

Module 3 -Nucleation, plastic deformation and Heat treatment

Subject: Material Science and Engineering

Subject Code: 21ME33

Syllabus

Nucleation and growth: Introduction to homogeneous and heterogeneous nucleation, critical radius for nucleation.

Plastic Deformation: Slip, Twinning; Recovery- Recrystallization-Grain Growth, Introduction to Strengthening mechanisms. Lever rule and phase diagram.

Heat treatment: Annealing, Normalizing, hardening, Tempering, Nitriding, Cyaniding, Induction Hardening and Flame Hardening,, Recent advances in heat treat technology. TTT diagram, microstructural effects brought about by these processes and their influence on mechanical properties

Nucleation and growth

Introduction to homogeneous and heterogeneous nucleation:

Nucleation in metals & alloys may occur by 2 mechanisms namely:

- 1] Homogeneous nucleation
- 2] Heterogeneous nucleation

The distinction between them is made according to the site at which nucleating events occur. For the homogeneous type, nuclei of the new phase form uniformly throughout the parent phase, whereas for the heterogeneous type, nuclei form preferentially at structural inhomogeneities, such as container surfaces, insoluble impurities, grain boundaries, and dislocations.

Homogeneous Nucleation

In homogeneous nucleation, the probability of nucleation occurring at any given site is identical to that at any other site within the volume of the parent phase. If Δf is the free energy change accompanying the formation of a spherical new phase particle, we can write

$$\Delta f = \frac{4}{3} \pi r^3 \Delta g + 4\pi r^2 \gamma$$

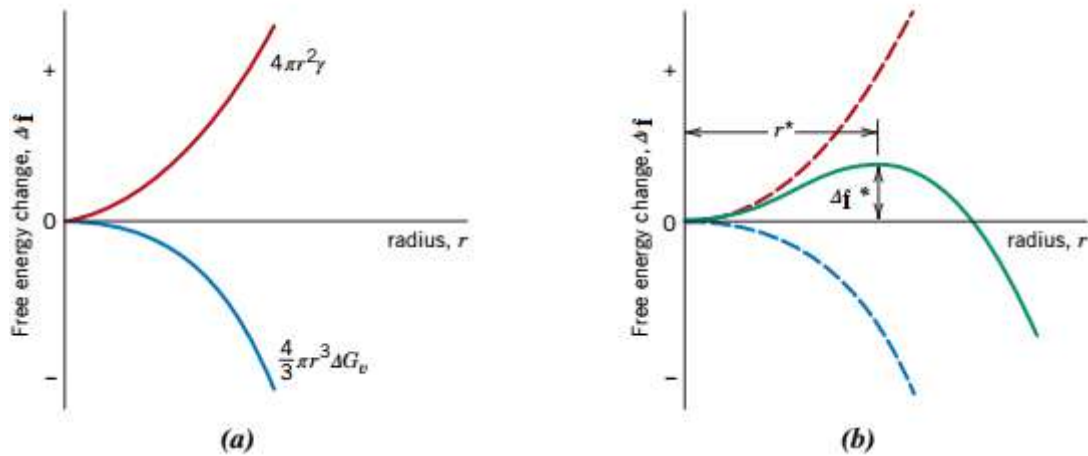
where r is the radius of the particle, Δg is the Gibbs free energy change per unit volume and γ is the surface energy per unit area of the interface separating the parent and the product phases. The surface energy term is always positive. If Δg is negative, the function f passes through a maximum. Initially as the new phase particle starts to form, the energy of the system increases, as the surface energy term is dominant. At the maximum, the variations with r of the surface energy and the volume (Gibbs) free energy exactly balance each other. Thereafter, the variation in the volume term becomes dominant and, as this term is negative, there is a continuous decrease in the energy of the system.

Critical radius of nucleation

By setting $(df/dr) = 0$, in the above equation, the values corresponding to the maximum, called the critical values and denoted by the superscript *, are obtained:

$$r^* = -\frac{2\gamma}{\Delta g}$$

$$\Delta f^* = \frac{16}{3} \pi \gamma^3 / (\Delta g)^2$$

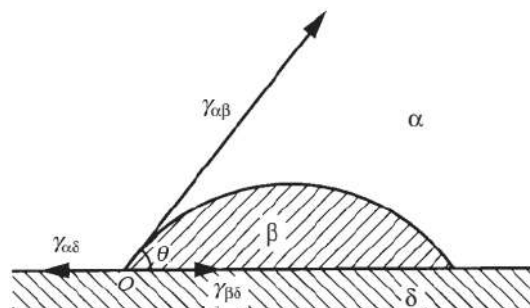


(a) Schematic curves for volume free energy and surface free energy contributions to the total free energy change attending the formation of a spherical embryo/nucleus during solidification. (b) Schematic plot of free energy versus embryo/nucleus radius, on which is shown the critical free energy change (Δf^*) and the critical nucleus radius (r^*)

Particles which are smaller than the critical size are called *embryos*. Those which are larger than the critical size are called *nuclei* (not to be confused with atomic nuclei). The critical sized particle is a *critical nucleus*. As Δg becomes more negative with a lowering of the temperature, the critical condition occurs at smaller values of f and r .

Heterogeneous nucleation

In *heterogeneous nucleation*, the probability of nucleation occurring at certain preferred sites in the assembly is much greater than that at other sites. During solidification of a liquid, inclusions of foreign particles in the liquid and the walls of the container holding the liquid provide preferred nucleation sites. In a solid-solid transformation, foreign inclusions, grain boundaries, interfaces, stacking faults and dislocations can provide sites for preferred nucleation. consider the nucleation of β from α occurring on the planar surface of a foreign inclusion, as illustrated in Fig.



The β particle nucleates on the surface of δ , which is a foreign inclusion in the matrix of α .

The β particle forms in the shape of a half lens on the δ surface. To calculate the nucleation kinetics, we assume that the surface tension forces at point such as O in Fig. are in equilibrium

$$\gamma_{\alpha\delta} = \gamma_{\alpha\beta} \cos \theta + \gamma_{\beta\delta} \quad \text{Eq (1)}$$

where θ is the contact angle. An expression for Δf can now be written in terms of the volume energy and the surface energies of the interfaces involved.

During nucleation, a new α - β interface of area equal to the curved surface of the lens is created. In addition, a β - δ interface equal in area to the circle of intersection of the lens with the planar surface is created. But an interface of the same area is consumed. This leads to a total of three surface energy terms: two of them positive and one negative. When the expression for Δf is written taking all these terms into account and the equilibrium condition from Eq. (1) is substituted, Δf^* comes out to be

$$\Delta f_{\text{het}}^* = \frac{4\pi\gamma_{\alpha\beta}^3}{3(\Delta g)^2} (2 - 3 \cos \theta + \cos^3 \theta)$$

This is the expression for the critical free energy in the case of heterogeneous nucleation.

Note: On comparing the term on the left hand side of this equation outside the bracket of the above equation with the critical free energy equation for homogeneous nucleation we can see that $\Delta f_{\text{het}}^* = 1/4 \Delta f_{\text{homo}}^*$.

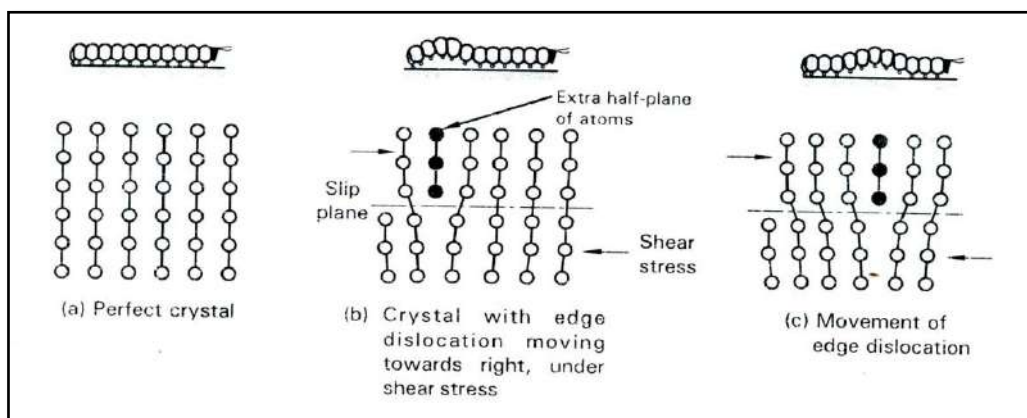
Plastic Deformation:

Plastic deformation in a single crystal occurs by:

- Slip
- Twinning, or
- Combination of slip and twinning

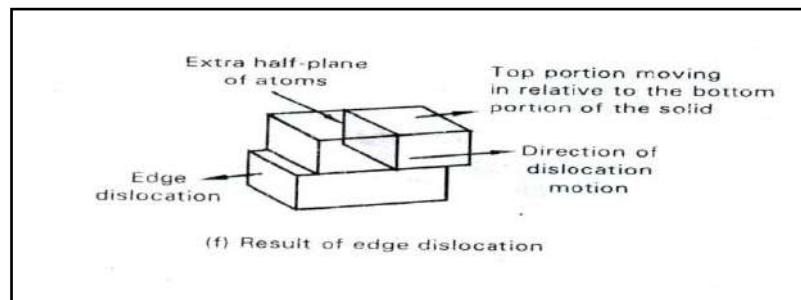
1. Plastic Deformation by Slip:

The process by which plastic deformation is produced by the motion of a large number of dislocations is termed as slip.



