

Unit III

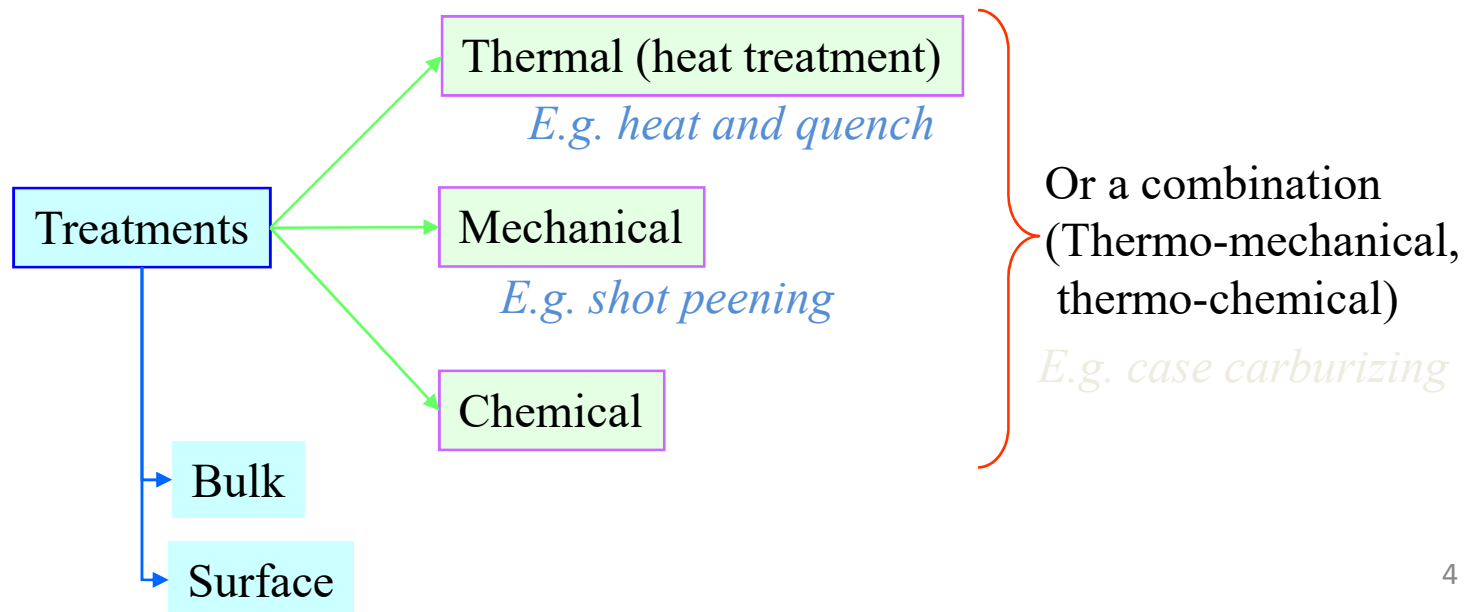
Content

- **Heat treatment processes:**
 - Stress relieving,
 - Annealing
 - full annealing,
 - partial annealing,
 - bright annealing,
 - diffusion annealing,
 - recrystallization annealing,
 - Spheroidizing,
 - Normalizing,
 - Hardening and Tempering, **Hardening of typical steels, cast irons and non-ferrous alloys.**
 - Hardening & Hardenability of steels.

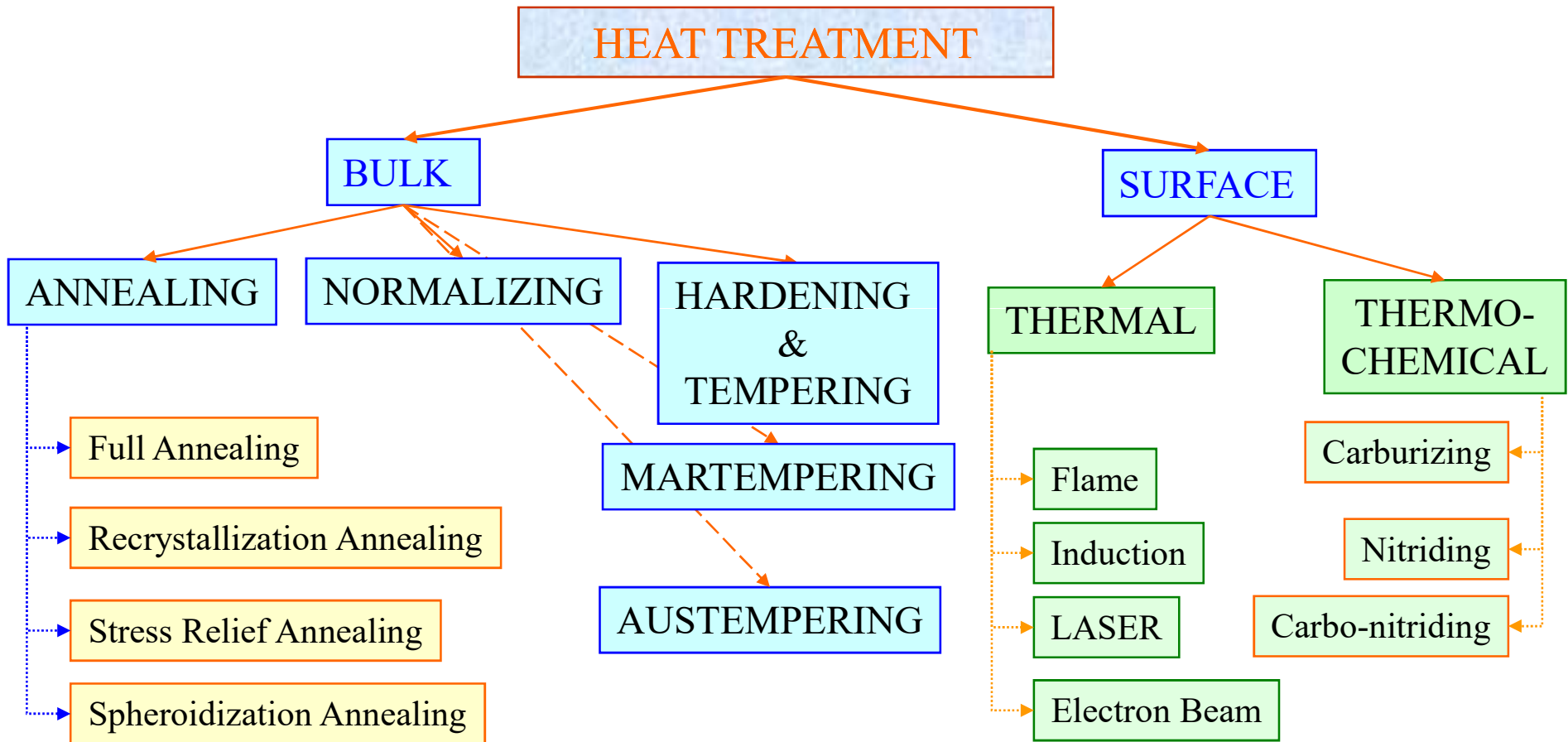
- **Surface hardening of metals:**
 - Principles and application of
 - Thermal Treatment
 - induction hardening
 - flame hardening methods
 - Laser hardening,
 - Thermo-Chemical Treatment
 - Case carburizing (solid, liquid and gaseous),
 - Cyaniding,
 - Carbonitriding,
 - Nitriding,
 - Plasma nitriding
 - etc.,
 - Depth of penetration - its measurement and relation with time and temperature

Introduction

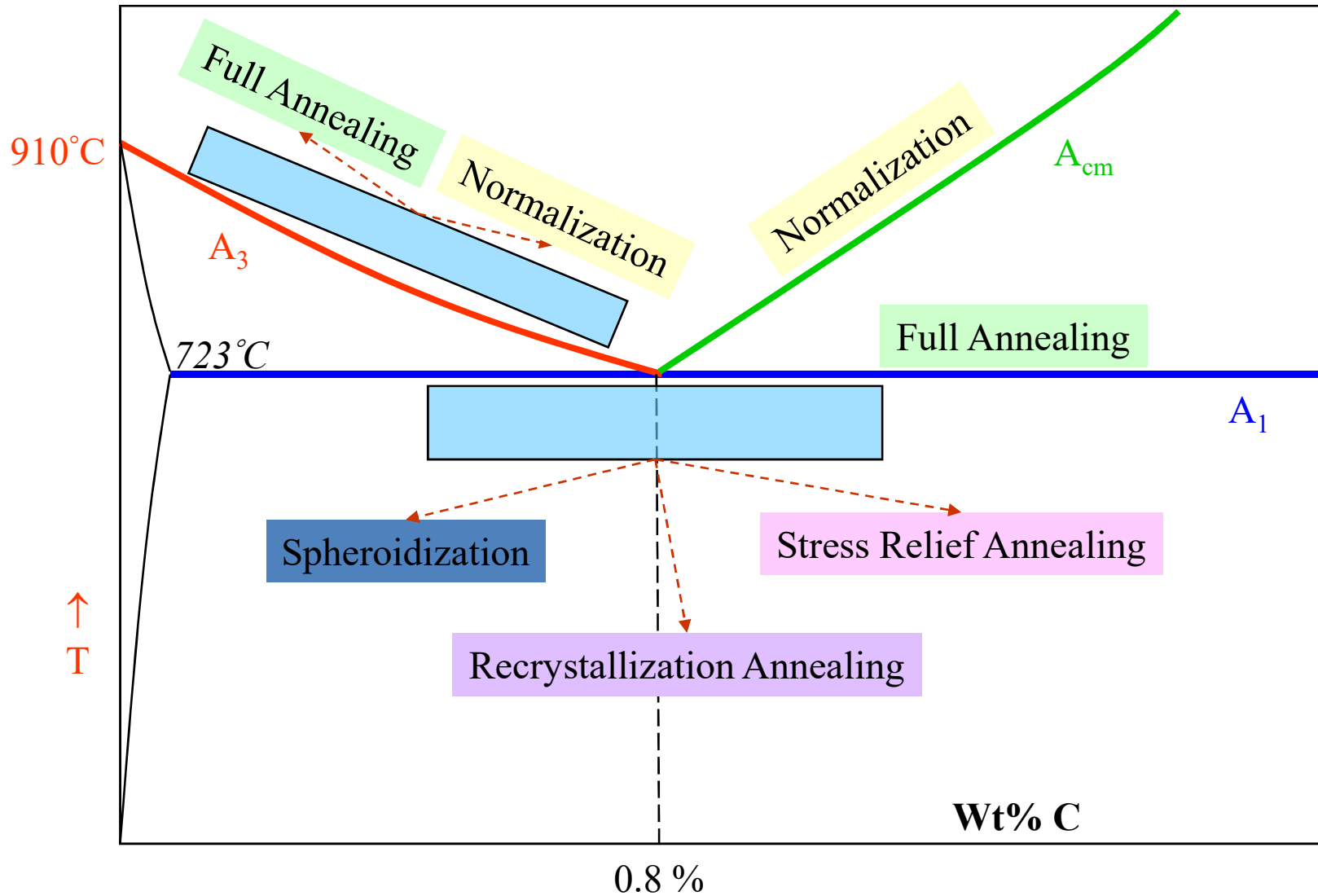
- In general three kinds of treatments are:
 - (i) Thermal (heat treatment),
 - (ii) Mechanical (working),
 - (iii) Chemical (alteration of composition).
 - (iv) A combination of these treatments are also possible (e.g. thermo-mechanical treatments, thermo-chemical treatments).
- The treatment may affect the whole sample or only the surface.



Overview of Heat Treatment



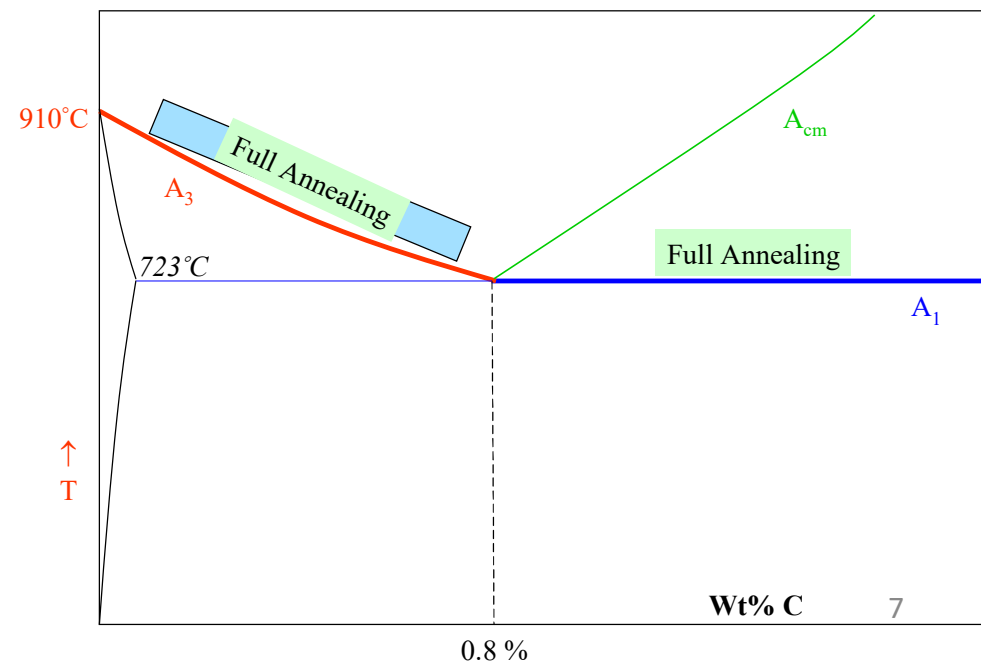
Ranges of temperature where Annealing, Normalizing and Spheroidization treatment are carried out for hypo- and hyper-eutectoid steels. Details are in the coming slides.



