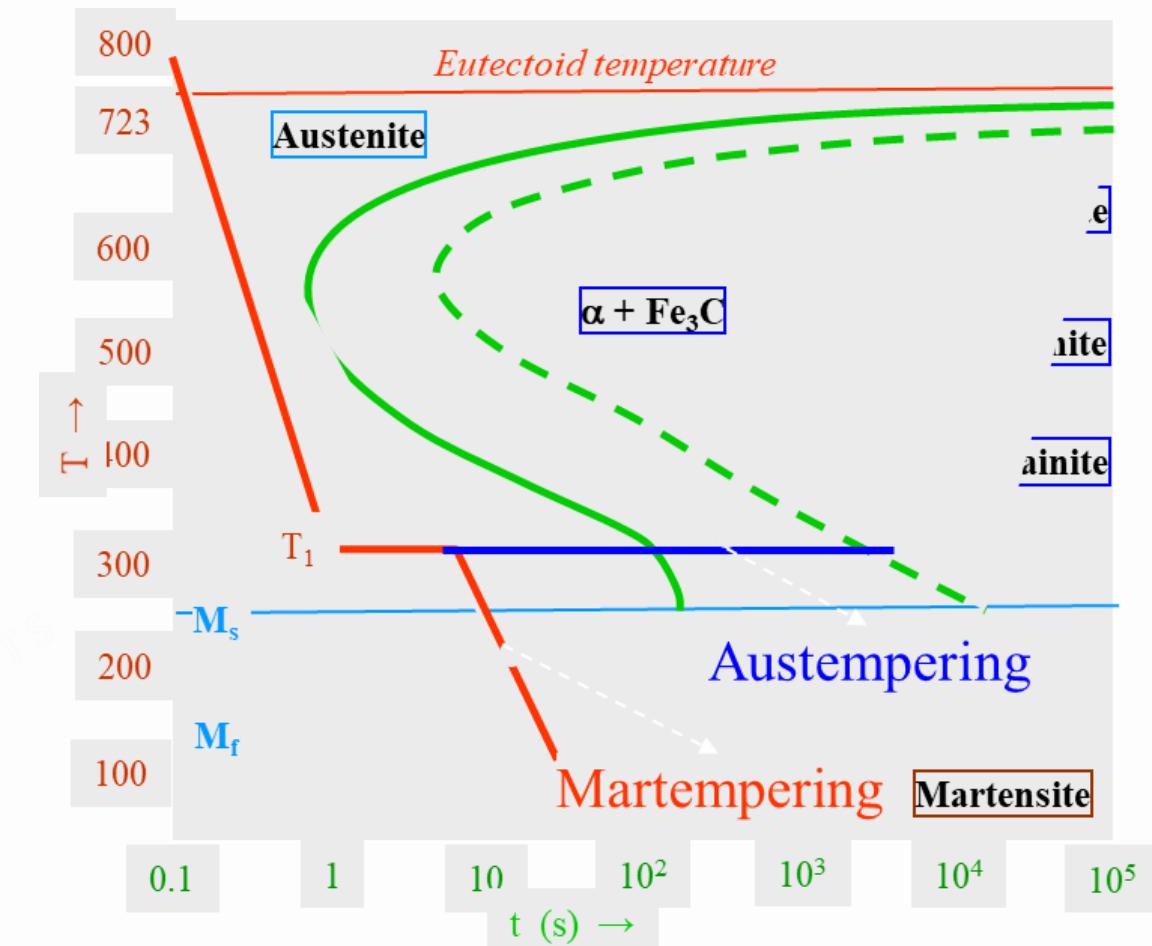
The cooling schedule is such that the cooling curves pass behind without touching the nose of the TTT diagram. The sample is isothermally hold at bainitic bay such that differential cooling rate at the centre and surface become equalize after some time.

The sample is allowed to cool by air through Ms-Mf such that martensite forms both at the surface and centre at the same time due to not much temperature difference and it avoids transformation stress because of volume expansion.