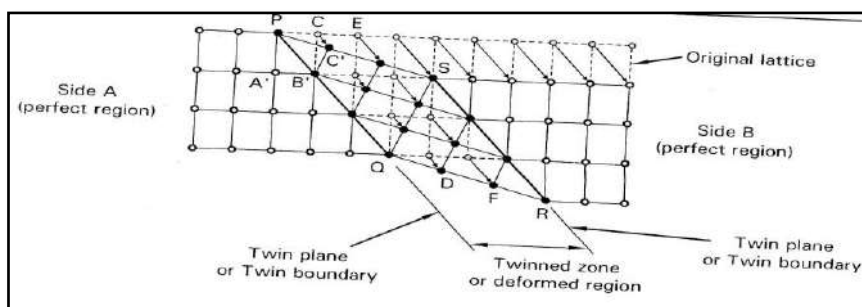
- ▶ Figure shows **an edge dislocation**, which helps in explaining slip under the action of shear stress. The figure illustrates how an edge dislocation gets glided along the slip plane under the action of shear stress, and how the slip is caused by the movement of edge dislocation.

- ▶ The dislocation motion is analogous to the movement of a caterpillar, which is also shown in figure above the crystal lattice for clear understanding. The caterpillar would have to exert a large force to move its entire body at once. Instead, it moves the rear portion of its body forward by a small amount and creates a hump. The hump then moves forward, and eventually moves all of the body forward by a small amount.
- ▶ Under the influence of shear stress, the dislocation in a crystal structure moves similarly by a small amount as that of a caterpillar. It can be seen in figure that, the dislocation in the top half of the crystal is slipping one plane at a time as it moves to the right from its initial position as is shown in figure (a), figure (b), and finally (c). In the process of slipping, the dislocation propagates across the crystal.
- ▶ The movement of the dislocation across the plane eventually causes the top half of the crystal to move with respect to the bottom half. Due to the actual movement of the atomic blocks, no return to the original lattice shape is possible, even after withdrawal of the straining force. This results in permanent deformation, which is clear from figure (e) when compared to figure (a)
- ▶ Figure (f) shows the 3-D view of the formation of a step on the surface of the crystal due to the motion of an edge dislocation



2. Plastic Deformation by Twinning:

Twinning is the movement of atoms in the lattice resulting in a division of lattice into two parts: twinned region and untwinned region, which are symmetrical, but differently oriented. Twinning occurs when a material is permanently or plastically deformed, or during the heat treatment of certain metals. As shown in figure, PQ and RS are called twin boundaries or twin planes, which separate the un-deformed and the deformed parts of the metal lattice. The region PQRS is known as twinned zone or twinned region.



- ▶ The part of the atomic lattice deformed in the twinned region is a mirror image of the un-deformed lattice. The mechanism of twinning may thus be **described as the simple sliding of one plane of atoms over the next**; the extent of the movement of each plane in the twinned region being proportional to its distance from the twinning plane. For example, the first plane CD moves one-third of an inter-atomic distance, the second plane EF moves two-thirds of the inter-atomic distance, and the third plane SR moves an entire spacing. Notice that the atom C gets displaced to C' exactly the same distance from either side of the twin plane, i.e., $B'C' = B'A'$.
- ▶ In simple words, PC'B' in the twinned region is mirror image of PA'B' in the undeformed region. Similar is the case with respect to other atoms in the twinned region.
- ▶ Twin boundaries interfere with the slip process, and increase the strength of the metal. Movement of twin boundaries causes a metal to deform. Twinning is observed as wide bands under the microscope, and is shown in figure above.

Difference between slip and twinning

Slip

1. All atom in one block move over same distance
2. Slip appear in thin lines
3. There is little change in lattice orientation
4. Requires lower shear stress
5. More pronounced at high temperature and gradual loadings
6. Occurs in metals having more number of slip systems. Eg. Cu (FCC) (12 slip system)

Twinning

1. Different planes of atoms moves fractional distance
2. They appear in broad lines
3. Lattice orientation change in the twinned region
4. Require higher shear stress
5. More pronounced at low temperature and impact loadings.
6. Occurs in metals having less number of slip systems. Eg. Zn (HCP) (3 slip system)

Recovery, Recrystallization and Grain Growth

Recovery, recrystallization and grain growth are phenomena intimately associated with the annealing of a plastically deformed crystalline material. In crystalline materials, the density of point imperfections and dislocations increases with increasing amount of plastic deformation carried out at temperatures below the range $0.3-0.5T_m$, where T_m is the melting point in kelvin. Plastic working below $0.3-0.5T_m$ is called cold work. Point imperfections and dislocations

have strain energy associated with them. Between 1 and 10% of the energy of plastic deformation is stored in the material in the form of this strain energy. On annealing, that is, on heating the deformed material to higher temperatures and holding. The material tends to lose the extra strain energy and revert to the original condition before deformation, by the processes of recovery and recrystallization

During recovery, which takes place at low temperatures of annealing, the excess point imperfections that are created during plastic deformation are absorbed at the surface or the grain boundaries or at dislocations. Also, random dislocations of opposite sign come together and mutually annihilate each other. Dislocations of the same sign arrange themselves into lower energy configurations, such as tilt and twist boundaries. However, the decrease in the dislocation density during recovery is not substantial.

Recrystallization is the process of nucleation and growth of new, strain-free crystals, which replace all the deformed crystals of the worked material. It starts on heating to temperatures in the range of $0.3-0.5T_m$, which is above the recovery range. There is **no crystal structure change during recrystallization. The free energy change during recrystallization arises from the excess strain energy of the deformed material as compared to the undeformed material. The strain energy difference between the cold-worked and the strain-free material is known as the driving force for recrystallization. The recrystallization temperature is arbitrarily defined as that temperature at which 50% of the material recrystallizes in 1 hr.**

Some well-known empirical laws of recrystallization are:

1. The higher is the degree of deformation, the lower is the recrystallization temperature.
2. The finer is the initial grain size, the lower is the recrystallization temperature.
3. Increasing the amount of cold work and decreasing the initial grain size produce finer recrystallized grains.
4. The higher is the temperature of cold working, the less is the strain energy stored in the material. The recrystallization temperature is correspondingly higher.
5. The recrystallization rate increases exponentially with temperature

Grain growth refers to the increase in the average grain size on further annealing, after all the cold worked material has recrystallized. As a reduction in the grain boundary area per unit volume of the material occurs during grain growth, there is a decrease in the free energy of the material. Smaller grains get consumed by the larger ones. In practical applications, grain growth is usually not desirable.

The changes in the material properties due to the changes in the structure during the process of recovery, recrystallisation and grain growth are schematically shown in the figure below.

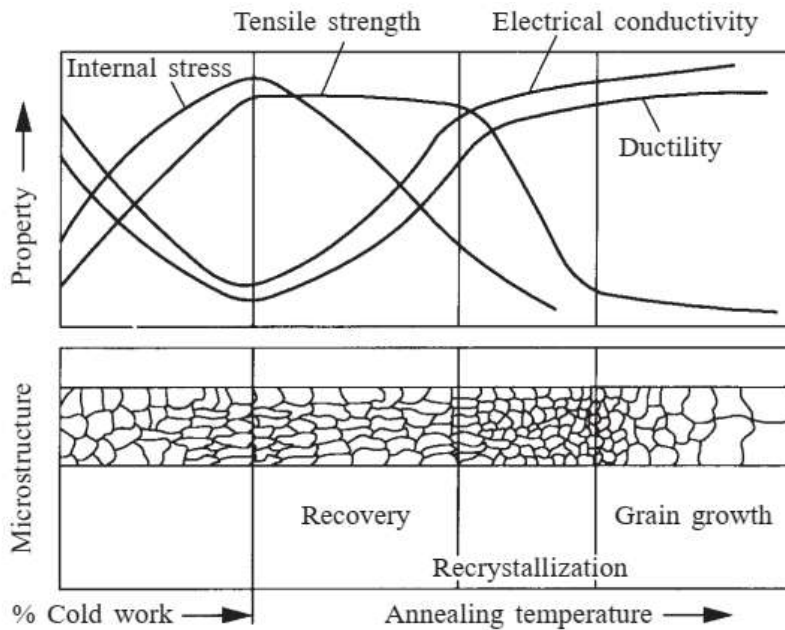


Figure: Effect of cold work, recovery, recrystallization and grain growth on some properties of crystalline materials.

The effect on mechanical and some physical properties of the phenomena discussed in this section are summarized in Fig. With increasing cold work, the tensile strength increases, but the electrical conductivity and the ductility decrease. On recovery, the electrical conductivity is mostly restored, as it depends mainly on the presence of point imperfections. On recrystallization, the tensile strength decreases and the ductility increases to the values prior to cold working. The microstructural changes are also sketched in Fig. During cold work, the grains become elongated in the direction of working. During recrystallization, new equiaxed grains form. During grain growth, these new grains increase in size, but decrease in number.

Introduction to Strengthening mechanisms

Mechanisms of Strengthening in Metals

The ease with which a metal is capable of plastic deformation is a function of dislocation mobility—that is, restricting dislocation motion leads to increased hardness and strength.

Among different properties that are resident in polycrystalline materials, we have two sets which are in opposite camps.

Strength with cost of ductility

Wear resistance – Toughness

Challenge for material engineer to balance between opposing properties3 broad mechanism through which metals can be strengthened

- Strengthening by grain size reduction
- Solid solution strengthening
- Strain hardening or work hardening
- Strain aging
- Strengthening by two-phase aggregates
- Strengthening from fine particles

- Strengthening due to point defect
- Martensite Strengthening

Strengthening by grain size reduction

The size of the grains, or average grain diameter, in a polycrystalline metal influences the mechanical properties. Adjacent grains normally have different crystallographic orientations and, of course, a common grain boundary. During plastic deformation, slip or dislocation motion must take place across this common boundary—say, from grain A to grain B in Figure below. The grain boundary acts as a barrier to dislocation motion for two reasons:

1. Because the two grains are of different orientations, a dislocation passing into grain B must change its direction of motion; this becomes more difficult as the crystallographic misorientation increases.
2. The atomic disorder within a grain boundary region results in a discontinuity of slip planes from one grain into the other.