Full Annealing

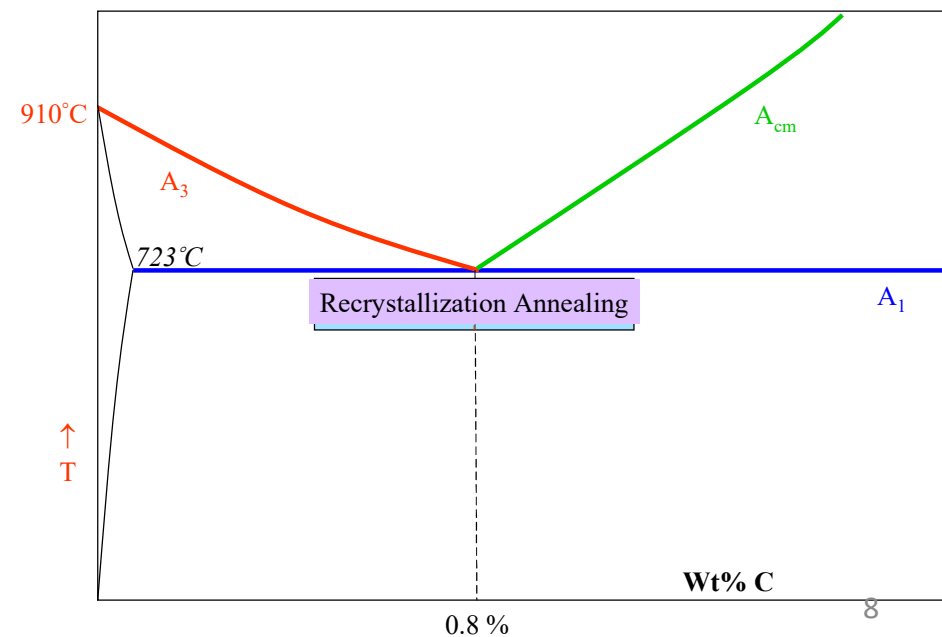
- Purpose → To obtain high ductility.
- A microstructure with coarse pearlite (i.e. pearlite having high interlamellar spacing) is endowed with such properties.
- Temp. Range: shown in figure.
- The steel is heated above A_3 (hypo-eutectoid steels) & A_1 (hyper-eutectoid steels) → (hold) → then the steel is furnace cooled to obtain Coarse Pearlite.
- Coarse Pearlite has low (↓) Hardness but high (↑) Ductility.

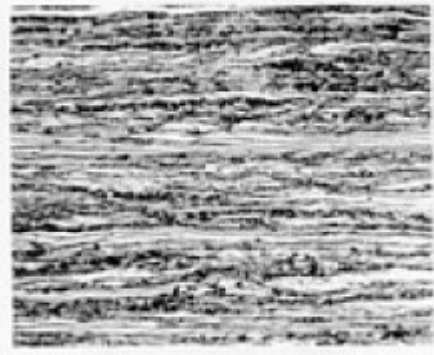
For hyper-eutectoid steels the heating is not done above A_{cm} to avoid a continuous network of proeutectoid cementite along prior Austenite grain boundaries



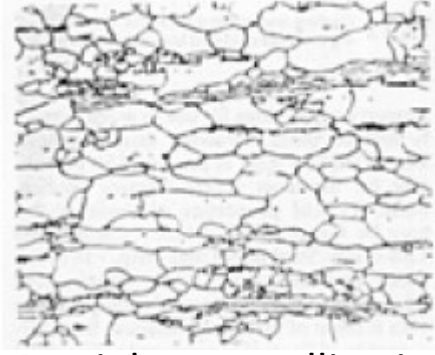
Recrystallization Annealing

- During any cold working operation, the material becomes harder, 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.

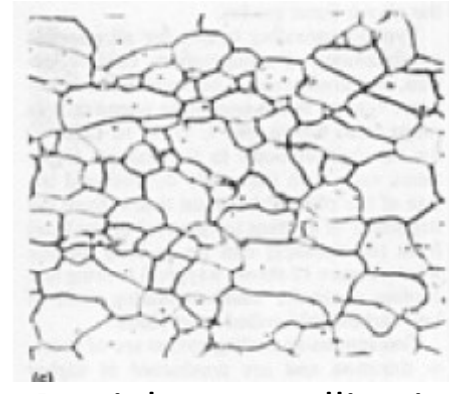




Cold rolled -unannealed



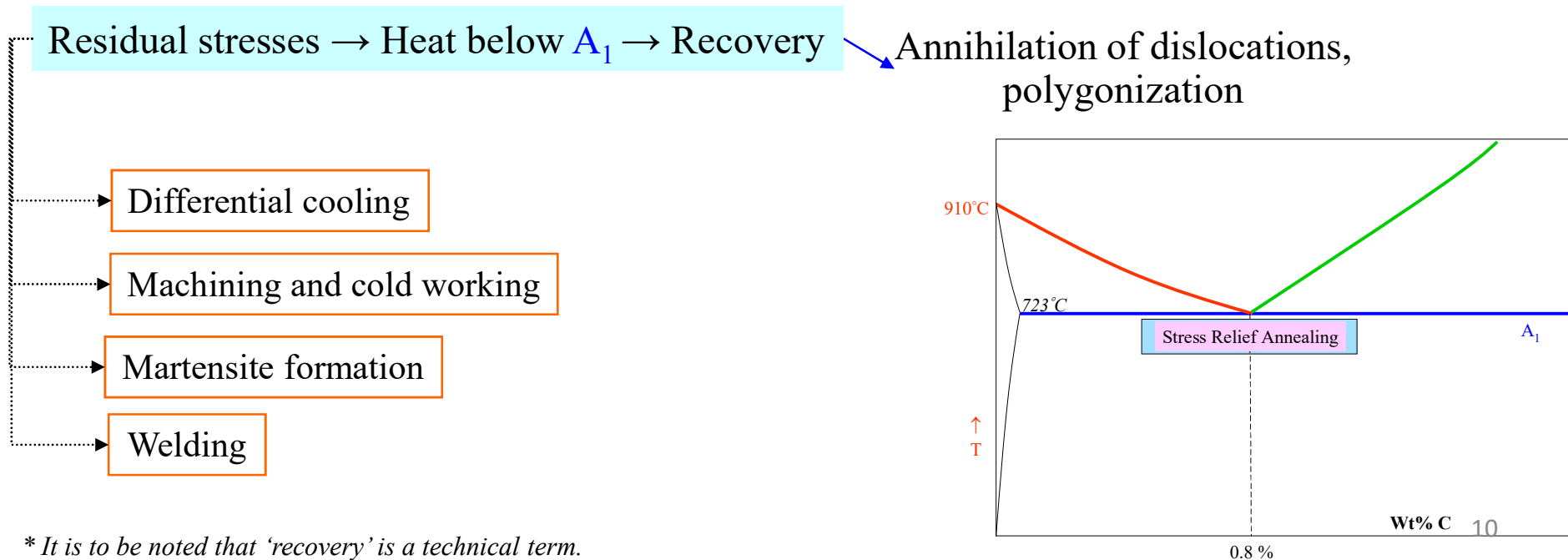
Partial recrystallization
annealing



Partial recrystallization
annealing

Stress Relieving Annealing

- Due to various processes like quenching, machining, phase transformations, 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).



Low angle grain boundary: Simple arrangement of dislocations.

The relation between θ and spacing between the dislocations is

$$\theta = 2 \tan^{-1} \frac{b}{2D} \approx \frac{b}{D}$$

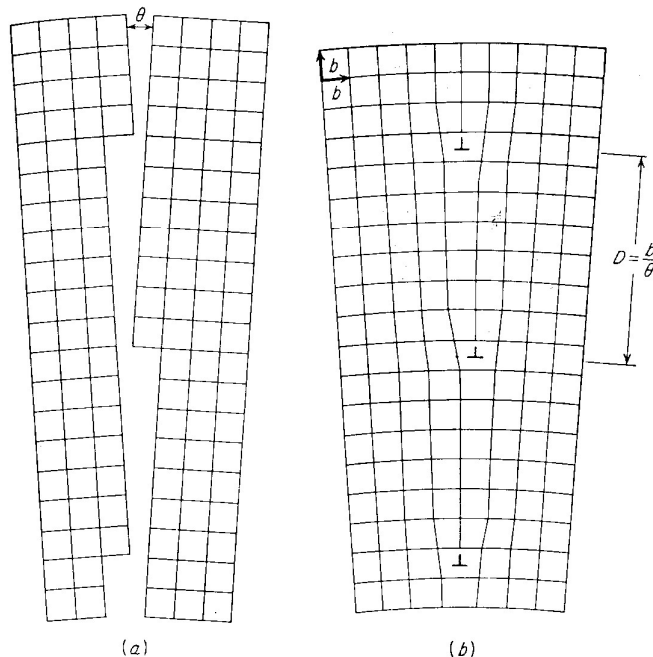
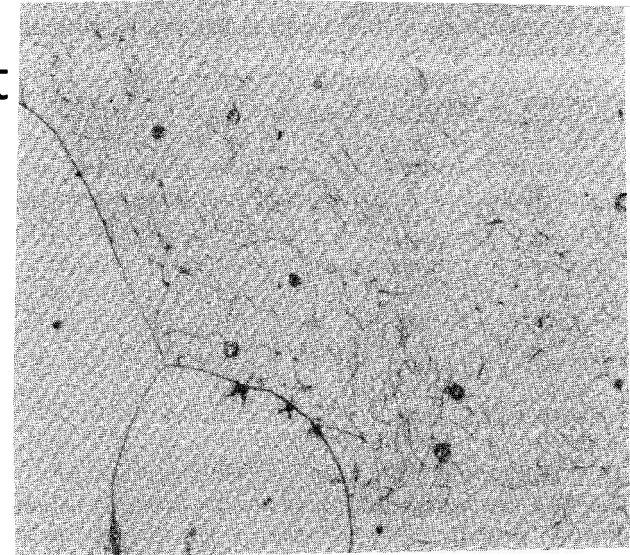
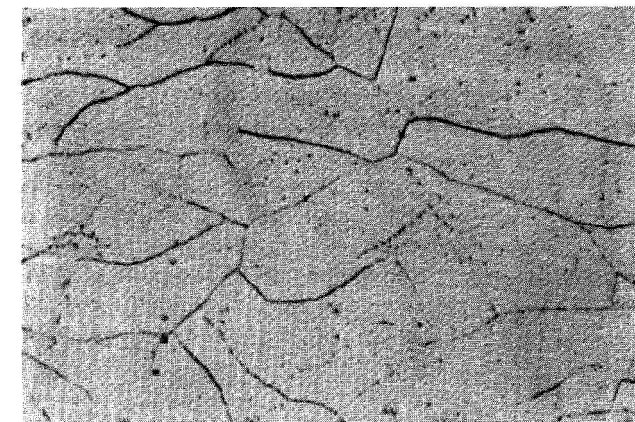


Diagram of low angle grain boundary

- a) Two grain having a common [001] axis and angular difference in orientation of θ
- b) Two grains joined together to form a low angle grain boundary made up of an array of edge dislocations



Substructure network of Fe-3%Si alloy (250X)



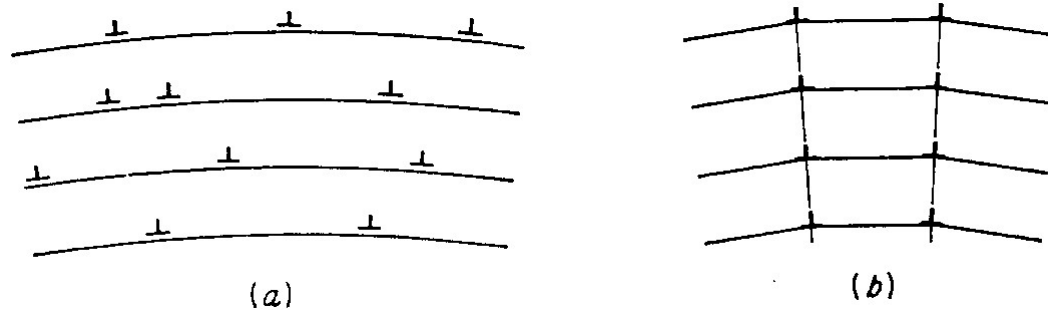
Substructure network of Fe-3%Si alloy (1000X)

Annihilation of Dislocations:

- Same dislocation repel each other
- Opposite dislocation attract each other.