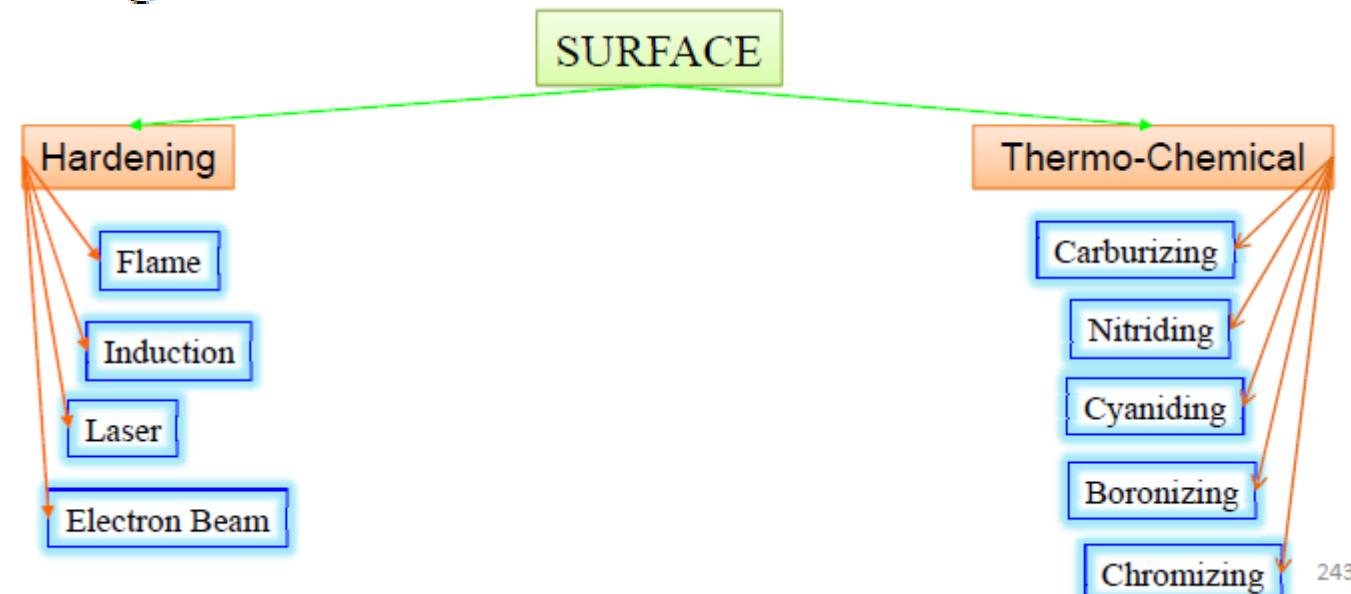
The rate of cooling from austenite temperature should avoid formation pearlite and bainite. Thus the success of Martempering depends on the incubation period at the paerlitic nose and also at the Bainitic bay and thus the steel suitable for Martempering should have sufficient hardenability. Plain carbon steels with diameter more than 10 mm are difficult to Martempering, this process is restricted to alloy steels.

# Martempering and Austempering

- **Martempering and Austempering** processes have been developed to avoid residual stresses generated during quenching.
- In **Martempering and Austempering** processes austenized steel is quenched above  $M_s$  ( $T_1$ ) for homogenization of temperature across the sample.
- In **Martempering** the steel is then quenched and the entire sample transforms simultaneously to martensite. This is followed by tempering.
- In **Austempering** instead of quenching the sample, it is held above  $M_s$  ( $T_1$ ) for it to transform to bainite.



- Chemical heat treatment is the process used to achieve different properties in core and steel components. Numerous industrial applications require a hard wear resistant surface called the *case*, and a relatively soft, tough inside called the *core*. Example: Gears
- They are two different methods. The first method is known as thermochemical treatment because the surface composition of steel changes by diffusion of carbon and/or nitrogen and sometimes other elements.
- The second method is known has surface hardening, it involves phase transformation by rapid heating and cooling of the outer surface . The aim of both methods are same



## Purpose of surface hardening

- To make wear resistant outer surface
- To obtain an inner surface which can withstand shock load
- To improve the corrosion resistance
- To improve the thermal resistance
- To improve the life of the material a

Carburizing is the process of saturating steel surface with carbon. Usually low carbon (0.2 % max) are subjected to this process and as high as 1.4 % C is diffused into their case. The operation is performed in a furnace or steel shell with carbonaceous material at about  $950^{\circ}\text{C}$ . After sufficient time the steel part is required to quench.