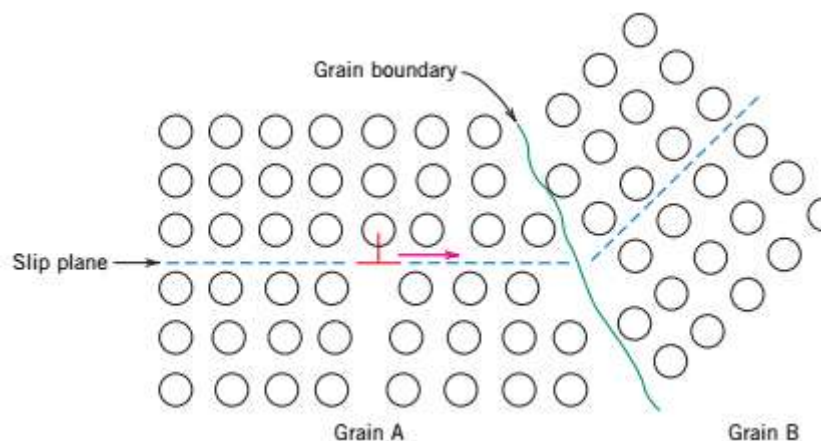


Figure: The motion of a dislocation as it encounters a grain boundary, illustrating how the boundary acts as a barrier to continued slip. Slip planes are discontinuous and change directions across the boundary

It should be mentioned that, for high-angle grain boundaries, it may not be the case that dislocations traverse grain boundaries during deformation; rather, dislocations tend to “pile up” (or back up) at grain boundaries which further strengthens the material.

A fine-grained material (one that has small grains) is harder and stronger than one that is coarse grained because the former has a greater total grain boundary area to impede dislocation motion. For many materials, the yield strength σ_y varies with grain size according to

$$\sigma_y = \sigma_0 + k_y d^{-1/2}$$

In this expression, termed the Hall–Petch equation, d is the average grain diameter, and σ_0 and k_y are constants for a particular material. The equation is not valid for both very large (i.e., coarse) grain and extremely fine grain polycrystalline materials. It should also be mentioned that grain size reduction improves not only the strength, but also the toughness of many alloy

Solid-solution strengthening

A technique to strengthen and harden metals is alloying with impurity atoms that go into either substitutional or interstitial solid solution. Accordingly, this is called solid-solution strengthening. Increasing the concentration of the impurity results in an attendant increase in tensile and yield strengths. Solid-solution strengthening results from lattice strain interactions between impurity atoms and dislocations; these interactions produce a decrease in dislocation mobility.

Strain hardening

Strain hardening is the enhancement in strength (and decrease of ductility) of a metal as it is deformed plastically. The Degree of plastic deformation may be expressed as percent cold work, which depends on original and deformed cross-sectional areas as described by the equation:

$$\%CW = \left(\frac{A_0 - A_d}{A_0} \right) \times 100$$

Yield strength, tensile strength, and hardness of a metal increase with increasing percent cold work; ductility decreases. During plastic deformation, dislocation density increases, the average distance between adjacent dislocations decreases, and—because dislocation–dislocation strain field interactions, are, on average, repulsive—dislocation mobility becomes more restricted; thus, the metal becomes harder and stronger.

Strain aging

It is associated with the yield point phenomenon. The strength of the metal is increased and its ductility is decreased on heating to a relatively low temperature after cold working. Consider the behaviour of a low carbon steel as shown in the below figure.

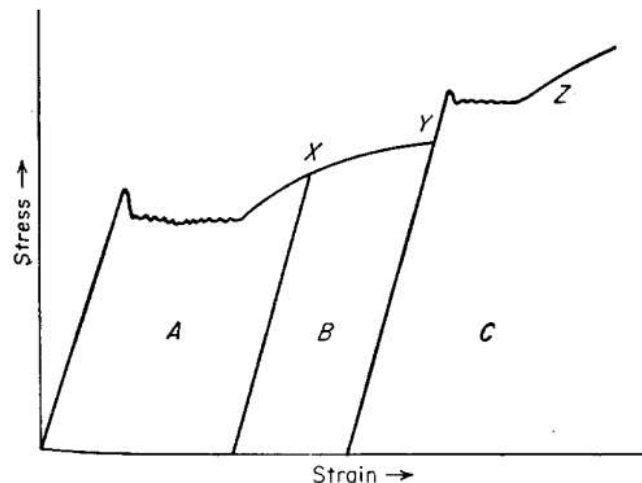


Figure: stress strain behaviour of low carbon steel

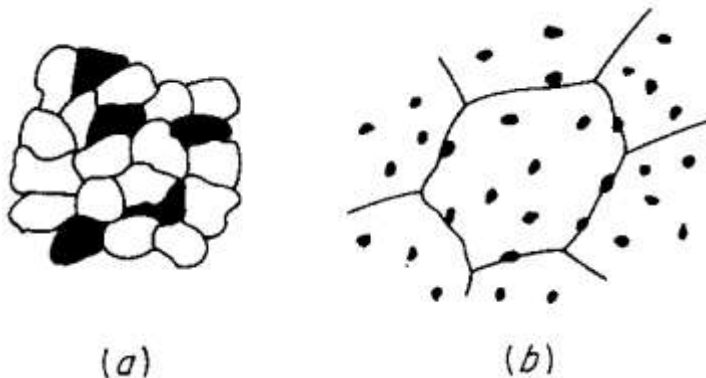
Region A shows the stress-strain curve of the steel stressed beyond the yield point to a strain corresponding to point X. the specimen is then unloaded and rested for few time without appreciable rest time and then reloaded to a point corresponding to point Y (region B). The yield point phenomenon does not occur. Now it is unloaded and rested for several days or kept for few hours at 127 °C and then cooled to room temperature and loaded again the yield point

reappears. Moreover, the yield point appears at higher value of stress indicating strengthening of the original metal (region C).

Strengthening by two-phase aggregates

There are of two types:

1. Containing second phased particle is of the size of the grain.
2. Dispersed two-phase structure-in this Each particle is completely surrounded by a matrix of single orientation



(a) Aggregate structure and (b) dispersed structure

In dispersion hardening the hard second phase particles are mixed with powder form of matrix material, consolidated and processed via powder metallurgy technique. The second phase has no solubility at high temperature as well as low temperature. There is no atomic matching between the lattice of the precipitate and the matrix. Used in materials that must display high thermal stability. Alloys in this category are resistant to recrystallization and grain growth compared to single phase alloys.

Strengthening from fine particles

Precipitation hardening or age hardening is produced by solution heat treatment and quenching an alloy. In this a second phase is in solid solution at the elevated temperature but precipitates upon quenching and aging at lower temperature. For this to occur the second phase must be soluble at elevated temperature but must show decreasing solubility with decreasing temperature. There is atomic matching between the lattice of the precipitate and the matrix. The precipitates formed obstruct the movement of dislocation thereby strengthening the alloy.

Strengthening due to point defect

Vacancies that are produced during hardening and interstitial defects cause strengthening. It is well known that the number of vacancies in metals increase considerable as the temperature increased. The equilibrium number of vacancies N_v for a given quantity of material (usually per meter cubed) depends on and increases with temperature according to the equation:

$$N_v = N \exp\left(-\frac{Q_v}{kT}\right)$$

Where N is the total number of atomic sites (most commonly per cubic meter), Q_v is the energy required for the formation of a vacancy (J/mol or eV/atom), T is the absolute temperature in kelvins, and k is the gas or Boltzmann's constant. The value of k is 1.38×10^{-23} J/atom · K,

or 8.62×10^{-5} eV/atom · K. aluminium metal that generally does not exhibit a sharp yield point showed one due to high energy radiation bombardment on it which caused interstitial defects. Ex: For Al single crystal the critically resolved shear stress increased from 5 to 50 kPa due to the presence of quenched in vacancies.

Martensite Strengthening

Martensite transformation in carbon steels increase the strength by producing strong barriers to dislocation motion. Martensite is obtained on fast cooling from the austenite phase in steels. It occurs by diffusionless shear-type transformation during quenching of steels.

Heat treatment

Annealing

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

Annealing is a rather generalized term. Annealing consists of heating a metal to a specific temperature and then cooling at a rate that will produce a refined microstructure. The rate of cooling is generally slow. Annealing is most often used to soften a metal for cold working, to improve machinability, or to enhance properties like electrical conductivity.

Ferrous alloys are usually either "full annealed" or "process annealed." Full annealing requires very slow cooling rates, in order to form coarse pearlite. In process annealing, the cooling rate may be faster; up to, and including normalizing. The main goal of process annealing is to produce a uniform microstructure. Non-ferrous alloys are often subjected to a variety of annealing techniques, including "recrystallization annealing," "partial annealing," "full annealing," and "final annealing." Not all annealing techniques involve recrystallization, such as stress relieving.

Purpose of annealing

The purpose of annealing is to achieve one or more of the following objectives

- To relieve internal stresses induced by some primary processes
- To remove coarseness of grains
- Soften the metal so as to improve mach inability, formability and sometimes control magnetic properties

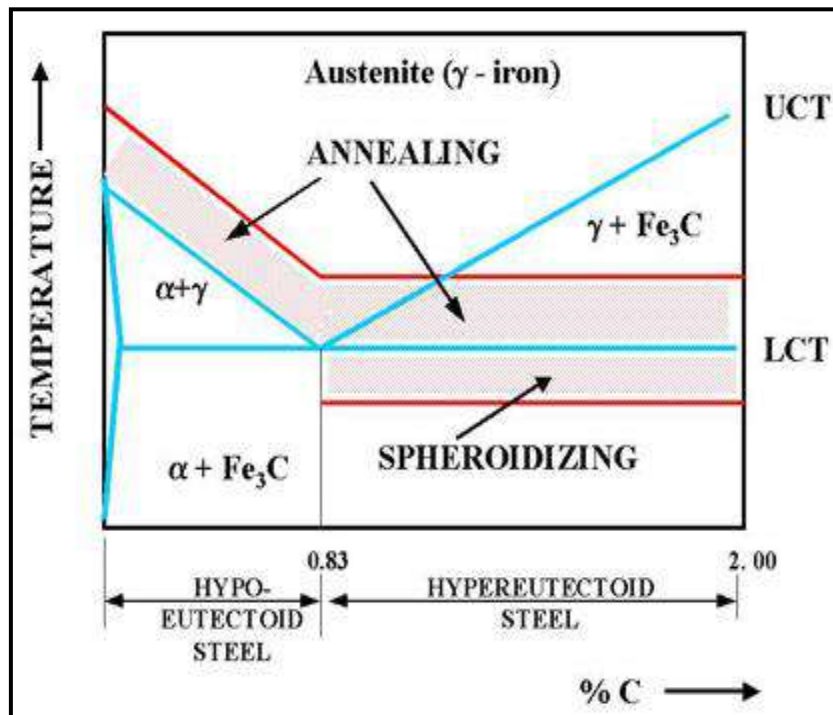
Types of annealing

- Full annealing
- Process annealing
- Step relief annealing
- Spheroid zing annealing

Full annealing

The term full annealing is used for the treatment that involves heating steel to its austenitic state before subjecting it to very slow cooling. Full annealing process consists of three steps. First step is heating the steel component to above A_3 (upper critical temperature for ferrite) temperature for hypoeutectoid steels and above A_1 (lower critical temperature) temperature for hypereutectoid steels by $30-50^{\circ}\text{C}$ the terms α , γ and Fe_3C refer to ferrite, austenite and cementite phases.