Polygonization:

Dislocation of same sign assemble into boundaries and forms subgrain formation



Diffusion Annealing

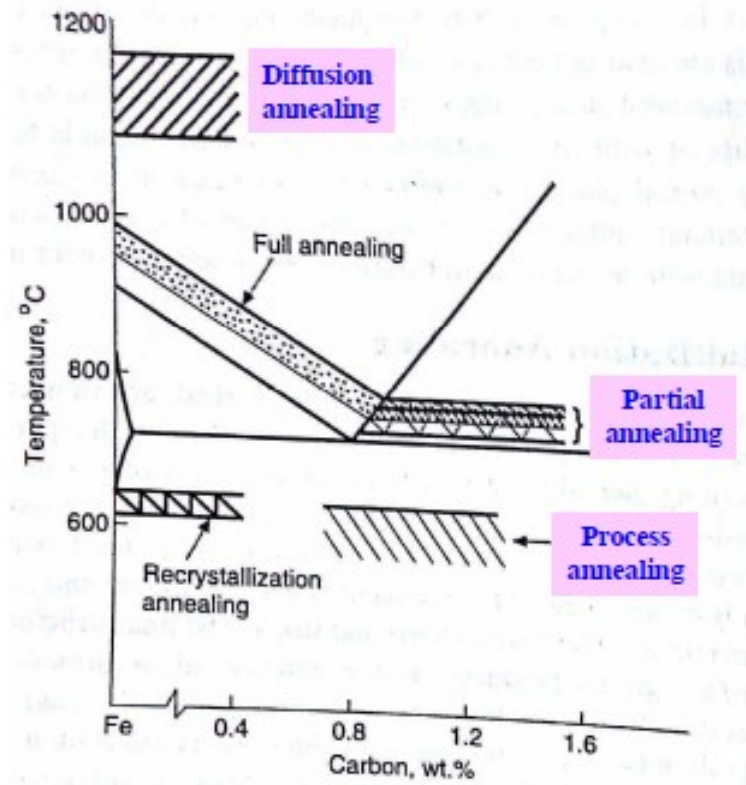
- This process also known as **homogenizing annealing**, is employed to **remove any structural non-uniformity**.
- Dendrites, columnar grains and chemical in-homogeneities are generally observed in the case of ingots, heavy plain carbon steel casting, and high alloy steel castings. These defects promote brittleness and reduce ductility and toughness of steel.
- In diffusion annealing treatment, steel is heated sufficiently above the upper critical temperature (1000-1200°C), and is held at this temperature for prolonged periods, usually 10-20 hours, followed by slow cooling.
- Segregated zones are eliminated and a **chemically homogeneous coarse grain steel** is obtained by this treatment as a result of diffusion.
- The coarse grained structure can be refined either by plastic working for ingots or by employing a second heat treatment for castings.

Partial / Intercritical /incomplete annealing:

- Steel is heated between A_1 and A_3 or A_{cm} followed by slow cooling
- Generally hypoeutecoid steels are subjected to this treatment. Microstructure \rightarrow fine pearlite + cementite.
- Temp. is low compared to full annealing.

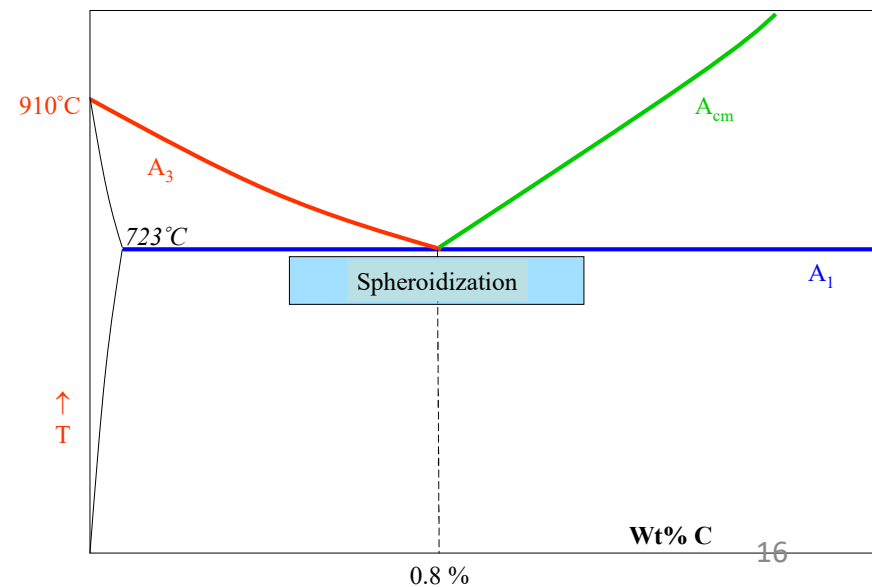
Process Annealing:

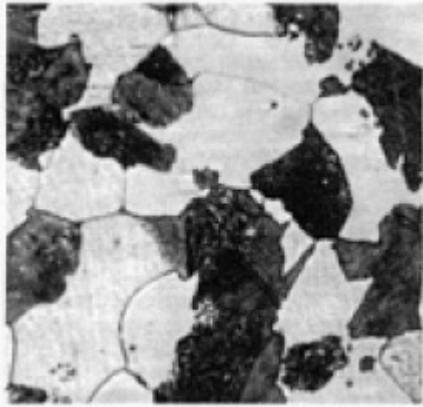
- Heated below lower critical temperature and hold at this temp. for sufficient time and then cooled.
- Purpose: reduce the hardness and increase ductility of cold worked steel for further working.
- It is an intermediate operation.
- Less expensive than recrystallization annealing.



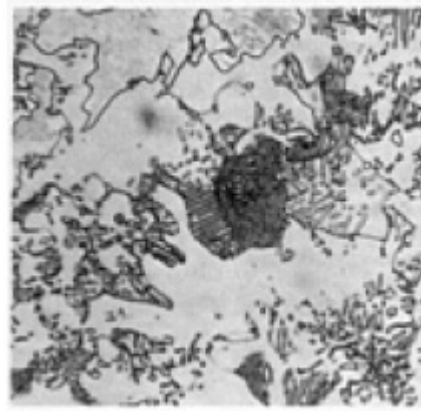
Spheroidization Annealing

- Applicable to high carbon steel requiring extensive machining prior to final hardening & 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.

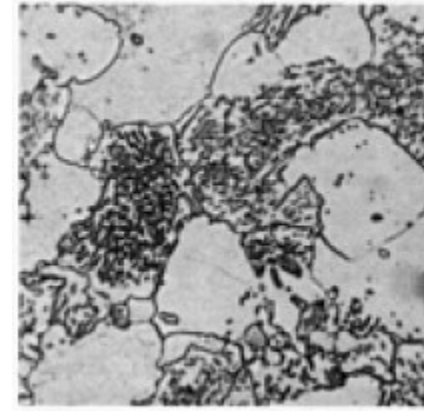




as-received hot-rolled condition, with blocky pearlite. Hardness is 87 to 88 HRB



Partially spheroidized condition
Hardness is 81 to 82 HRB.



Fully spheroidized condition
Hardness is 77 to 78 HRB.

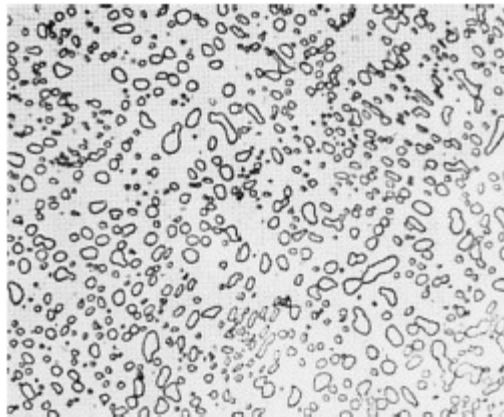


Figure The microstructure of spheroidite, with Fe₃C particles dispersed in a ferrite matrix (× 850). (From ASM Handbook, Vol. 7, (1972), ASM International, Materials Park, OH 44073.)

NORMALIZING

- 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°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.

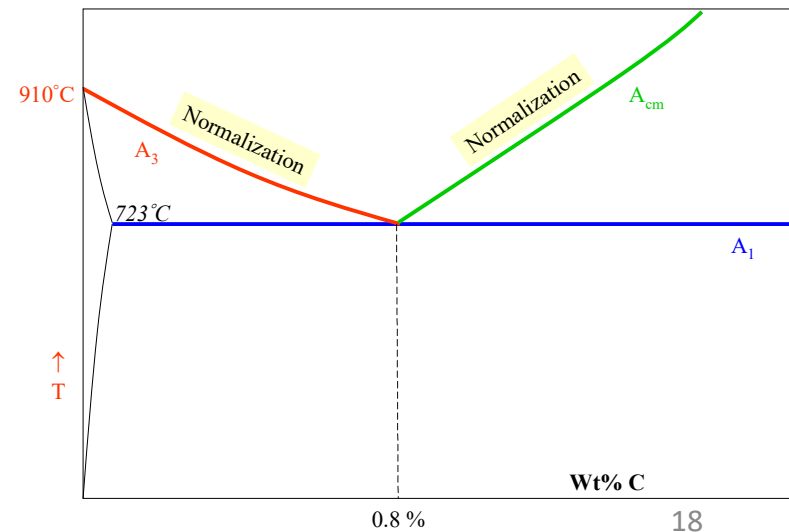
- The advantage of normalizing are

Purposes

Refine grain structure prior to hardening

To harden the steel slightly

To reduce segregation in casting or forgings



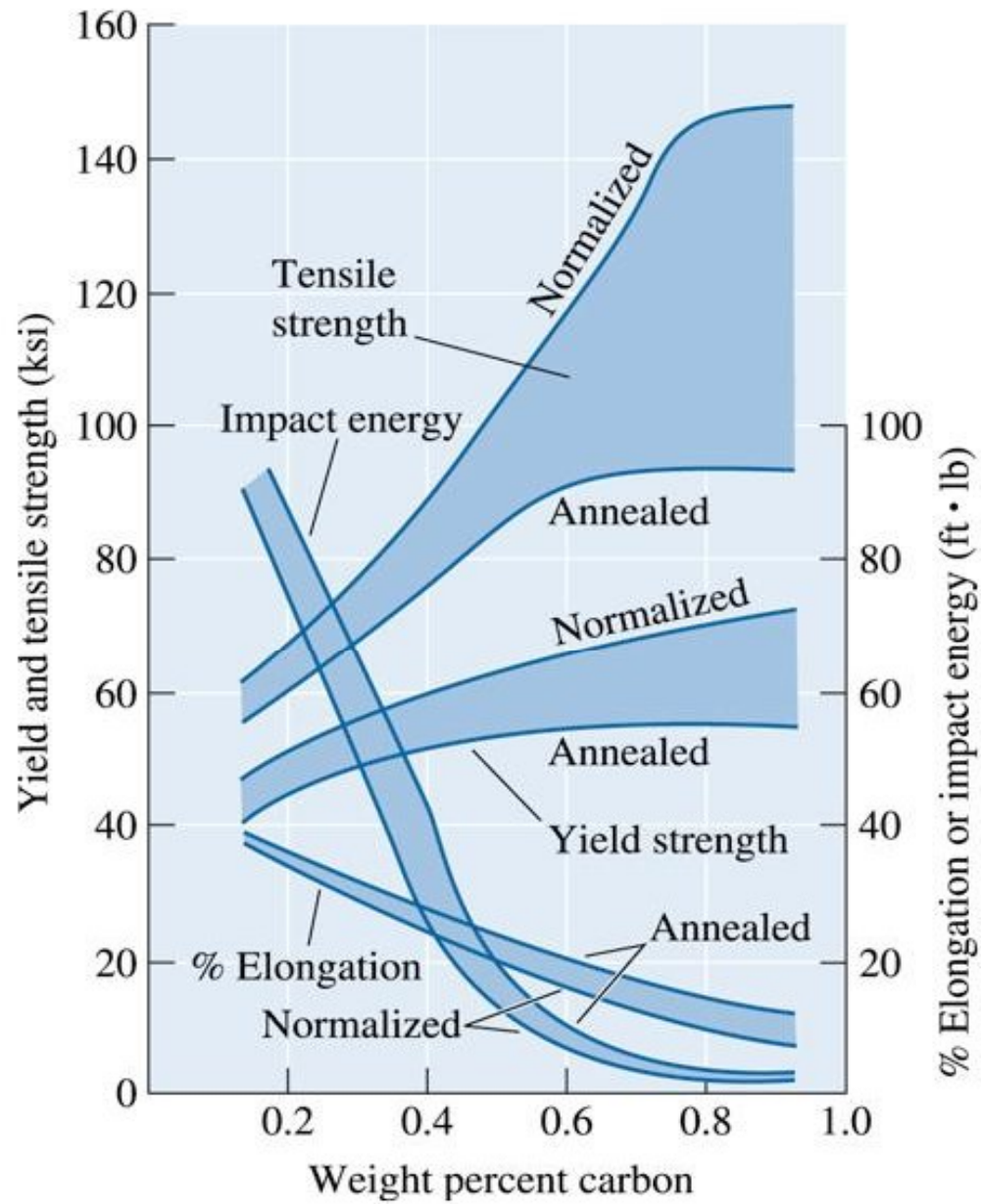


Figure :The effect of carbon and heat treatment on the properties of plain-carbon steels.