Different methods are

- **Pack carburizing** – Steel component is heated in a container packed with charcoal
- **Liquid carburizing** – Also known as salt bath carburizing. Carburizing occurs through molten Cyanide (CN) bath heated by a furnace maintained at  $815 - 900^{\circ}\text{C}$ . In this process heating time is short, heat transfer is rapid and produces thin and clean hardened layer of approx. 0.08 mm thick.
- **Gas carburizing** – It carried out in a electrical furnace maintained at  $870 - 950^{\circ}\text{C}$ . Gas atmosphere in the furnace is produced using liquid methanol, isopropanol or other gaseous hydrocarbons.

**Advantages :** Improved wear resistance, corrosion resistance, fatigue strength, surface toughness, easy, low cost, no atmosphere control furnace is required, good dimensional control, carbon concentration is possible to control, non-toxic. 0.8 to 1.5 mm case depth can be achieved.

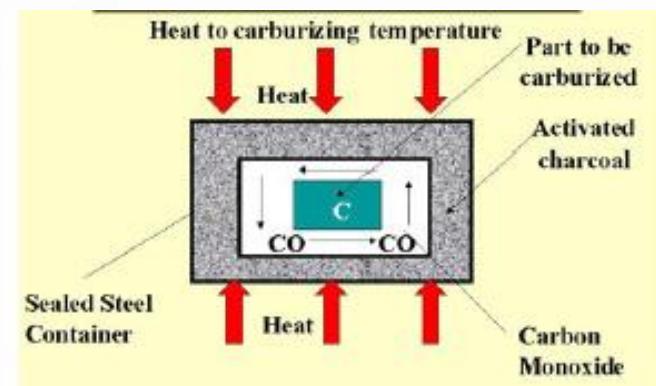
**Disadvantages :** Take longer time, High temperature, not a clean process, decarburization may occur, crack and distortion may occur, labour intensive.

**Applications :** Gears, shafts etc

**Pack carburizing** – also known as solid carburizing. Steel component to be heat treated are packed with 80% granular coal and 20% BaCO<sub>3</sub> as energizer in heat resistant boxes and heated at 930°C in furnace for a specific time which depends on the case depth required. The following reaction take place inside the box. The atomic C absorbed at the surface of the steel

1. BaCO<sub>3</sub> → BaO + CO<sub>2</sub>
2. CO<sub>2</sub> + C → 2CO
3. 2CO + Fe → Fe(c) + CO<sub>2</sub>

The free C will diffuse into the surface of the steel. After sufficient Time the steel part is required to quench.



- Such a high temperature in furnace helps in absorption of carbon at the outer layer.
- Case depth =  $k\sqrt{t}$  k is constant, t is time
- Generally carburizing time varies from 6 to 8 hours to obtain case depth of 1mm to 2mm.
- The carburizing equation at 3 is reversible and may proceed to left, removing carbon from the surface layer – known as decarburization

Nitriding involves diffusion of nitrogen into the product to form nitrides. The resulting **nitride case are harder than the carburised steel**.

This process is used for alloy steels containing elements (Al, Cr, Mo) which form stable nitrides.

In Nitriding the component is heated in a retort at a temperature of about 500 to 600°C. Ammonia gas is circulated through the retort. At this temperature ammonia dissociates by the following reactions



The atomic nitrogen diffuses into the steel surface and combines with the alloying elements (Cr, Mo, V, W etc. ) to form hard nitrides.

After nitriding the job is cooled slowly.