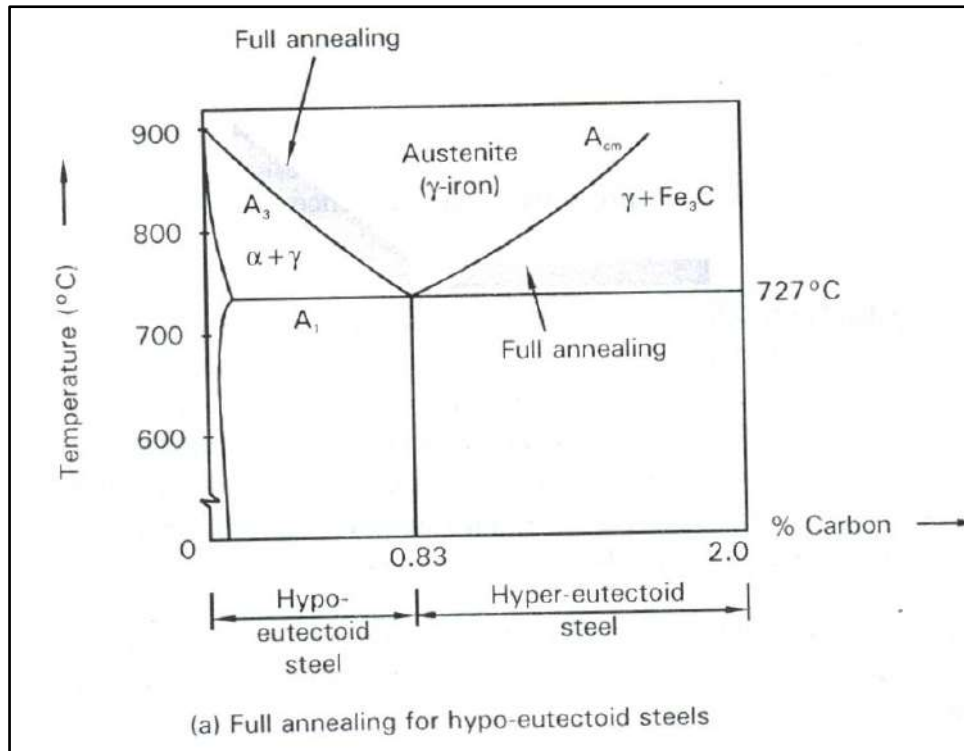
The second step is holding the steel component at this temperature for a definite holding (soaking) period of at least 20 minutes per cm of the thick section to assure equalization of temperature throughout the cross-section of the component and complete austenization. Final step is to cool the hot steel component to room temperature slowly in the furnace, which is also called as *furnace cooling*. The *full annealing* is used to relieve the internal stresses induced due to cold working, welding, etc, to reduce hardness and increase ductility, to refine the grain structure, to make the material homogenous in respect of chemical composition, to increase uniformity of phase distribution, and to increase machinability



Schematic representation of annealing operation

Purpose

To relieve internal stresses, improve ductility and grain grain refinement



Process

The process consists of heating the metal above the upper critical temperature holding it there for considerable time so that it becomes completely austenitic and then allowing it to cool slowly in the furnace. slow cooling associated with full annealing enables the austenite to decompose to form;

A pearlite + ferrite structure in case of hypo eutectoid steels

A pearlite + cementite structure in hyper eutectoid steels

The phase recrystallize completely in this process and the structure is refined. The metal gets softened with improved mechanical properties.

Holding or soaking time

Holding time depends on the thickness or diameter of the component to be heat treated

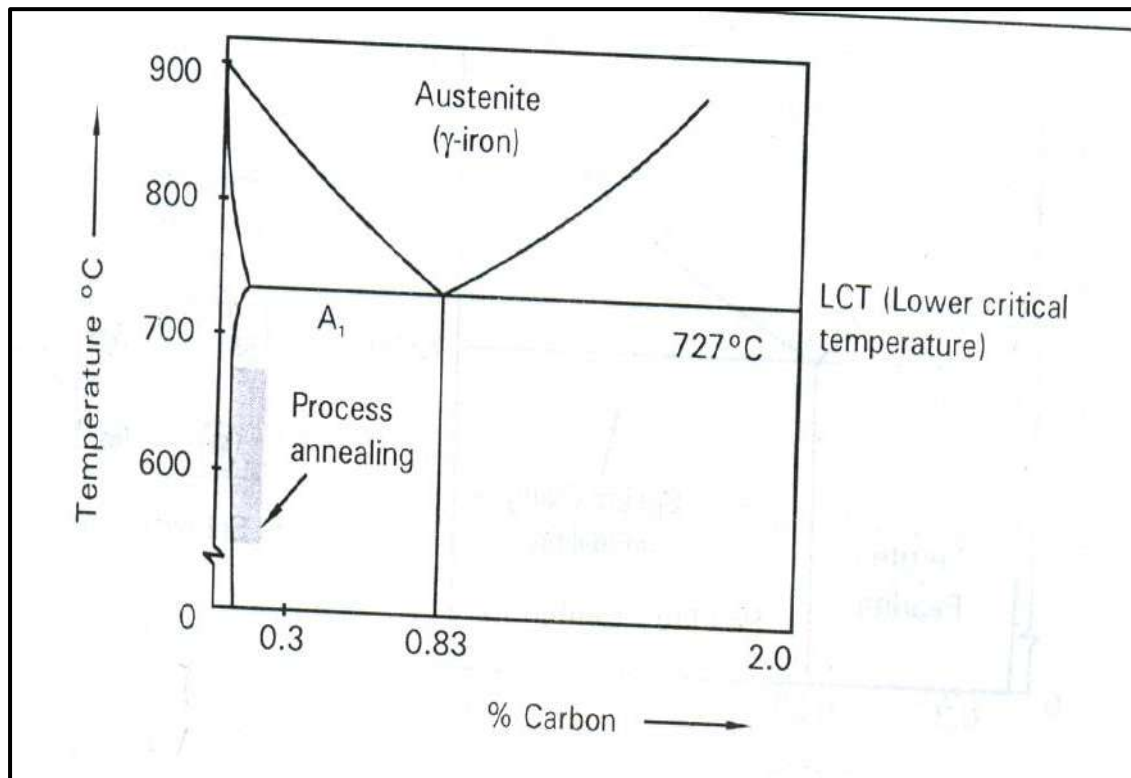
Applications

Full annealing is used for low and medium carbon steels that will be machined or experience extensive plastic deformation during forming operation.

Disadvantages

Process is time consuming due to slow cooling rates in the furnace

Process Annealing



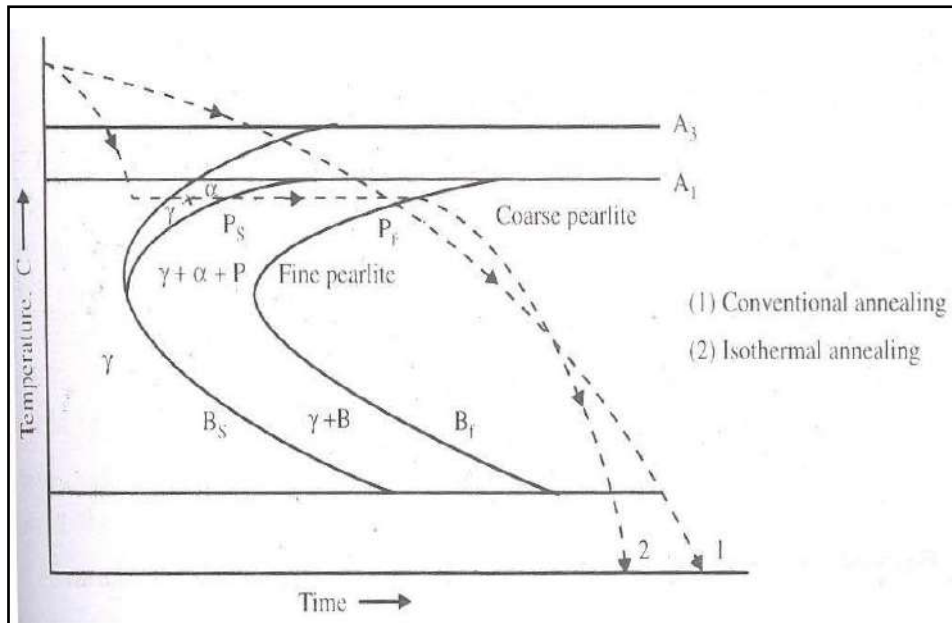
Isothermal annealing

Isothermal annealing consists of four steps. The *first step* is heating the steel components similar as in the case of full annealing. The *second step* is slightly fast cooling from the usual austenitizing temperature to a constant temperature just below A_1 . The *third step* is to hold at this reduced temperature for sufficient soaking period for the completion of transformation and the *final step* involves cooling the steel component to room temperature in air. Figure depicts the heat treatment cycles of full annealing and isothermal annealing. The terms α , γ , P, P_S and P_F refer to ferrite, austenite, pearlite, pearlite starting and pearlite finish, respectively.