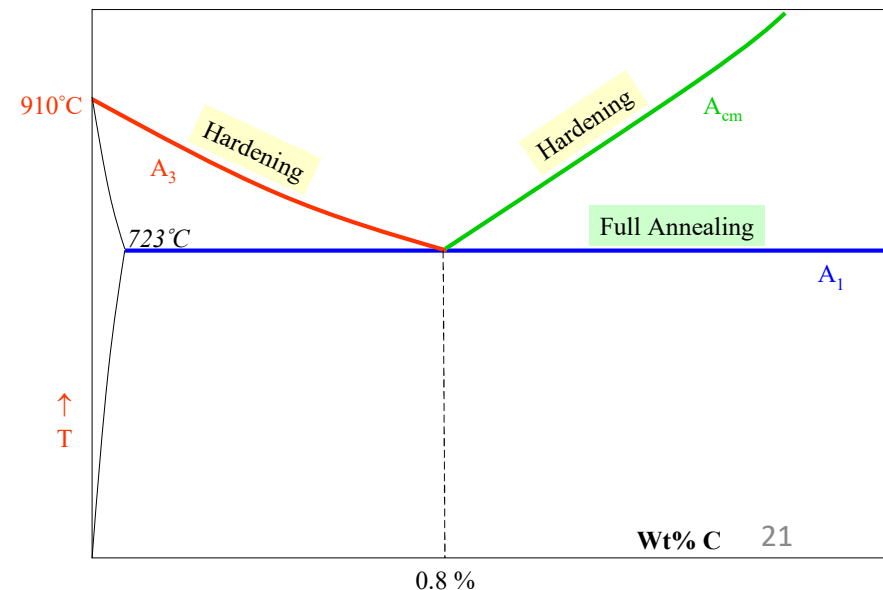
Difference between annealing and normalizing

Annealing	Normalizing
Low hardness and high ductility	High hardness and low ductility
Internal stress are nominal	Internal stresses are more due to faster air-cooling
Coarse grain	Fine grain
Expensive and takes longer time	Cheaper and takes less time
Hyper-eutectoid steel → Brittle cementite network	Hyper-eutectoid steel → globules of cementite
Rate of cooling is low	Rate of cooling is comparatively high
Amount of pearlite is low in hypoeutectoid steel	Amount of pearlite is high in hypoeutectoid steel
Chemical homogeneity is comparatively low	Chemical homogeneity is comparatively high

HARDENING

- Heated above A_3 | A_{cm} → Austenization → quenched (higher than the critical cooling rate)
- 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.

Purpose: High tensile strength and hardness



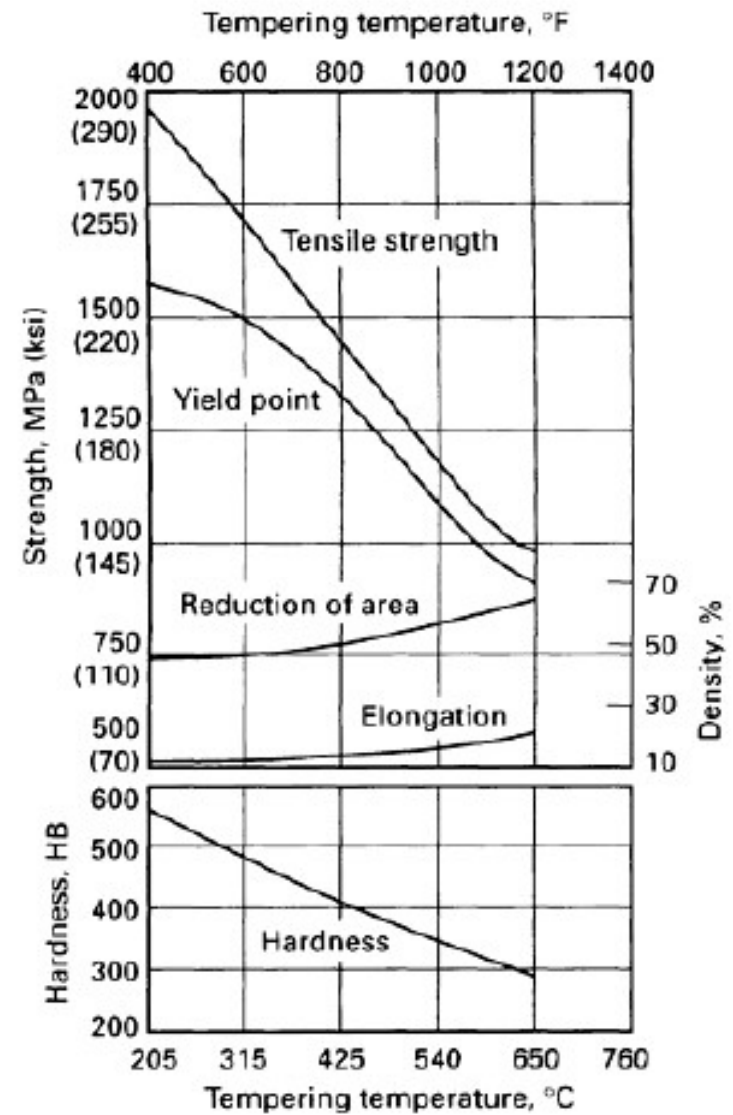
Tempering

- 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**.

TEMPERING is a process in which hardened steel is usually heated to a temperature below the lower critical temperature and cooled at a suitable rate.

TEMPERING

- Lowers hardness, strength and wear resistance
- Relieves the internal stress
- Restores ductility and toughness
- Transforms retained austenite



During tempering, based on the temperature, steel undergoes structural changes such as

- Isothermal transformation of retained austenite
- Ejection of carbon from martensite
- Formation of ferrite-carbide mixture

Types of tempering

- First stage / low temp. tempering
- Second stage / medium temp. tempering
- Third stage / high temp. tempering

First stage tempering

High carbon martensite $\xrightarrow{\leq 250^{\circ}\text{C}}$ Low carbon or tempered martensite + carbide

Min. C amt.=0.3%

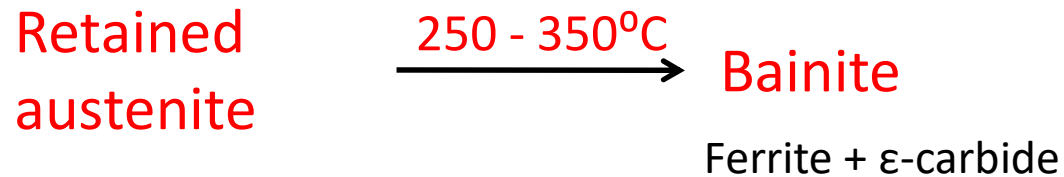
$\text{Fe}_{2.4}\text{C}$ in
HCP structure

- Marginal decrease in hardness
- Improves strength
- Improves toughness
- Excellent wear resistance
- Reduced internal stress
- Useful for high carbon & low alloy steels

LCS \rightarrow No formation of ϵ carbide.

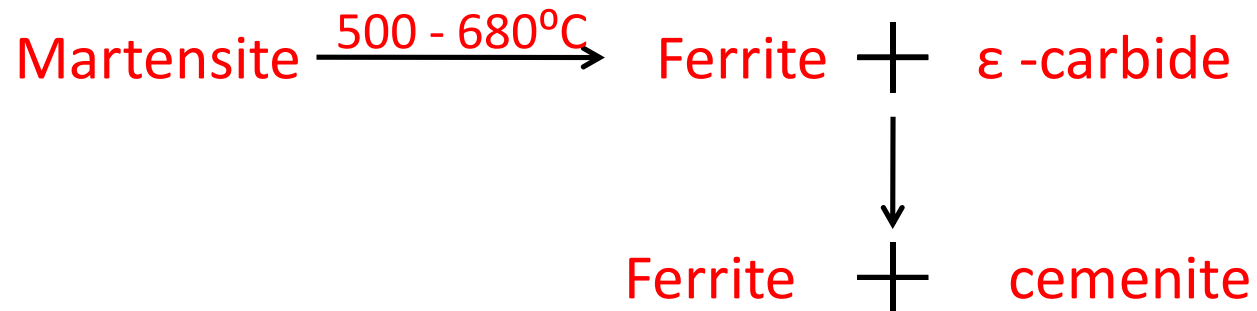
HCS \rightarrow formation of ϵ carbide.

Second stage tempering



- Ductility and toughness increases
- Hardness and strength decreases
- Develops max. elastic properties
- Suited for coil and laminated springs

Third stage tempering



- Transformation takes place by diffusion and nucleation
- Better tensile, yield and impact strength than annealed or normalized steel
- Free from internal stresses
- Suited for medium carbon and low alloys steel
- Ex: connecting rods, shafts & gears

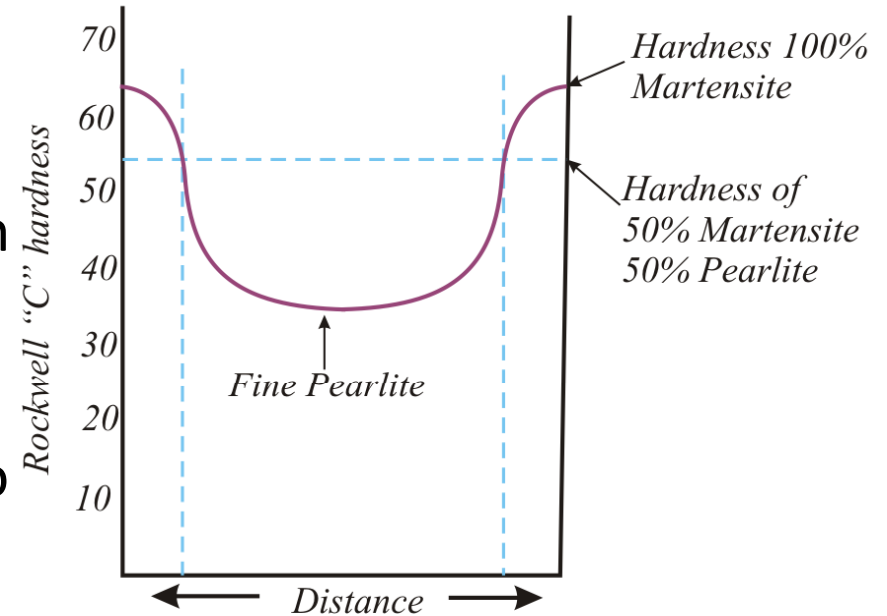
Effect of alloying elements on Tempering

- Alloys steels require higher tempering temperatures than carbon steels
- Non carbide forming elements such as Ni, Al, Si & Mn have little impact on hardness of steel
- Steels with carbide forming elements such as Cr, Mo, W, V, Ta & Ti improves the hardness value.

- **Secondary hardening**
 - Highly alloyed steel hardness value increases during tempering at elevated temperature. This behaviour is called as secondary hardening
- **Temper brittleness / embrittlement**
 - In certain steel either cooling slowly or on holding for prolonged periods within a specific temperature range (400 to 600°C), toughness values decreases.
 - Occurs in alloyed steels and not in carbon steels.
 - Steels with P, Sb, Sn & Ar prone to this
 - Mo, Ti & Zr suppress these kind of embrittlement

Hardenability

- The ability of steel to form martensite on quenching is referred to as the hardenability.
- **Hardenability** - a measure of the capacity of a steel to be hardened in depth when quenched from its austenitizing temperature.
- **Hardenability** - Ability of the steel to harden throughout its cross section while quenching
- Steels with high hardenability form martensite even on slow cooling from surface to core.



Typical hardness test survey made along a diameter of a quenched cylinder

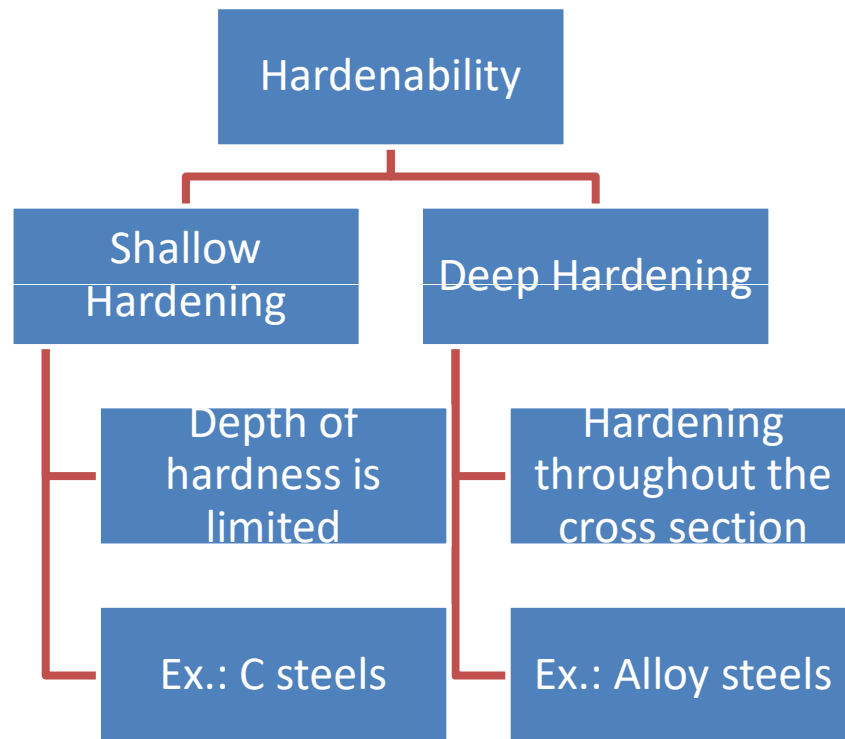
Max. Hardness of depends on

- Carbon content of the steel
- All the carbon in austenite solid solution
- Critical cooling rate
- No RA present after quenching
- No tempering of martensite take place