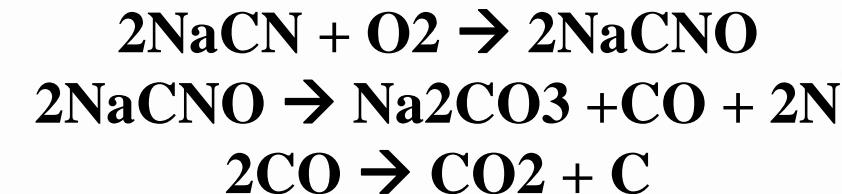
**Advantages :** Depth of nitride case is in the range of 0.2 to 0.4 mm and depends on the temperature and time of nitriding. Increase wear and corrosion resistance and fatigue strength of the steel. Quenching is not involved in this process. Chances of cracking and distortion of component are less. No machining is done after nitriding.

**Disadvantages :** Nitriding is done at low temperature so, Time required are longer than carburising. Also capital cost is higher than carburising. Not suitable for plain carbon steel

**Applications :** Small gears, bearings, valve seats, aircraft parts, guideways

In Cyaniding the surface of steel (with 0.3 – 0.4 % C) is hardened by addition of both carbon and nitrogen.

Liquid salt bath is used for Cyaniding and temperature used are generally lower than carburizing process between 750- 900°C. Exposure is for shorter time and thinner cases are produced. Case depth is generally in the range of 0.25 mm for Cyaniding. In the salt bath following reaction occurs.



Carbon and nitrogen so formed in atomic form diffuse into the steel and give this wear resistant layer of the carbonitride  $\varepsilon$ -phase.

**Advantages :** Surface hardenability, wear resistance and corrosion resistance is better than carburizing, thin case depth can be produced.

**Disadvantages :** Not suitable for parts subjected to shock, fatigue and impact, because nitrogen has adverse effect on these properties of steels.

**Applications :** Gears, bolts, nuts etc.

In Carbonitriding process also, the surface of steel (with 0.3 – 0.4 % C) is hardened by addition of both carbon and nitrogen. The process is also known as gas cyaniding or dry cyaniding.

In this process steel heated in a gaseous atmosphere ( $\text{NH}_3$ ,  $\text{CH}_4$  and neutral gas) temperature used are generally lower than carburizing process between  $750\text{-}900^\circ\text{C}$ .

With the increase of temperature carbon and nitrogen enters the steel and make carbon – nitrogen austenite stable at lower temperatures. The case depth produced is in the range of 0.8 mm.

**Advantages :** Surface hardenability, wear resistance and corrosion resistance is better than carburizing, thin case depth can be produced. fast and economic, suitable for mass production, less distortion.

**Disadvantages :** Time required is longer than carburizing. Temperature required is lower than carburizing.

**Applications :** Suitable for plain C steel such as small gears, bolts, nuts, pins etc.

The flame hardening involves heating the surface of a steel to a temperature above upper critical temperature ( $847^{\circ}\text{C}$ ) with a oxyacetylene flame and then quenched the surface with cold water. Suitable for steels having carbon content 0.4 to 0.95 % C and low alloy steels. Case depth up to 3mm can be achieved.

Heating transforms the structure of surface layers to austenite and the quenching changes it to martensite. The surface layers are hardened to about 50-60 HRC. Flame hardened parts must be tempered after hardening.

## **Methods :**

Stationary - Torch and work is stationary

Progressive – Torch moves over the workpiece

Spinning – Torch is stationary while workpiece rotates

Progressive - spinning – Torch moves over a rotating work piece

**Advantages :** Simple and economical, portable, suitable for large and complex machine parts, Distortion and scale formation is less.