Isothermal annealing has distinct advantages over full annealing which are given below.

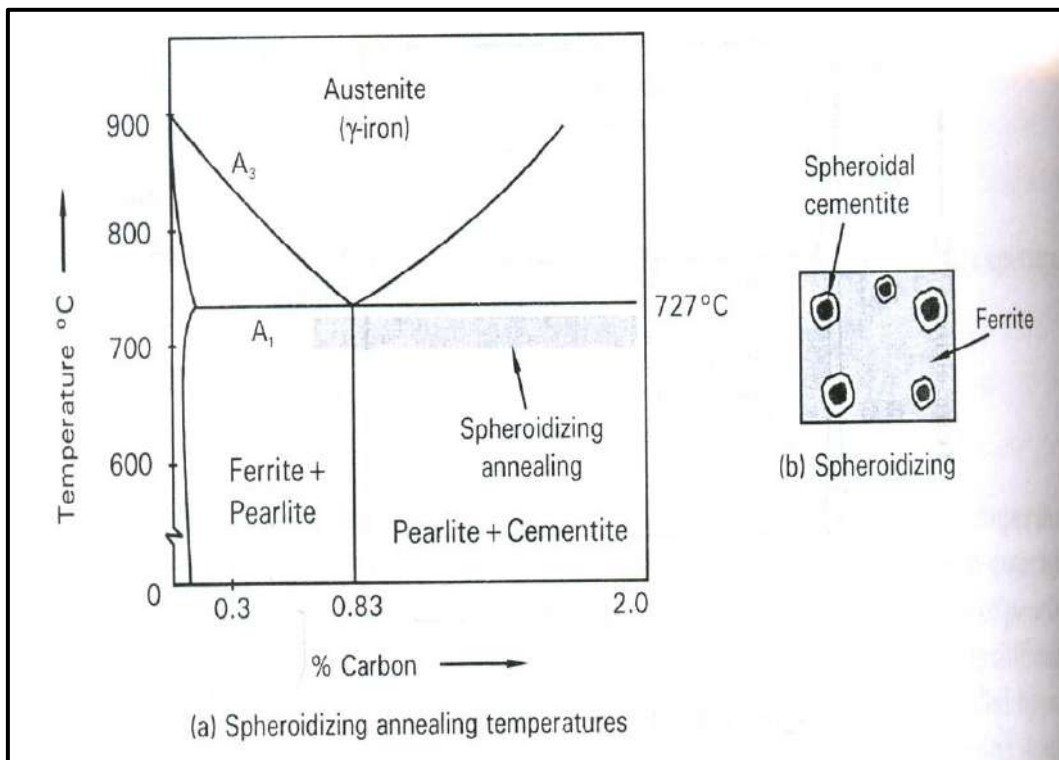
1. Reduced annealing time, especially for alloy steels which need very slow cooling to obtain the required reduction in hardness by the full annealing.
2. More homogeneity in structure is obtained as the transformation occurs at the same time throughout the cross section.
3. Improved machinability and surface finish is obtained after machining as compared to that of the full annealed components.

Isothermal annealing is primarily used for medium carbon, high carbon and some of the alloy steels to improve their machinability.



Heat treatment cycles of full annealing and isothermal annealing

Spheroidizing annealing



Spheroidise annealing is one of the variant of the annealing process that produces typical microstructure consisting of the globules (spheroid) of cementite or carbides in the matrix of ferrite. The following methods are used for spheroidise annealing Holding the steel component at just below the lower critical temperature (A_1) transforms the pearlite to globular cementite particles. But this process is very slow and requires more time for obtaining spheroidised structure. In this method, the thermal cycling in the narrow temperature range around A_1 transforms cementite lamellae from pearlite to spheroidal. *Figure 4.7.4* depicts a typical heat

treatment cycle to produce spheroidised structure. During heating above A_1 , cementite or carbides try to dissolve and during cooling they try to re-form. This repeated action spheroidises the carbide particles. Spheroidised structures are softer than the fully annealed structures and have excellent machinability. This heat treatment is utilized to high carbon and air hardened alloy steels to soften them and to increase machinability, and to reduce the decarburization while hardening of thin sections such as safety razor blades and needles.

Recrystallization annealing

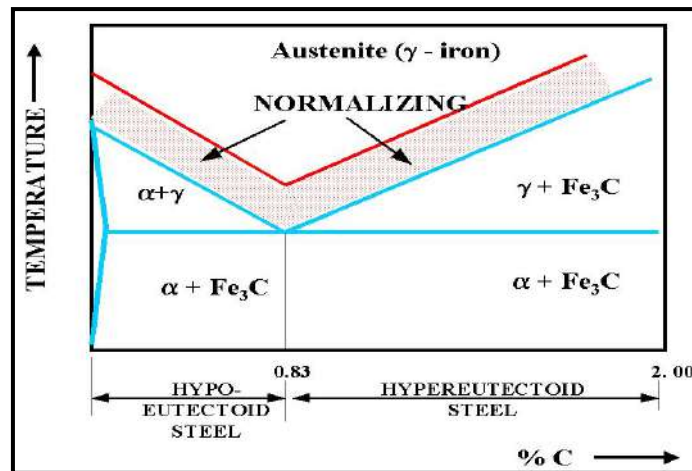
Recrystallization annealing process consists of heating a steel component below A_1 temperature i.e. at temperature between 625°C and 675°C (recrystallization temperature range of steel), holding at this temperature and subsequent cooling. This type of annealing is applied either before cold working or as an intermediate operation to remove strain hardening between multi-step cold working operations. In certain case, recrystallization annealing may also be applied as final heat treatment. The cold worked ferrite recrystallizes and cementite tries to spheroidise during this annealing process. Recrystallization annealing relieves the internal stresses in the cold worked steels and weldments, and improves the ductility and softness of the steel. Refinement in grain size is also possible by the control of degree of cold work prior to annealing or by control of annealing temperature and time.

Stress relief annealing

Stress relief annealing process consists of three steps. The *first step* is heating the cold worked steel to a temperature between 500°C and 550°C i.e. below its recrystallization temperature. The *second step* involves holding the steel component at this temperature for 1-2 hours. The *final step* is to cool the steel component to room temperature in air. The *stress relief annealing* partly relieves the internal stress in cold worked steels without loss of strength and hardness i.e. without change in the microstructure. It reduces the risk of distortion while machining, and increases corrosion resistance. Since only low carbon steels can be cold worked, the process is applicable to hypoeutectoid steels containing less than 0.4% carbon. This annealing process is also used on components to relieve internal stresses developed from rapid cooling and phase changes.

Normalizing

Normalizing process consists of three steps. The *first step* involves heating the steel component above the $A_{3\text{ cm}}$ temperature for hypoeutectoid steels and above A (upper critical temperature for cementite) temperature for hypereutectoid steels by 30°C to 50°C . The *second step* involves holding the steel component long enough at this temperature for homogeneous austenization. The final step involves cooling the hot steel component to room temperature in still air. Due to air cooling, normalized components show slightly different structure and properties than annealed components.



The properties of normalized components are not much different from those of annealed components. However, normalizing takes less time and is more convenient and economical than annealing and hence is a more common heat treatment in industries. Normalizing is used for high-carbon (hypereutectoid) steels to eliminate the cementite network that may develop upon slow cooling in the temperature range from point A_{cm} to point A_1 . Normalizing is also used to relieve internal stresses induced by heat treating, welding, casting, forging, forming, or machining. Normalizing also improves the ductility without reducing the hardness and strength

Hardening

Different techniques to improve the hardness of the steels are conventional hardening, martempering and austempering

Conventional hardening

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

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

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

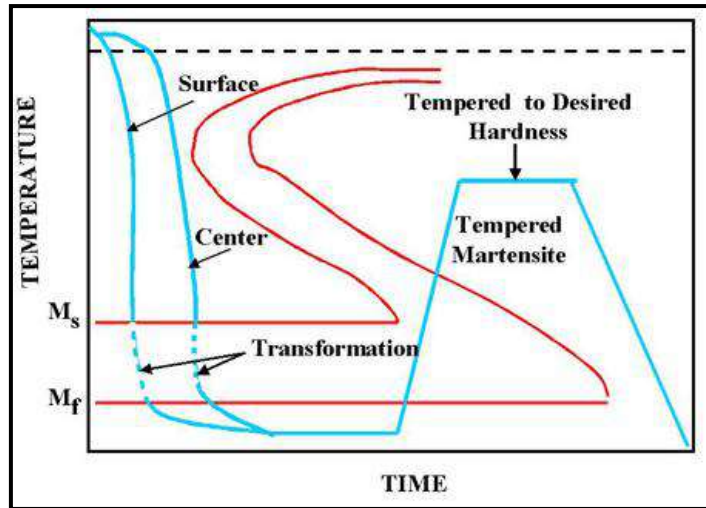


Fig Heat hardening process treatment cycle for conventional

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

Martempering (marquenching)

Martempering process overcomes the limitation of the conventional hardening process. *Figure* depicts the martempering process. This process follows interrupted quenching operation. In other words, the cooling is stopped at a point above the martensite transformation region to allow sufficient time for the center to cool to the temperature as the surface. Further cooling is continued through the martensite region, followed by the usual tempering. In this process, the transformation of austenite to martensite takes place at the same time throughout the structure of the metal part.