Factors affecting Hardening Processes

- Chemical composition of steel
- Size and shape of the steel part
- Hardening cycle (heating/cooling rate, temp, soaking time)
- Homogeneity and grain size of austenite
- Quenching media
- Surface condition of steel part

On the basis of hardenability, the steel can be classified as,



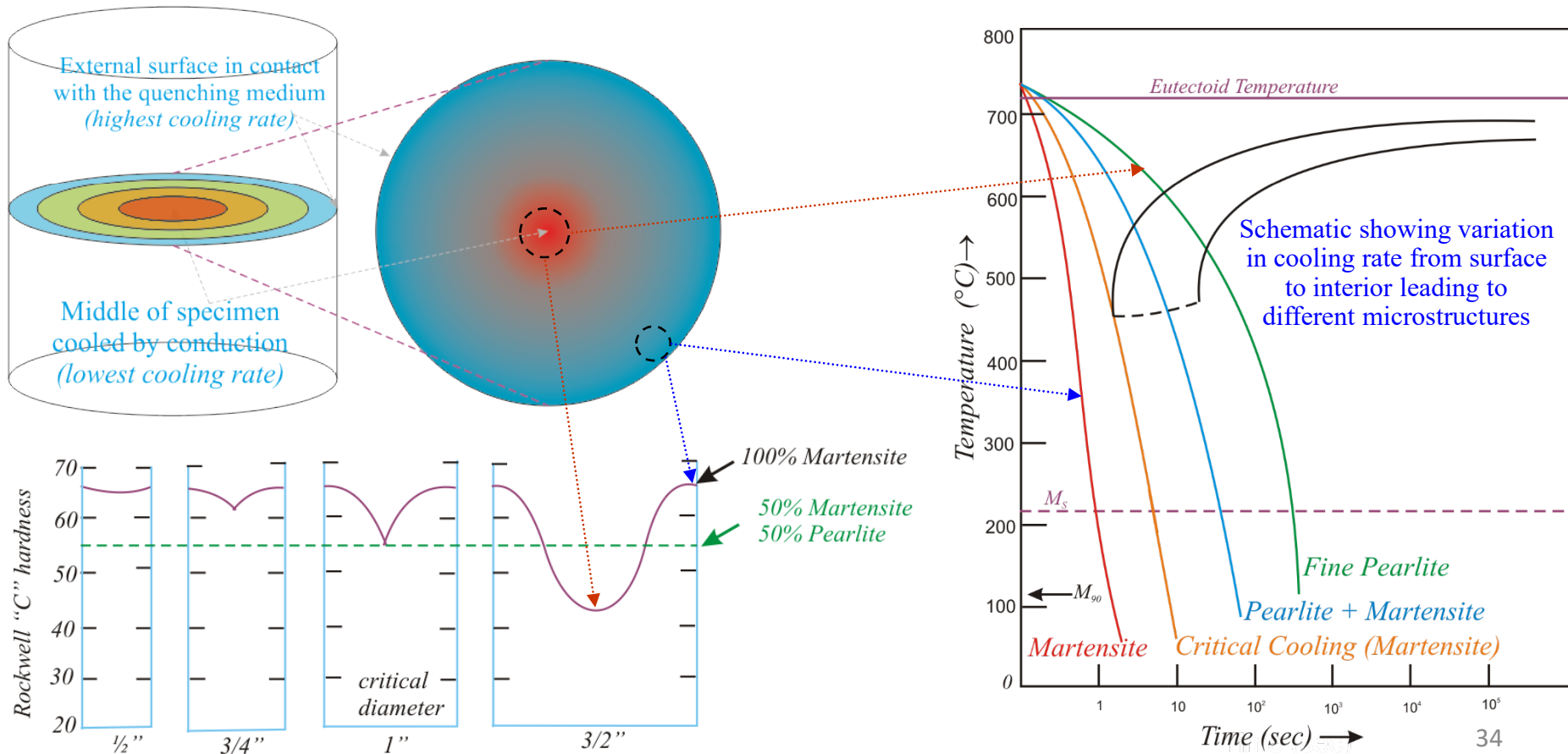
Martensite and pearlite regions are distinguished in the steel by

- Change in microstructure
- Change in hardness
- Fracture surface modifications

- Hardening Methods
 - Conventional or direct quenching
 - Quenching in stages in sequence in different media
 - Spray Quenching
 - Quenching with self tempering
 - Austempering or Isothermal Quenching
 - Martempering

Hardening of Steel

Critical diameter (D_c) is that diameter, which can be through hardened (i.e. **50% Martensite and 50% pearlite** at the centre of the sample).



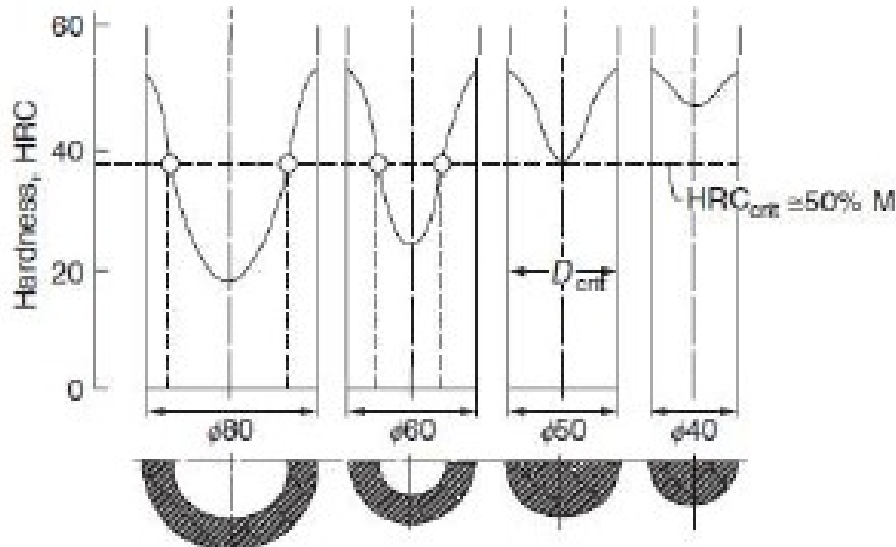
Determination of Hardenability

Hardenability of steel is determined by the following methods

- Grossman's critical diameter method
- Jominy end quench test
- Estimation of hardenability from chemical composition
- Fracture test

Grossman's critical diameter method

- In Grossman's method, round bars of different diameters are used
- These bars are quenched in a suitable quenchants.
- Further, the critical diameter (D_c) (50% martensite) on quenching is determined. D_c depends on composition of steel and quenching
- The ideal diameter (D_i) is then determined from the curve.
- This type of experiment requires multiple Austenitisation and quenching treatments on specimens of varying diameter just to quantify the hardenability of a single material.



The critical diameter (D_c) - the maximum diameter of the rod which produced 50% of martensite at the centre on quenching.

The ideal diameter (D_i) - The critical diameter corresponds to quenching an ideal medium

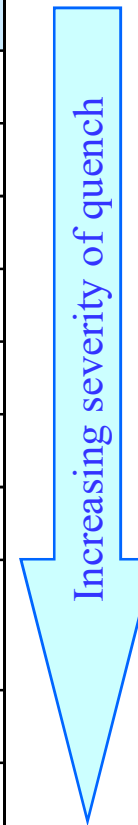
Severity of Quench as indicated by the heat transfer equivalent **H**

$$H = \frac{f}{K} \quad [m^{-1}]$$

f → heat transfer coefficient between steel and medium

K → Thermal conductivity

Process	Variable	H
Air	No agitation	0.02
Oil quench	No agitation	0.2
"	Slight agitation	0.35
"	Good agitation	0.5
"	Vigorous agitation	0.7
Water quench	No agitation	1.0
"	Vigorous agitation	1.5
Brine quench (saturated Salt water)	No agitation	2.0
"	Vigorous agitation	5.0
Ideal quench		∞



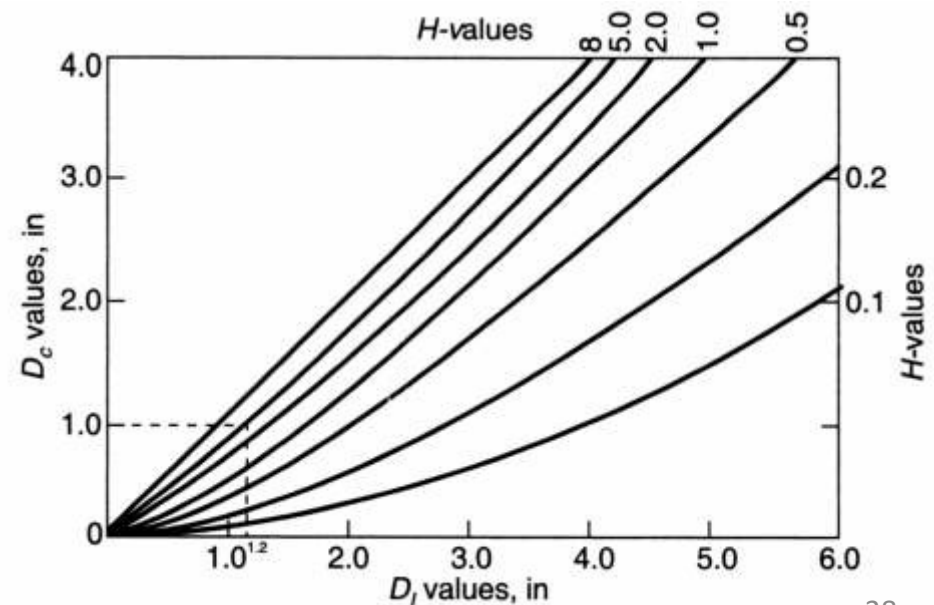
The **H** value/index compares the relative ability of various media (gases and liquids) to cool a hot solid. Ideal quench is a conceptual idea with a heat transfer factor of ∞ (⇒ H = ∞).

- The relation between ideal critical diameter D_i and critical diameter D_c and severity of quench (H) can be determined from thermodynamic considerations.
- A number curves are plotted in this graph and each belongs to different rates of cooling. In every case, the rate of cooling is measured by the H -value or the severity of quench.
- From this graph, by knowing the value of D_c , the corresponding value for D_i can be found out. For example, assuming $D_c = 1$ inch and $H = 5$ (for agitated brine quench), the critical diameter or hardenability works out to 1.2 inch with help of figure.

From figure it can be observed that

$$D_i = D_c \text{ for } H = \alpha$$

$$D_i > D_c \text{ for } H < \alpha$$

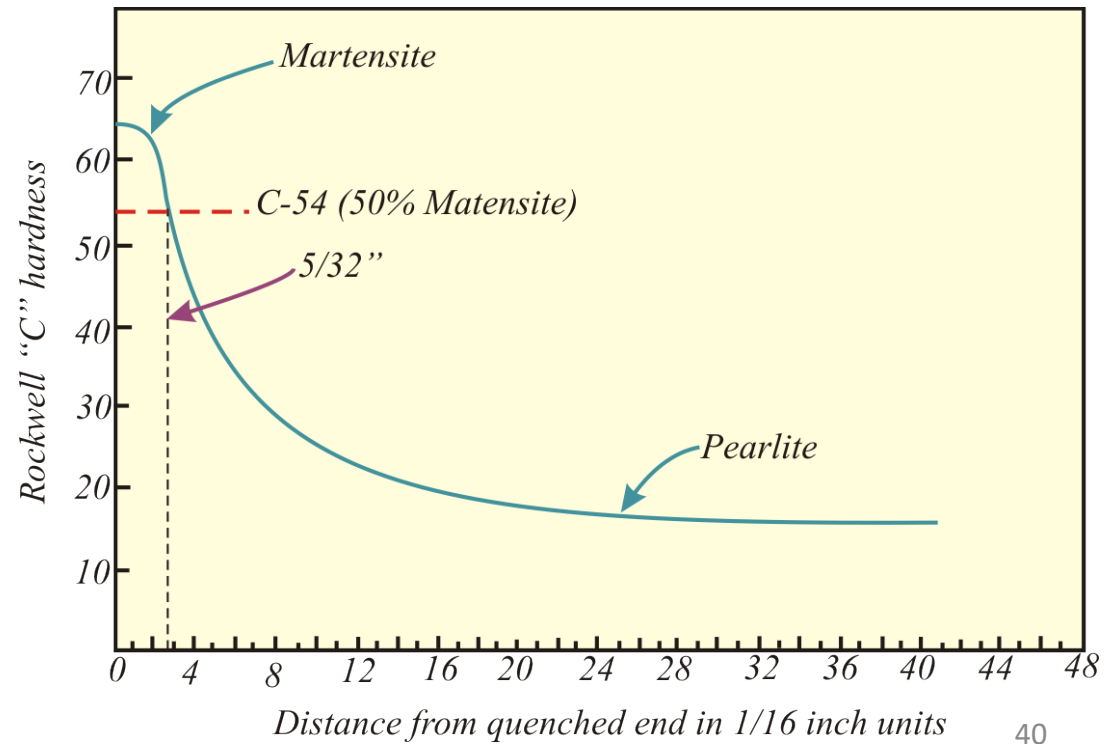
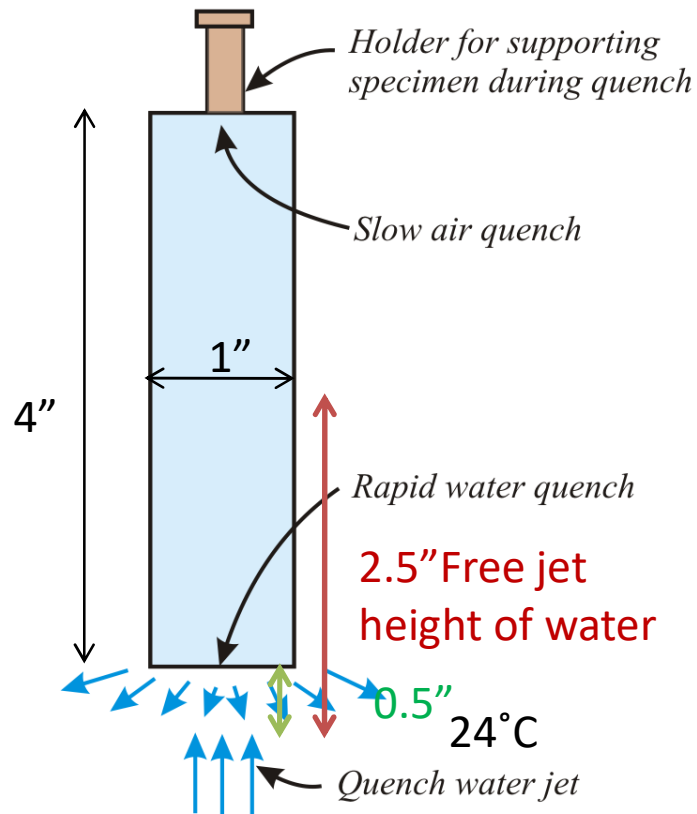