**Applications :** Machine guide ways, gears, shafts, mill rolls, piston pins, cams

# Flame Hardening

Go, change the world

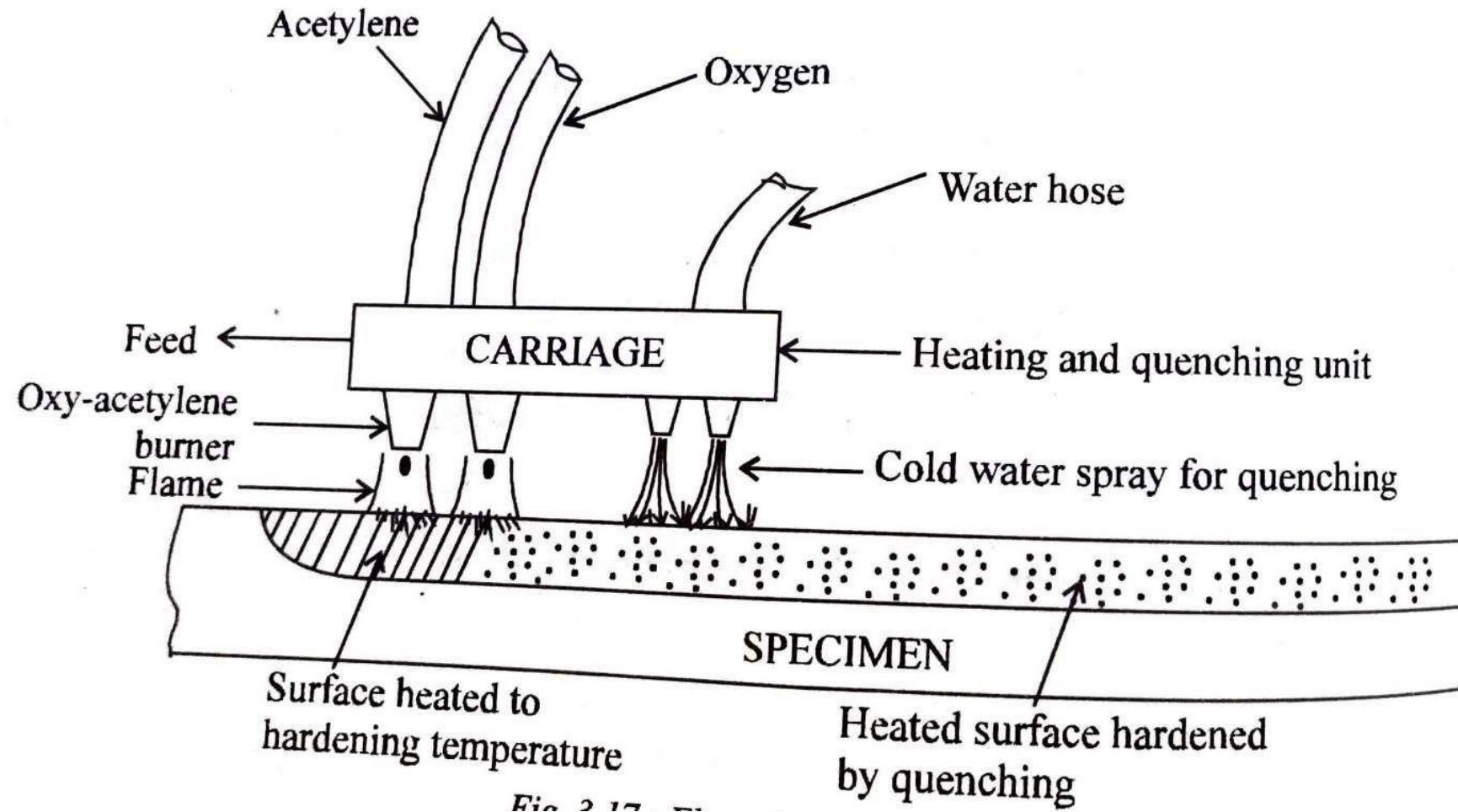


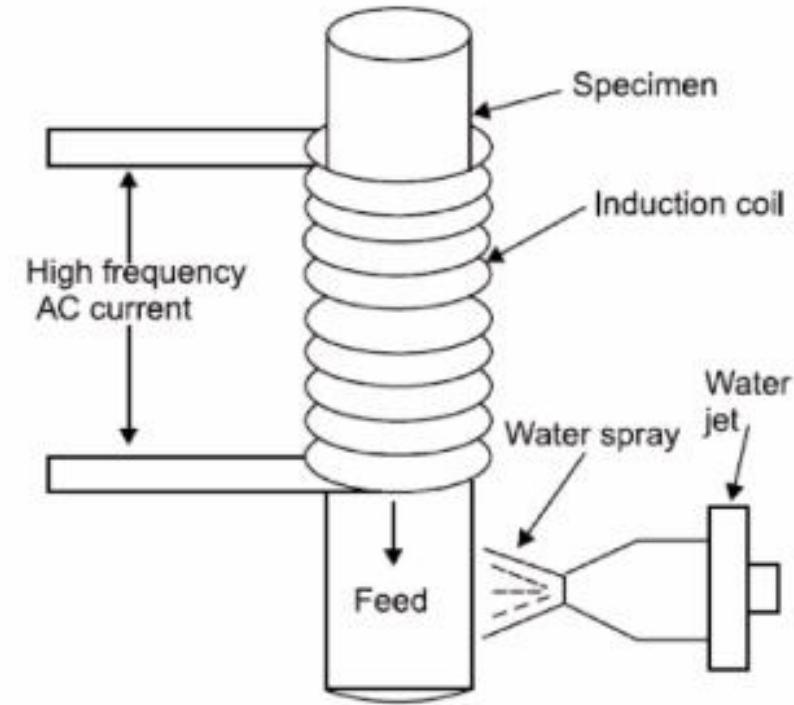
Fig. 2.17

Induction hardening involves heating the steel component through electromagnetic induction. Steel components are placed within a coil through which high frequency current is passed. The current in the coil induces eddy current in the surface layers, and heat the surface layers up to austenite state. Then the surface is immediately quenched with the cold water to transform the austenite to martensite.

**Advantages :** Improved wear resistance, increased strength and fatigue life, minimal distortion, low cost, selective hardening is possible, precision can be achieved, faster process, can be automated.

**Disadvantages :** Costly, low C steels cannot be hardened, difficult to harden complicated shapes.

**Applications :** Surface hardening of crankshafts, camshafts, gears, crank pins, axles, piston rods, cylinder liners etc.



Heat treatment defects can occur during various processes such as annealing, quenching, tempering, and stress relieving. These defects can have detrimental effects on the mechanical properties and performance of the treated material. Here are some common heat treatment defects

- Decarburization
- Quench cracks
- Fatigue cracking
- Rough surface finish
- Soft spots
- Distortion and warping
- Oxidation

To minimize these heat treatment defects, it is crucial to have a thorough understanding of the material properties, suitable heat treatment processes, and strict adherence to process parameters. Additionally, regular quality checks, monitoring, and adjustment of heat treatment equipment can help maintain consistent and controlled heat treatment outcomes.

## □ Decarburization:

- Cause: Exposure to oxygen at high temperatures leads to the loss of carbon from the surface layer of the material.
- Effects: Reduced hardness, decreased wear resistance, and compromised strength.