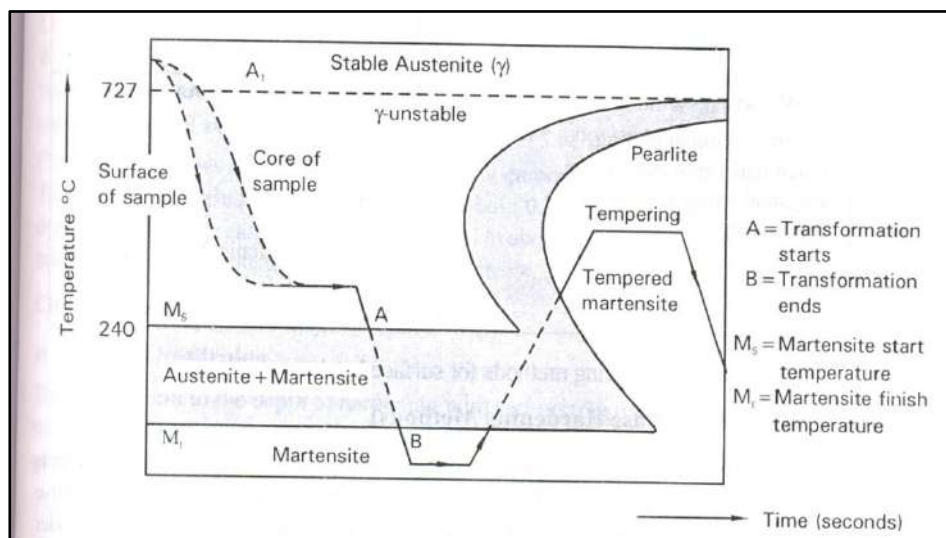
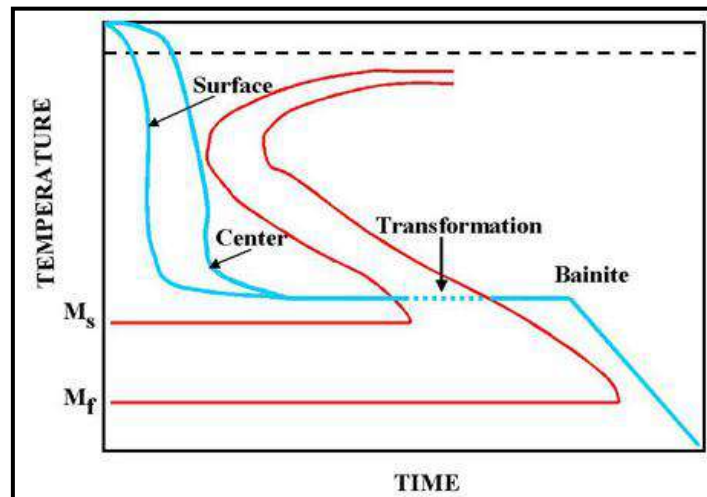


Fig Martempering followed by tempering

Austempering

This process is also used to overcome the limitation of the conventional hardening process. *Figure* depicts the austempering process. Here the quench is interrupted at a higher temperature than for martempering to allow the metal at the center of the part to reach the same temperature as the surface. By maintaining that temperature, both the center and surface are allowed to transform to bainite and are then cooled to room temperature. Austempering causes less distortion and cracking than that in the case of martempering and avoids the tempering operation. Austempering also improves the impact toughness and the ductility of the metal than that in the case of martempering and conventional hardening.



Heat treatment cycle for austempering

Tempering

The hardened steel is not readily suitable for engineering applications. It possesses following three drawbacks.

- Martensite obtained after hardening is extremely brittle and will result in failure of engineering components by cracking.
- Formation of martensite from austenite by quenching produces high internal stresses in the hardened steel.
- Structures obtained after hardening consists of martensite and retained austenite. Both these phases are metastable and will change to stable phases with time which subsequently results in change in dimensions and properties of the steel in service.

Tempering helps in reduce these problems. Tempering is achieved by heating hardened steel to a temperature below A_1 , which is in the range of 100°C to 680°C , hold the component at this temperature for a soaking period of 1 to 2 hours (can be increases up to 4 hours for large sections and alloy steels), and subsequently cooling back to room temperature. The tempering temperature is decided based on the type of steel. Highly alloyed tool steels are tempered in the range of 500°C - 600°C . Low alloy construction steels are tempered above 400°C to get a good combination of strength and ductility. Spring steels are tempered between 300°C - 400°C to get the desired properties. *Figure* depicts the influence of tempering temperature on the properties of steel. It is observed that the increase in the tempering temperature decreases the hardness and internal stresses while increases the toughness

The variation in the properties of the annealed and normalized components

Annealed

- Less hardness, tensile strength and toughness.
- Pearlite is coarse and usually gets resolved by the optical microscope.
- Grain size distribution is more uniform.
- Internal stresses are least.

Normalised

- Slightly more hardness, tensile strength and toughness.
- Pearlite is fine and usually appears unresolved with optical microscope.
- Grain size distribution is slightly less uniform.
- Internal stresses are slightly more.

Nitriding

Nitriding is carried out in a ferritic region below 590°C. So there is no phase change after nitriding. Before nitriding, proper heat treatment should be given to steel components (hardening by heating to 930°C and quenching in oil, then tempering at 650°C) to develop bainitic structure, which increases the strength and toughness of the core. All machining and grinding operations are finished before nitriding. The portions which are not to be nitrided are covered by thin coating of tin deposited by electrolysis. The coating checks diffusion of nitrogen due to its surface tension. Anhydrous ammonia gas is passed into the furnace at about 550°C, where it dissociates into nascent nitrogen and hydrogen. Thus,



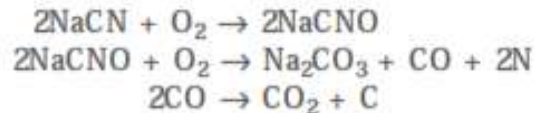
The treatment time varies from 21 hours to 100 hours. Actually, it depends on the desired case depth and size of the steel parts. After nitriding, the steel part is allowed to cool in furnace itself in the presence of ammonia. The furnace container is made of heat resisting alloy steel. The time required for a case depth of 0.5 mm is about 100 hours.

The hardness achieved at the surface of steel varies from 900 VHN to 1100 VHN. The hard layer so formed imparts good wear resistance, hot hardness and corrosion resistance.

During nitriding process, a white layer of Fe_4N (γ' nitride) and Fe_2N (ϵ nitride) form on the outer layer of surface. This layer is problematic as it is very brittle and tends to crack. Formation of this layer is caused by high nitrogen potential. So this can be avoided by keeping nitrogen potential at the desired level. White layer should be removed from component by mechanical polishing or by boost diffusion techniques.

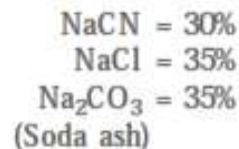
Cyaniding

In this process, the parts to be heat treated are immersed in a liquid bath (at 800-960°C) of NaCN with the concentration varying between 25% and 90%. The measured amount of air is passed through the molten bath. NaCN reacts with the oxygen in the air and is oxidized. The basic reactions in the bath are



Carbon and nitrogen so formed in atomic form diffuse into the steel and give thin wear resistant layer of the carbonitride ϵ -phase. Usually, this process requires 30-90 minutes for completion.

For obtaining case depth from 0.5 mm to 2 mm, the process is carried out at higher temperature (950°C) in a bath containing 8% NaCN, 82% BaCl₂ and 10% NaCl. This process takes 1.5-6 hours for completion. For case depth from 0.13 mm to 0.35 mm, the bath is maintained at 850°C with the following concentration:



The higher the temperature at a given cyanide level, the higher is the percentage of carbon diffusion (up to 0.8-1.2% C) into the surface of steel as compared to nitrogen (0.2-0.3%). After cyaniding, the pieces are taken out and are quenched in water or oil. For thick sections, mineral oil is preferred for quenching. The final operation is low temperature tempering. After heat treatment, the case has a hardness of 850 VHN. This process is less time consuming. Because of the high heat transfer coefficient in liquid bath and uniform bath temperature, distortion of pieces is less. This process is not suitable for hardening those parts which are subjected to shock, fatigue and impact because nitrogen addition has adverse effects on such properties of steels.

Induction hardening

Induction hardening is a surface hardening technique in which the surface of the metal is heated very quickly, using a no-contact method of induction heating. The alloy is then quenched, producing a martensite transformation at the surface while leaving the underlying metal unchanged. This creates a very hard, wear resistant surface while maintaining the proper toughness in the majority of the object. Crankshaft journals are a good example of an induction hardened surface.

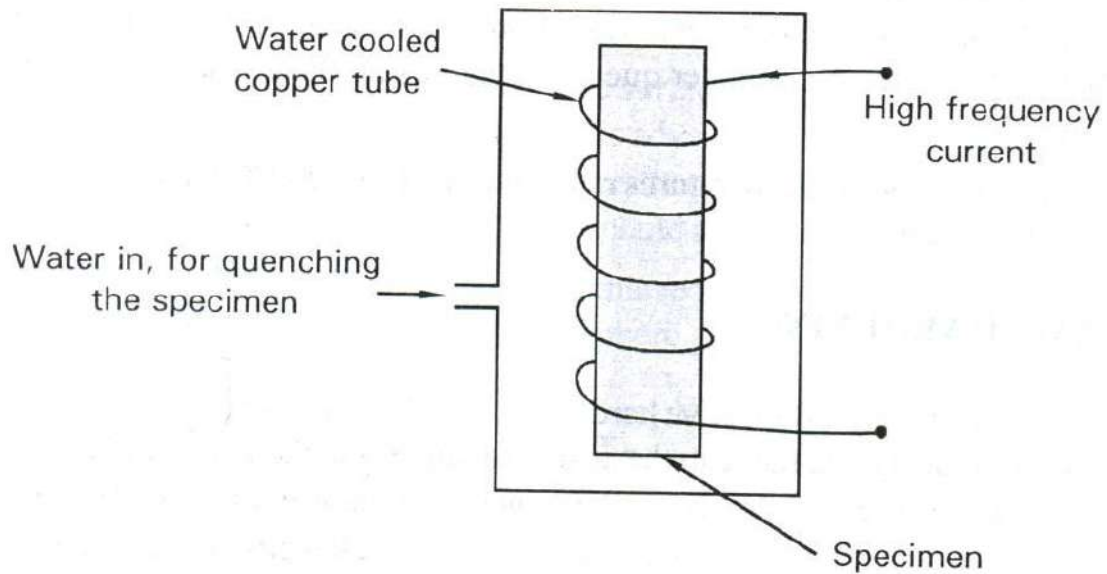


Fig. Induction Hardening

Flame hardening

Flame hardening is the simplest form of surface hardening heat treatment. This process consists of heating the large work-piece, such as crank shaft, axle, large gear, cam, bending roller, or any other complicated cross-section, by an oxy-acetylene or oxy-fuel blow pipe, followed by spraying of jet of water as coolant. After hardening, reheating of the parts is carried out in furnace or oil bath at about 180–200°C for stress relieving. Such a treatment does not appreciably reduce the hardness at the surface. Hardness in flame hardened steel is due to martensitic and lower bainitic structure.