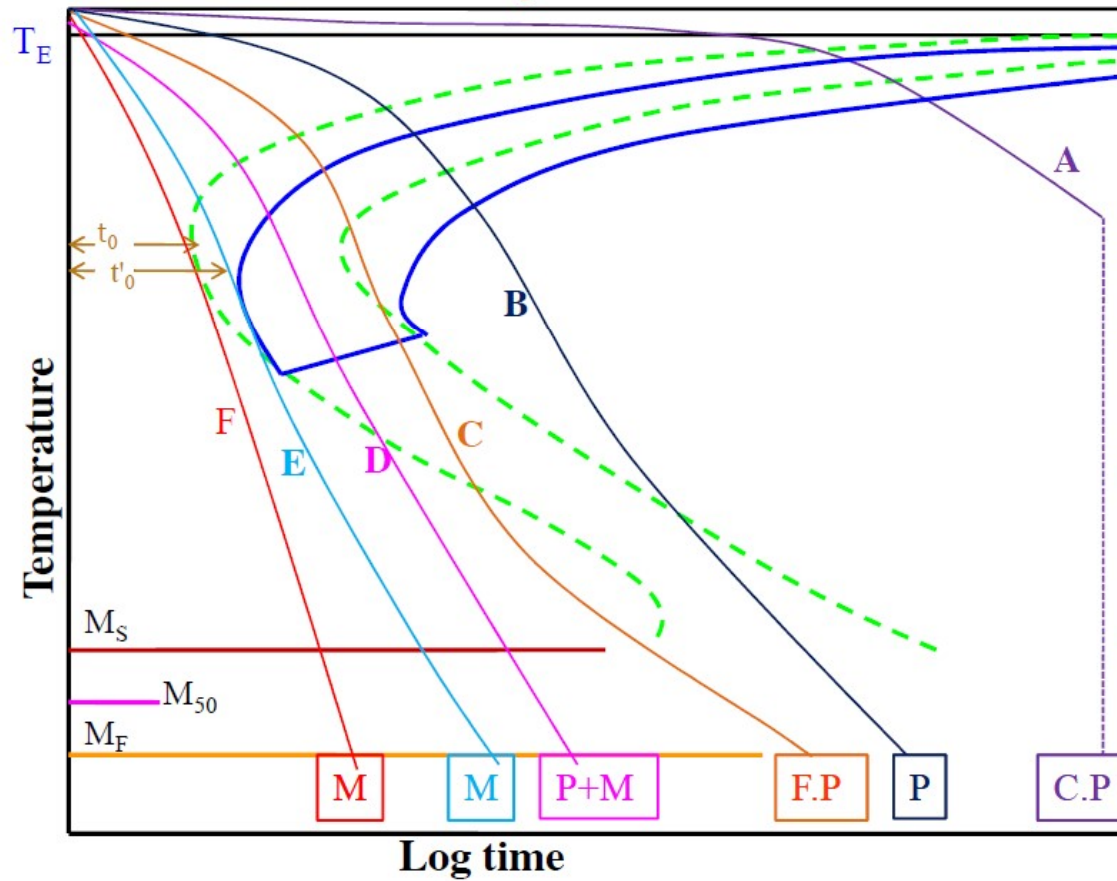
Jominy End Quench Test

- Commonly used method for determining hardenability is the end quench test developed by **Jominy and Boegehold**
- Grossmans method requires multiple Austenitisation and quenching treatments on specimens of varying diameter just to quantify the hardenability of a single material.
- An alternative approach is the Jominy end-quench test which require less samples.
- The Jominy end-quench test is specified in ASTM standard A255 and is a widely used method for quantifying hardenability.
- Information gained from this test is necessary in selecting the proper combination of alloy steel and heat treatment to **minimize thermal stresses and distortion** when manufacturing components of various sizes.

Jominy End Quench Test

- Sample Size: Diameter-1" length -4"
- Heated to reach austenising temp. Followed by quenching from bottom for 20min.
- Hardness measurement : from each 1/16" interval from the longitudinal direction of the sample from the centre.



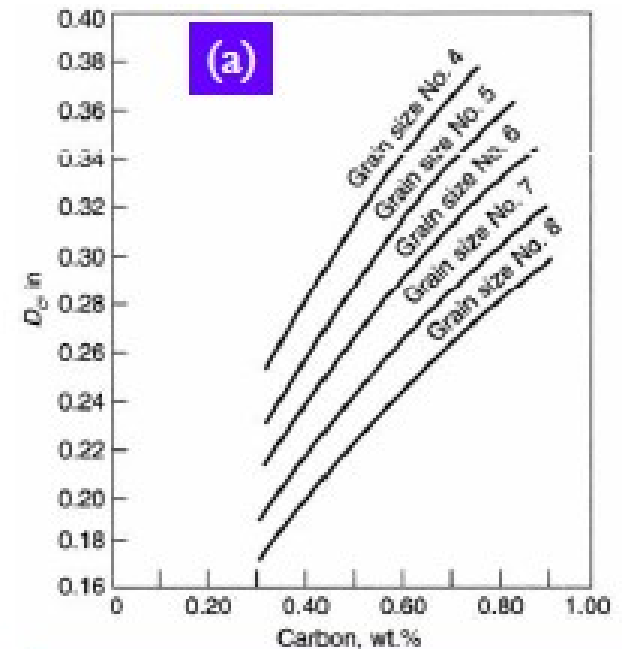


t_0 = Minimum incubation period at the nose of the TTT diagram.
 t'_0 = minimum incubation period at the nose of the CCT diagram

M – Martensite
 P – Pearlite
 F.P – Fine pearlite
 C.P – Coarse pearlite
 M_S – Martensite start
 M_F – Martensite finish
 T_E – Eutectoid temp.

Estimation of Hardenability from chemical composition

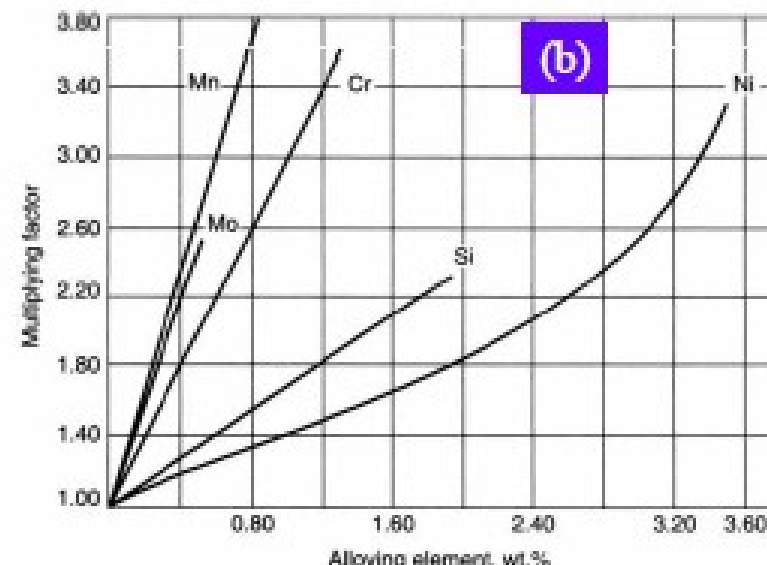
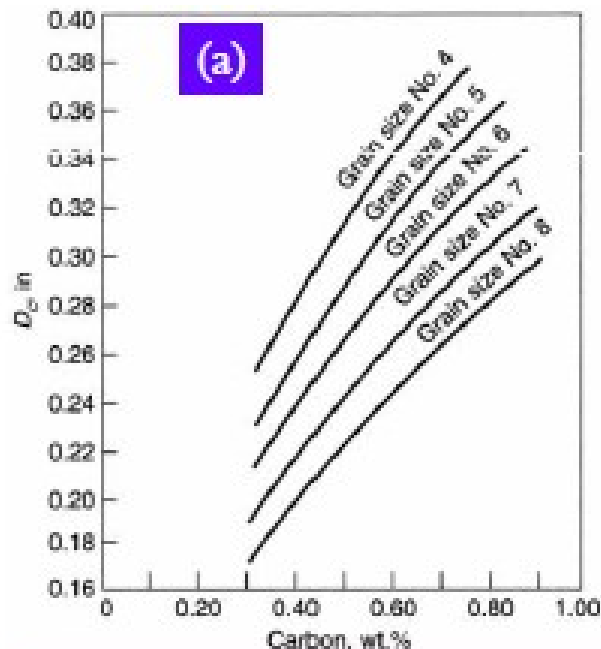
- Although the ideal critical diameter is generally determined experimentally, it is also possible to estimate it from **chemical composition and the grain size of steel**
- This method is based on the fact that the hardening of steel is controlled basically by the carbon content.
- Every steel has a base hardenability which depends only on **carbon content and grain size**.
- **Alloying additions change the rate of reaction.**
 - Furthermore, the effect of each alloying element is independent of other alloying elements.
 - The effect is also independent of carbon content and grain size.
 - Mo, Mn and Cr are seen to very effective.



- The base diameter obtained from Figure (a) is multiplied by the multiplying factors F for each of the alloying elements given in figure (b)

$$D_i = D_c(\text{base diameter}) \times F_{Mn} \times F_{Cr} \times F_{Ni}$$

- For example, for a steel of grain size ASTM 8, with 0.5% C, 0.6% Mn, 1% Cr and 2% Ni, $D_i = 0.22$ (fig. a) $\times 3.00$ (Mn factor from figure b) $\times 3.17$ (Cr factor from figure b) $\times 1.77$ (Ni factor from figure b) = 3.70"
- S and P are present in low concentrations in steel as impurity. Their combined effect can be ignored in most of the cases. Multiplying factors for S and P are unity



Fracture Test

- There is a contrast in the nature of fracture undergone by martensitic and pearlitic regions.
- Where as **martensite** formed on the case exhibits **brittle fracture**, the **pearlite** formed in the core undergoes **ductile fracture**.
- Where there is **change over from martensitic to pearlitic structure**, there is corresponding **sharp change from brittle to ductile fracture**.
- It is similar to a sudden change in hardness or microstructure as one passes from martensitic to pearlite region. This region of sudden change is the one that contains 50% pearlite and 50% martensite.
- The method based on the nature of fractured surface is successful when the transformation processes is quick and a sharp boundary is formed.
- **When the transformation is sluggish, this method cannot be applied** since the gradient in hardness is gradual, and it is not possible to get a clear demarcating boundary.