- Prevention: Using protective atmospheres (e.g., controlled atmospheres or vacuum) or coating the material with a protective layer.

## □ Quench Cracking:

- Cause: Rapid cooling during quenching results in thermal stresses that exceed the material's strength, leading to cracking.
- Effects: Component failure, reduced strength, and compromised dimensional stability.
- Prevention: Proper selection of quenching media, minimizing temperature gradients, and preheating the material.

## □ Distortion / Warping:

- Cause: Non-uniform cooling or heating rates cause differential expansion or contraction, leading to warping or bending of the material.
- Effects: Incorrect component fit, misalignment, or assembly issues.
- Prevention: Proper fixture design, controlled heating and cooling rates, and stress relief techniques like tempering or annealing.

## □ Soft Spots:

- Cause: Non-uniform heating or cooling during the treatment, resulting in localized areas with insufficient hardness.
- Effects: Reduced strength, compromised wear resistance, and premature failure.
- Prevention: Ensuring uniform heat distribution, using proper heating and quenching techniques, and employing temperature monitoring devices.

## □ Overheating:

- Cause: Excessive exposure to high temperatures during heating, leading to grain growth, burning, or material degradation.
- Effects: Coarsening of the microstructure, reduced mechanical properties, and dimensional instability.
- Prevention: Proper control of heating parameters, avoiding prolonged exposure to high temperatures, and using temperature controls and monitoring.

## □ Case Hardening Issues:

- Cause: Inadequate carbon diffusion or incorrect composition of the case-hardening medium.
- Effects: Insufficient hardness depth, low surface hardness, and poor wear resistance.
- Prevention: Proper selection and control of case-hardening methods, ensuring sufficient carbon availability, and appropriate quenching practices.

*The End*