The carbon content required for flame hardening varies from 0.3 to 0.6%. normally a case depth of 3mm can be achieved. High rate of heating is necessary for thin cases with proper control in time and position of flame.

There are four different methods which are used in general for flame hardening: (i) stationary, (ii) progressive, (iii) spinning, and (iv) progressive-spinning.

In the first, both burner and work-piece are stationary. Progressive hardening is carried out by using a burner combined with a waterspray, as shown in Figure 9.1(a). In this case, the burner moves over the large stationary work-piece. This is followed by quenching. In the spinning method, the work-piece is rotated, while the burner remains stationary. After heating, the flame is removed and quenching is carried out by a water jet. In the progressive-spinning method, the burner moves over a rotating work-piece [see Figure 9.1(b)]. In all the cases, rapid quenching is carried out after heating. There is little scaling, decarburization, or distortion in flame hardening. Since the heating and cooling are very fast, the core remains unaffected.

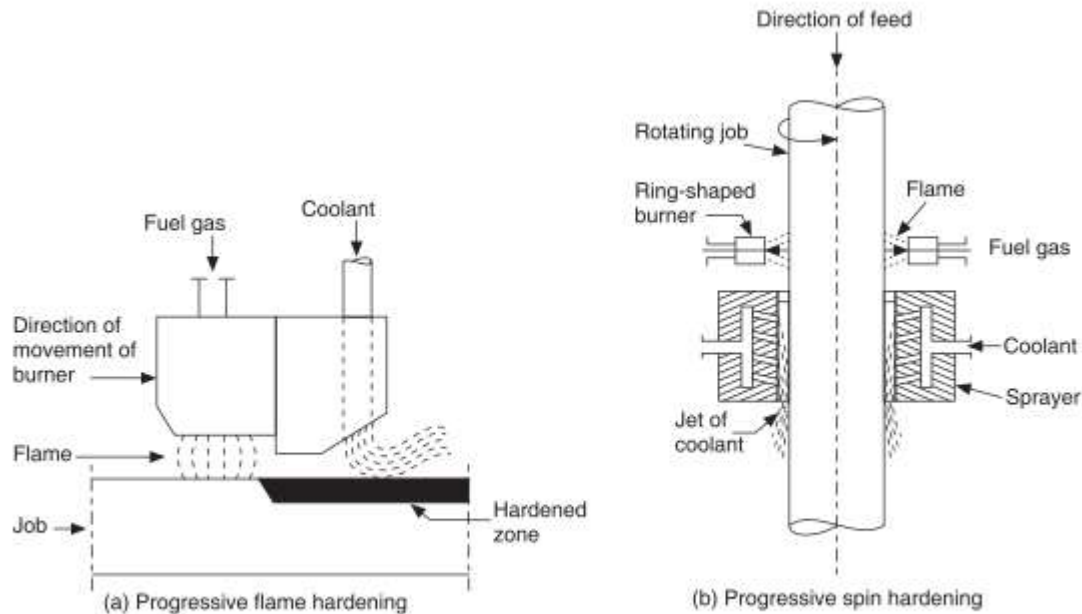


Figure 9.1 Schematic diagram illustrating two principles,

Recent advances in heat treat technology

Electron beam hardening

This process is used for hardening those components which cannot be induction hardened because of associated distortion. Automatic transmission clutch cams (SAE 5060 steel) are hardened by this process. The work-piece is kept in vacuum at 0.06 m bar pressure. Electron beam is focused on the work-piece to heat the surface. In the beginning, energy input is kept high. With time, power input is reduced as the component gets heated up. This is done to avoid melting. Normally, case depth up to 0.75 mm can be achieved by this method. A mini-computer is used to control voltage, current, beam, dwell time and focus.

Laser Hardening

Laser beams are also used for surface hardening treatment. Since these have very high intensity, they may melt the work-piece when they are used at such high intensity. Therefore, a lens is used to reduce the intensity by producing a defocused spot or scans from 1–25 mm wide. A laser beam of 1 kW produces a circular spot whose diameter may vary from 0.50 mm to 0.25 mm. Industrial lasers up to 20 kW are now available. Case depth of about 0.75 mm is obtained by self quenching. The depth of hardening is governed by both time and energy density. In laser hardening process, less time is required than in induction and flame hardening processes, and the effect of heat on the surrounding surface is less, thus leading to less distortion.

Toyota Diffusion (TD) Process

In this the carbide forming elements such as vanadium and niobium are diffused into the steel using a salt bath maintained at about 1000 °C. carbide layer of 5-12µm very high hardness, about 3000 HVN are produced. Mainly meant to surface harden large automotive press tools by Toyota. The carbide forming elements are introduced into the salt bath in their ferro alloy form. After salt bath treatment, the components are quenched and tempered. Superior wear, seizure resistance and reduced lubrication requirements for the components developed through the TD process are noteworthy properties obtained. Screws, shaft, bushes and blades can also be subjected to the TD process.

TTT diagram/ Time-Temperature-Transformation curves/ isothermal transformation curves.

These diagrams give the kinetics of austenite transformation at a given temperature with progress of time. As the transformation of austenite occurs at constant temperatures during the study it is also termed as isothermal transformation curves.

To obtain the diagram a number of small pieces of the steel under consideration are taken. These are austenitised by heating above their austenite transformation temperature and are held at that temperature to homogenize using a salt bath. The samples are then quickly transferred into another salt bath at sub critical temperature (particular temperature below the eutectoid temperature) without much time delay to study kinetics of transformation. The samples are taken one by one from the bath at different intervals of time and quenched immediately. The quenching results in formation of martensite from untransformed austenite. By this method the amount of austenite transformed austenite can be determined as a function of time at a constant temperature. The result of this exercise is an isothermal transformation plot of austenite into pearlite at a constant temperature (Figure 1).

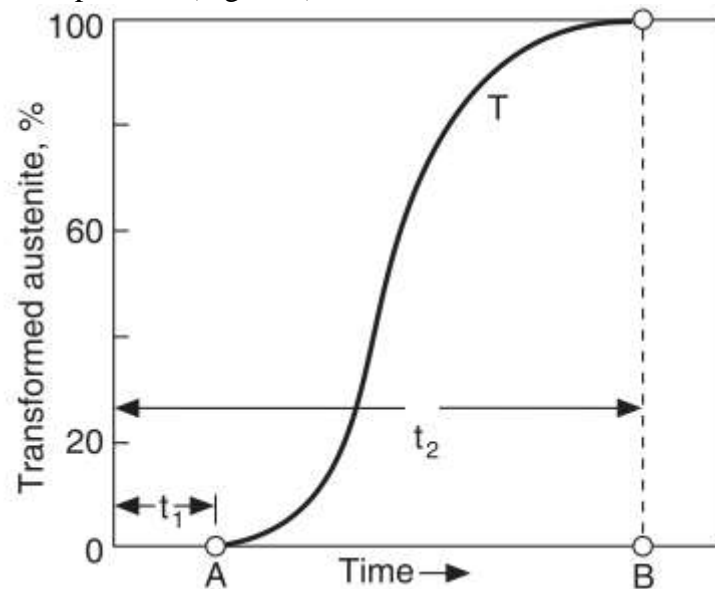


Figure 1: Isothermal Transformation of austenite into pearlite at constant temperature.

The sigmoidal curve here shows the progress of transformation with time from point A, 0% transformation to point B, 100% transformation. t_1 is the incubation time which refers to the stability of the parent austenite. As time and temperature both influence the kinetics of transformation of austenite the above figure has limited use. To accommodate the effect of temperature along with time, the above process is repeated several times at different temperatures to obtain a number of isothermal plots of transformation as shown in Figure 2. The time and transformation data from individual curves of Figure 2 are then translated onto another plot that accommodates temperatures to provide a holistic view of transformation kinetics as shown in Figure 3. This plot is called the TTT plot. The X-axis in all the three plots is in Logarithmic scale. Hence these plots are semi-log plots.

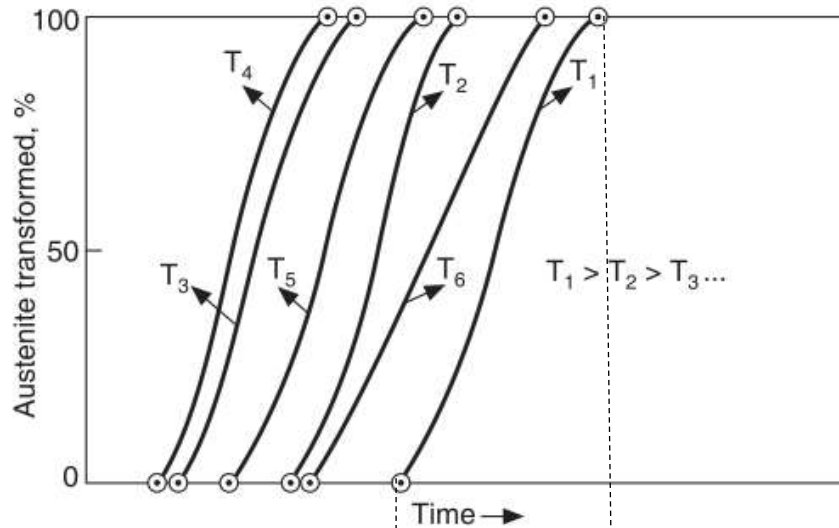


Figure 2: Isothermal Transformation of austenite into pearlite at different temperatures.