Factors effecting Hardenability

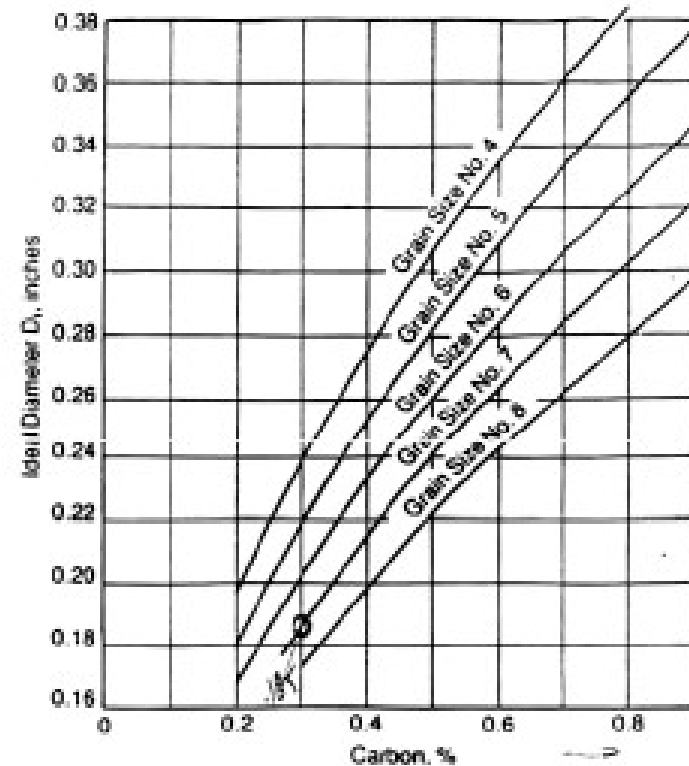
- The variables influence hardenability are
 - Quenching conditions
 - Austenitic grain size
 - Carbon content
 - Alloying elements

Quenching Conditions

- Each fluid has its own thermal properties like..
 - Thermal conductivity
 - Specific heat
 - Heat of vaporization
- These cause rate of cooling differences
- Ideal quenchant : It is one which brings down the surface temperature to room temperature instantaneously and keeps it at that temperature thereafter.

Austenitic Grain Size

- The hardenability increases with increasing austenite grain size, because the grain boundary area which act as nucleating site is decreasing.
- The sites for the nucleation of ferrite and pearlite are being reduced in number, with the result that these transformations are slowed down, and the hardenability is therefore increased.
- Smaller γ -grain size \rightarrow lower hardenability
- Larger γ -grain size \rightarrow higher hardenability



Percentage of C:

- C is primarily a hardening agent in steel.
- It also increases hardenability by slowing the formation of pearlite and ferrite.
- But its use at higher levels is limited, because of the lack of toughness which results in greater difficulties in fabrication and, most important, increased probability of distortion and cracking during heat treatment and welding.

Effect of Alloying Element:

- Most **metallic alloying elements** slow down the ferrite and pearlite reactions, and so also **increase hardenability**. However, quantitative assessment of these effects is needed.
- Cr, Mo, Mn, Si, Ni and V all effect the hardenability of steels.
- B can be an effective alloy for improving hardenability at levels as low as .0005%.
- **B** has a particularly large effect when it's added to **fully deoxidized low carbon steel**, even in concentrations of the order of 0.001%, and would be more widely used if its distribution in steel could be more easily controlled.
- The most economical way of increasing the hardenability of **plain carbon steel is to increase Mn content**, from 0.60 wt% to 1.40 wt%, giving a substantial improvement in hardenability.

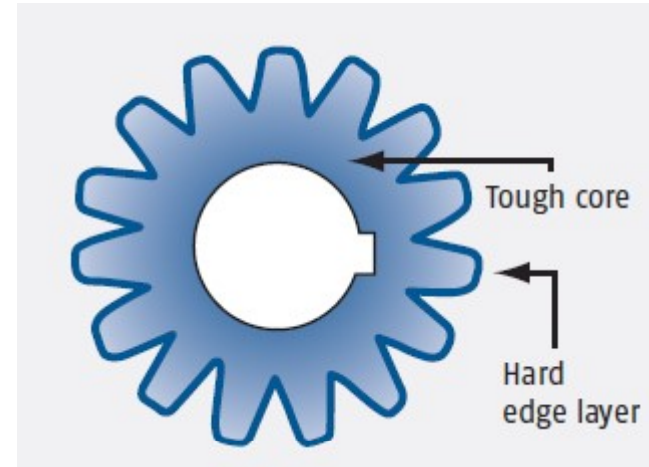
- **Cr and Mo** are also very effective, and amongst **the cheaper alloying** additions per unit of increased hardenability.
- Hardenability of a steel increases with addition of alloying elements such as Cr, V, Mo, Ni, W-→TTT diagram moves to the right.

Exceptions

- S - reduces hardenability because of formation of MnS and takes Mn out of solution as MnS
- Ti - reduces hardenability because it reacts with C to form TiC and takes C out of solution; TiC is very stable and does not easily dissolve
- Co - reduces hardenability because it increases the rate of nucleation and growth of pearlite

Surface hardening

- Combination of wear resistance (to give long life) and toughness (to withstand shock loads)
- Heavy cross section - impossible to cool quickly to produce a uniformly martensitic structure throughout
- Applications: ball and tapered bearings, gears, bushings, rock drill bits, dies, gun barrels, cam shafts, crank pins, valve rocker shafts and axles.



Surface Hardening



High wear resistance



Increased mechanical properties



High stability



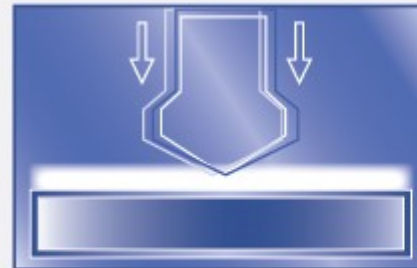
Improved fatigue strength



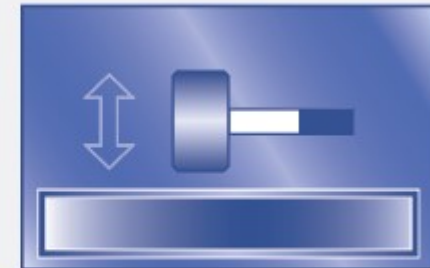
High surface hardness



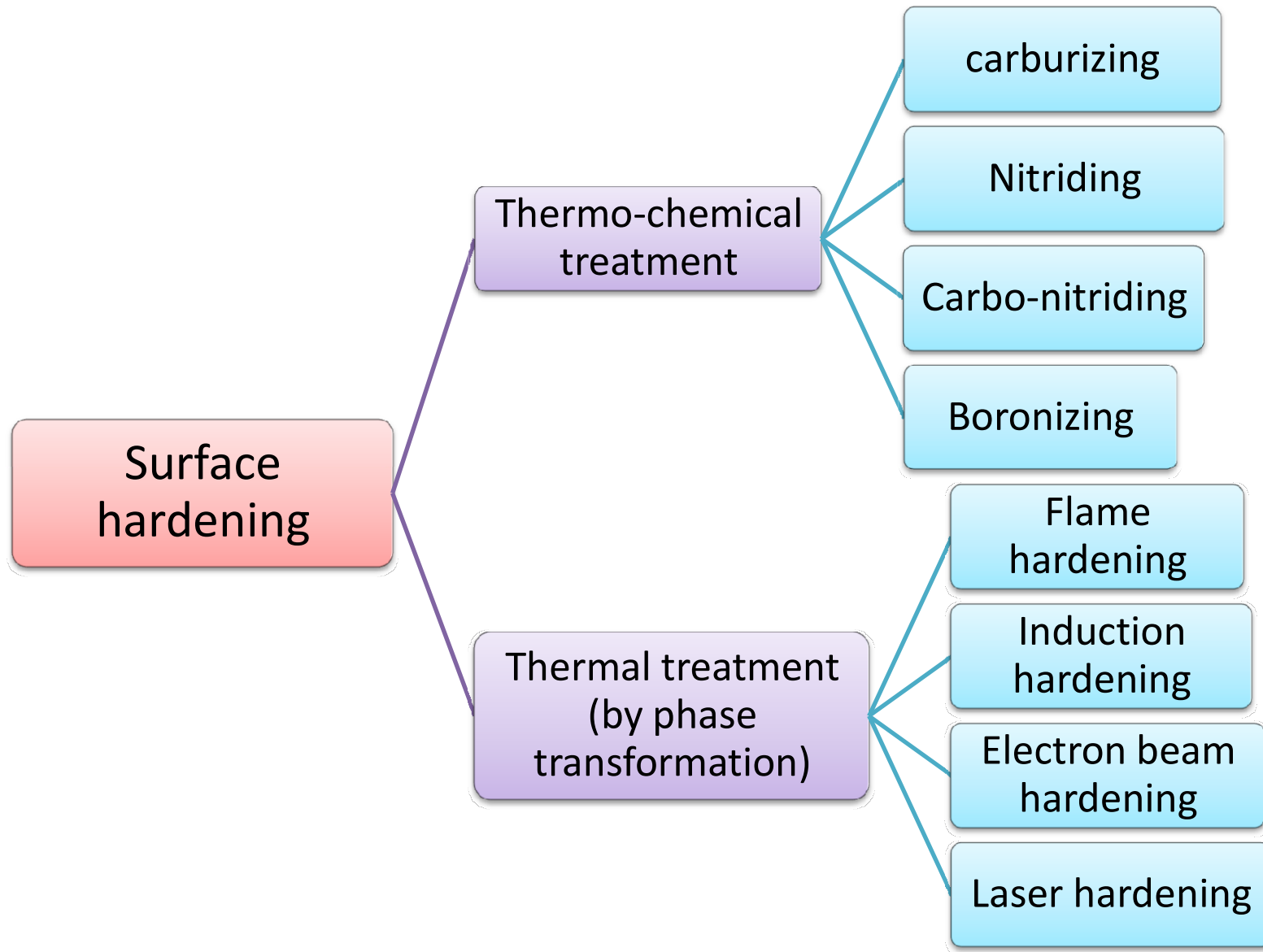
Improved ductility



Increased impact resistance

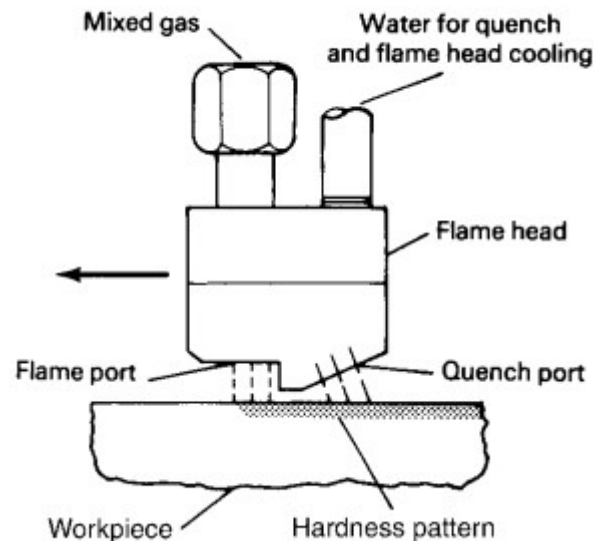


High resistance against impacts

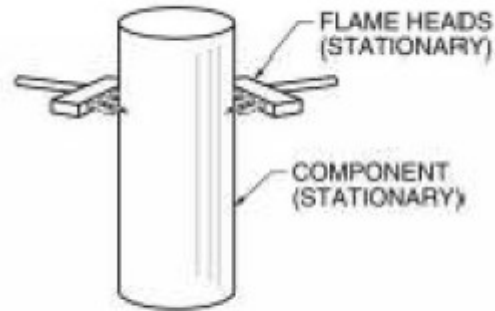


Flame Hardening

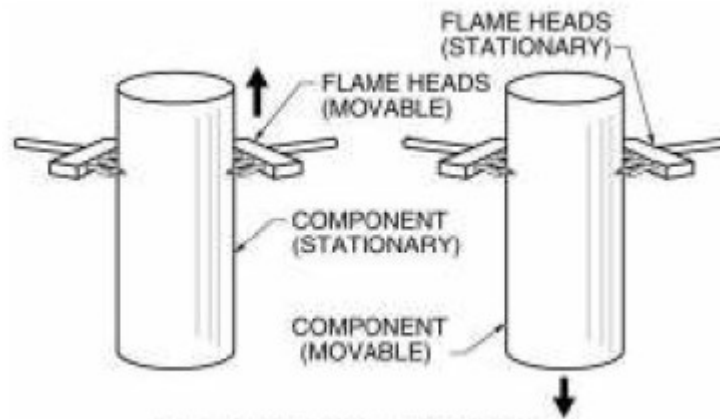
- Simplest form of surface hardening heat treatment.
- This process consists of heating the large work-piece, such as crank shaft, axle, large gear, cam, bending roller, or any other complicated cross-section, by an oxy-acetylene, or oxyfuel blow pipe, followed by spraying of jet of water as coolant.
- After hardening, reheating of the parts is carried out in furnace or oil bath at about 180-200°C for stress relieving.
- Normally, case depth up to 3mm can be achieved.



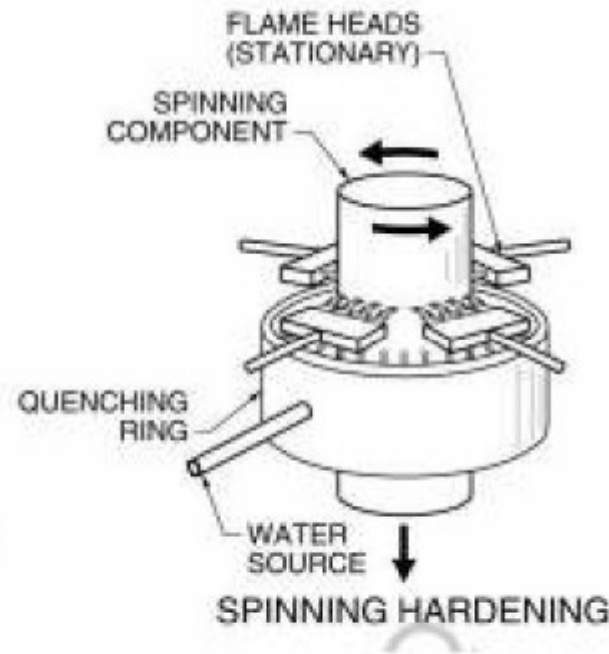
- Four methods are generally use for Flame Hardening
 - Stationary (Spot): Torch and work is stationary
 - Progressive: Torch moves over a work piece
 - Spinning: Torch is stationary while work piece rotates
 - Progressive-spinning: Torch moves over a rotating work piece.