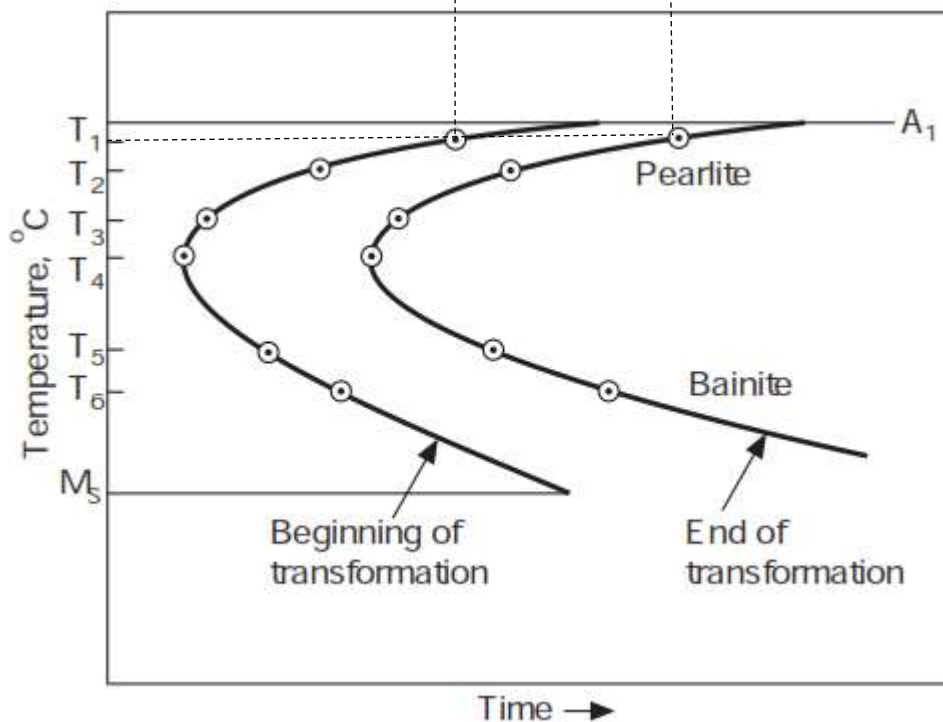


Figure 3: TTT diagram for the transformation of austenite for steel.

The transformation rate depends on the nucleation and diffusion rates. At higher temperatures close to the austenitic temperature the rate of diffusion is higher while nucleation is lower while at lower temperatures, close to the martensite start temperature, the rate of diffusion is low and nucleation is high. In between the extremities the condition is such that the rate of nucleation and diffusion match each other. At this balance the nose of the TTT, the point on the curve that is nearest to the Y-axis is obtained. The stability of the austenite also varies with the transformation temperature. Incubation and transformation time decreases as the temperature is lowered from the A1 temperature. However, after T₄ the trend reverses, due to reduction in the rate of diffusion. [M_s represents the temperature at which martensite formation starts.]

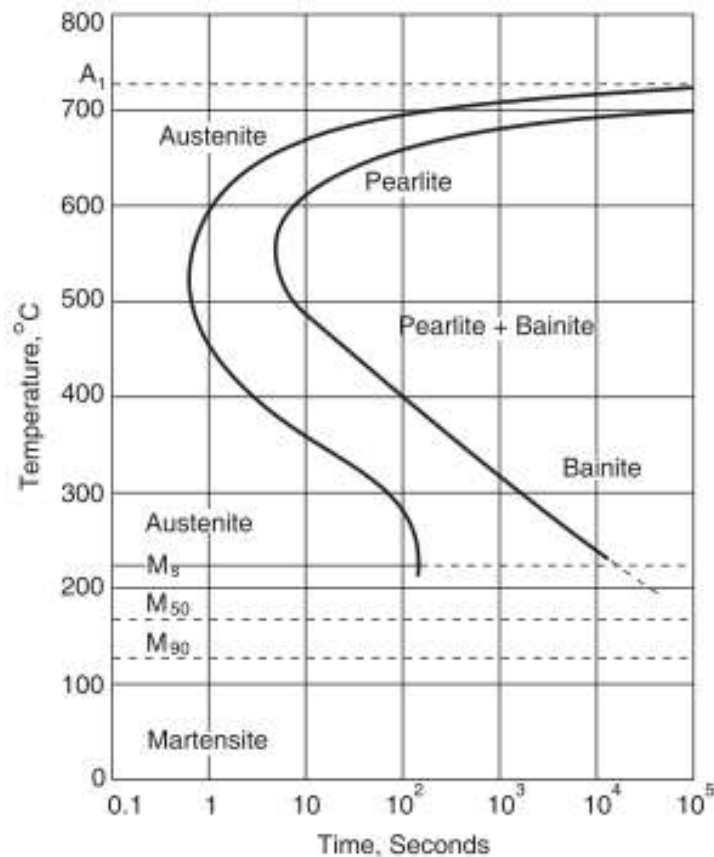


Figure 4.13 TTT diagram for eutectoid steel.

Figures 4.14 and 4.15 represent the TTT diagrams for hypoeutectoid and hypereutectoid steels, respectively. A common feature of these TTT diagrams is that proeutectoid phase (ferrite for hypoeutectoid and cementite for hypereutectoid steels) separates out in upper temperature region. For hypoeutectoid steels, ferrite starts separating out from the austenite as soon as austenite is cooled below the upper critical temperature (A_3). The amount of proeutectoid ferrite decreases as austenite is undercooled more and more below the upper critical temperature. After a certain degree of undercooling, austenite will transform directly to pearlite. On further cooling, there will be no surplus ferrite.

Similarly, cementite is separated out in hypereutectoid steels from austenite on cooling below the upper critical temperature (A_{cm}). The amount of cementite decreases with increased degree of supercooling and finally reduces to zero when austenite is cooled below a particular temperature.

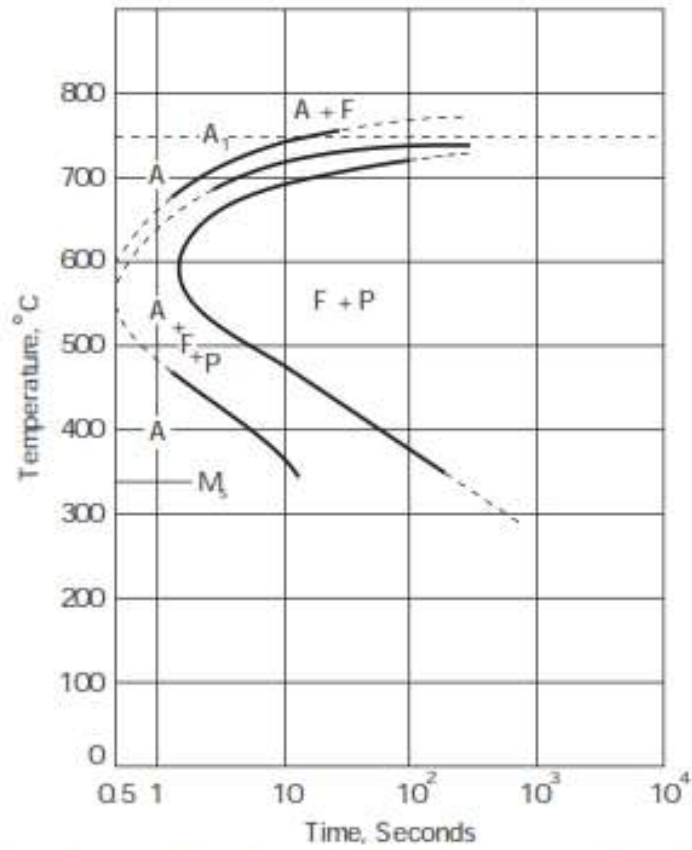


Figure 4.14 TTT diagram for hypoeutectoid steel.

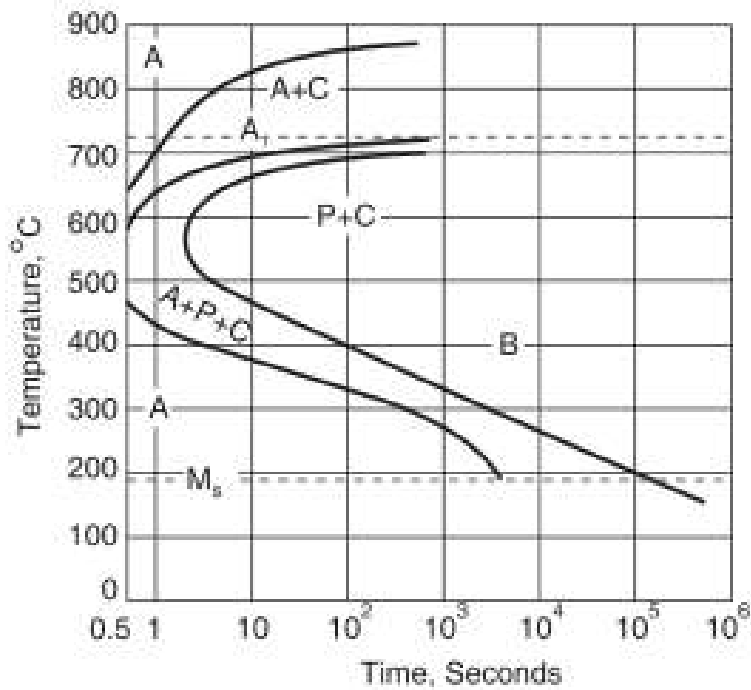


Figure 4.15 TTT diagram for hypereutectoid steel.