SPOT HARDENING



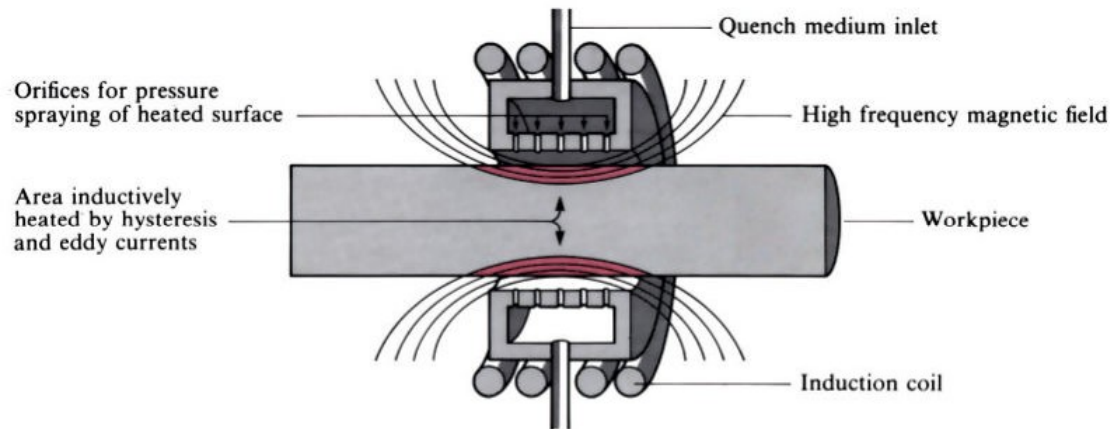
PROGRESSIVE HARDENING



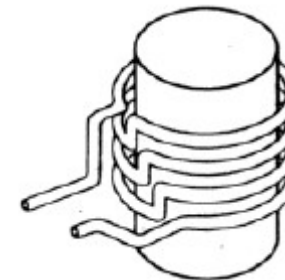
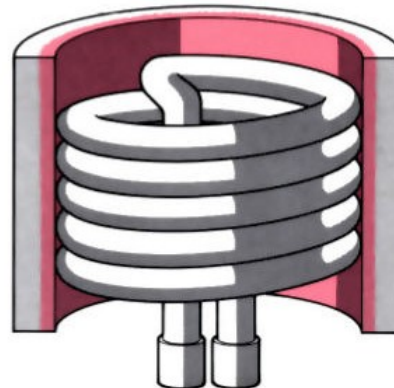
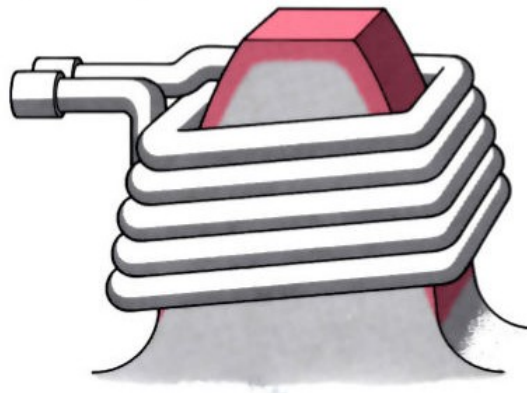
SPINNING HARDENING

Induction hardening

- In this process, heating of the component is achieved by electromagnetic induction.
- Used to surface harden crank shafts, cam shafts, gears, crank pins and axles.



Specially shaped coils (internal or external) are used for tubes or gear teeth.



- Here, an alternating current of high frequency passes through an induction coil enclosing the steel part to be heat treated. The induced emf heats the steel.
- The depth up to which the heat penetrates and rises the temperature above A_{c_3} is inversely proportional to the square root of the AC frequency. The depth of penetration of heat

$$d_{20} = 20 / \sqrt{f} \quad \text{At cold state, } T=20^\circ\text{C}$$

$$d_{800} = 500 / \sqrt{f} \quad \text{At hot state, } T=800^\circ\text{C}$$

f – frequency of current, d(mm)-depth of penetration

Due to heating of core by conduction of heat, overall depth of heating is greater than the given equation.

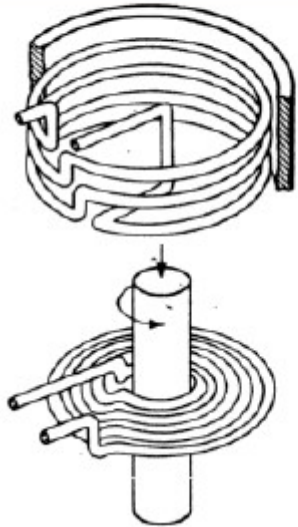
$$(d_o)_{800} = d_{800} + d_c$$

$$d_c = 0.2\sqrt{t}$$

t is heating time (seconds)

- Correspondingly, the hardened depth decreases with increasing frequency in induction hardening, the heating time is usually a few seconds
- Immediately after heating water jets are activated to quench the surface . Martensite is produced at the surface, making it hard and wear resistant.
- The microstructure of the core remains unaltered. Induction hardening is suitable for mass production of articles of uniform cross section

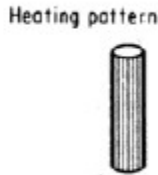
Induction Hardening



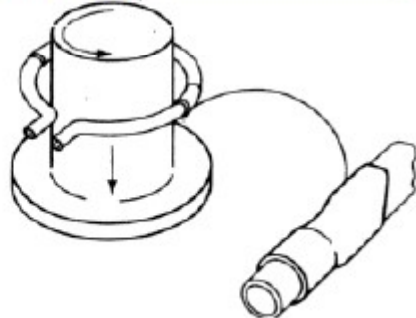
A coil to be used internally for heating bores



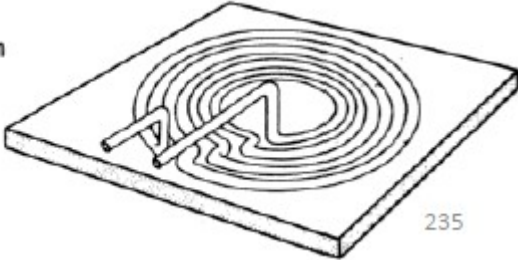
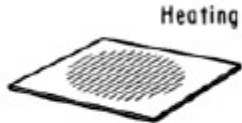
A "pie-plate" type of coil designed to provide high current densities in a narrow band for scanning applications.



A Single turn coil for scanning a rotating surface, provided with a contoured half turn that will aid in heating the fillet



A 'Pancake' coil for spot heating.



Laser Hardening

- Laser hardening treatment is widely used to harden localized areas of steel and cast iron machine components.
- This process is sometimes referred to as **laser transformation hardening** to differentiate it from laser surface melting phenomena.
- As laser beams are of high intensity, a lens is used to reduce the intensity by producing a defocused spot of size ranging from 0.5 to 25 mm. proper control of energy input is necessary to avoid melting.
- Laser transformation hardening produces thin surface zones that are heated and cooled very rapidly, resulting in very fine Martensitic microstructures, even in steels with relatively low hardenability.
- High hardness and good wear resistance with less distortion result from this process.

- Laser hardening has the advantage of precise control over the area to be hardened, an ability to harden re-entrant surfaces, very high speed of hardening and no separate quenching step (the quench is effected by the mass of the unheated material).
- The relationship between depth of hardening and power is as follows

$$casedepth(mm) = -0.11 + \frac{3.02P}{(D_b V)^{1/2}}$$

*P = laser power, D_b = Incident beam diameter
V = traverse speed (mm/s)*

- The disadvantage is that the hardening is shallower than in induction and flame hardening.

Electron Beam Hardening

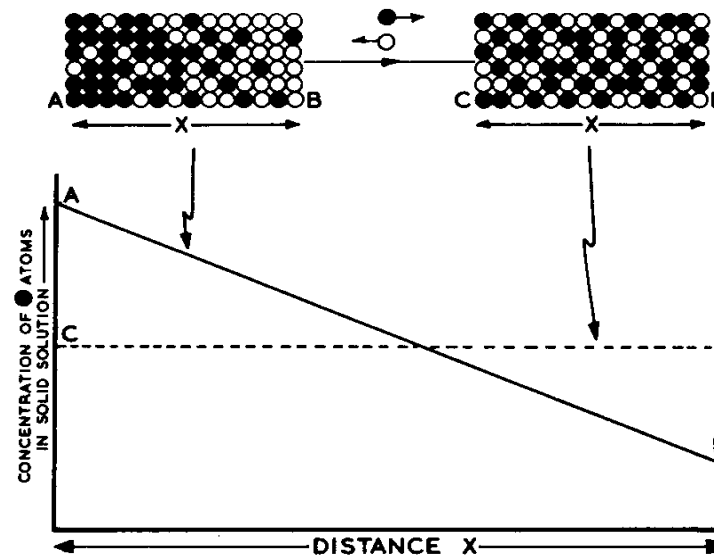
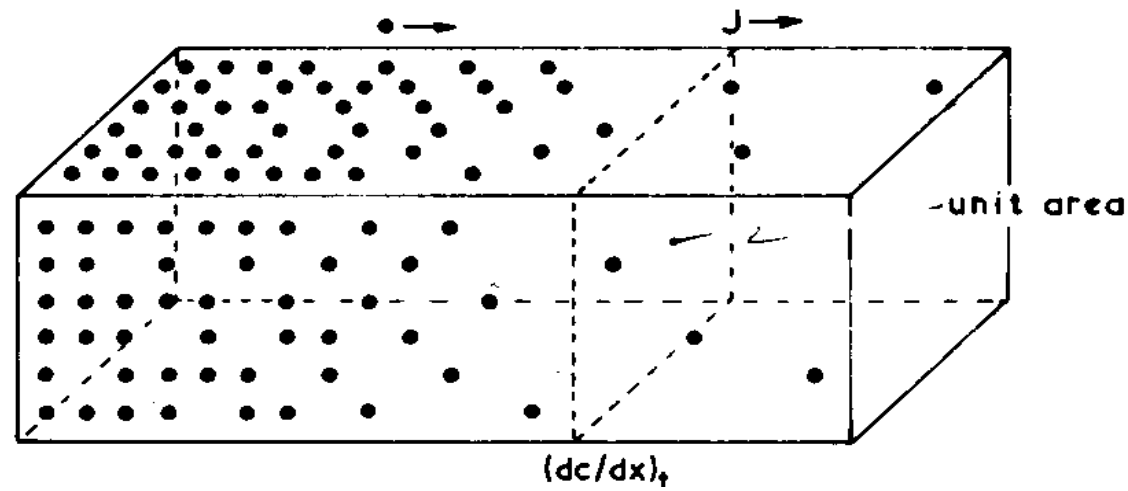
- This process is used for hardening those components which cannot be induction hardened because of associated distortion.
- Automatic transmission clutch cams (SAE 5060 steel) are hardened by this processes.
- Electron Beam (EB) hardening is like laser treatment, is used to harden the surfaces of steels.
- The EB heat treating process uses a concentrated beam of high-velocity electrons as an energy source to heat selected surface areas of ferrous parts.
- The electron beam passes through a focus coil, which precisely controls beam density levels (spot size).

- Require - high vacuum of 10^{-5} torr
- This vacuum environment protects the emitter from oxidizing and avoids scattering of the electrons
- No need for quenchants - self quenching.
- A mass of up to eight times that of the volume to be EB hardened is required around and beneath the heated surfaces.
- Normally, case depth upto 0.75 mm can be achieved by this method.
- A minicomputer is used to control voltage, current, beam time and focus.

Thermo-chemical Treatments

- Thermochemical treatments to harden surface of part (C, B, N₂)
- Also called **case hardening**
- May or may not require quenching
- Interior remains tough and strong

FICK'S LAWS OF DIFFUSION



- AB is the initial concentration gradient and CD the concentration gradient after a long time during which diffusion has been taking place.

FIRST LAW

$$\frac{dn}{dt} = -DA \frac{dc}{dx}$$

dn/dt = no. of moles of B atoms crossing per unit time

D= Diffusion coefficient

A= Planar area

dc/dx = concentration gradient

If J = flux flow / unit area per unit time,

$$J = \frac{1}{A} \frac{dn}{dt} = -D \frac{dc}{dx}$$

Carburizing

- Low-carbon steel is heated in a carbon-rich environment
 - Pack carburizing - packing parts in charcoal or coke -makes thick layer (0.025 - 0.150 in)
 - Gas carburizing - use of propane or other gas in a closed furnace - makes thin layer (0.005 - 0.030 in)
 - Liquid carburizing - molten salt bath containing sodium cyanide, barium chloride - thickness between other two methods
- Heated to temperature = 900-930⁰C for austenization (0.10 to 0.25% C → 0.7 to 0.9%C)
- Followed by quenching, hardness about HRC 60

Pack Carburizing

- Steel components + 80% granular coal + 20% BaCO₃ (energizer)
- Temperature: 930°C

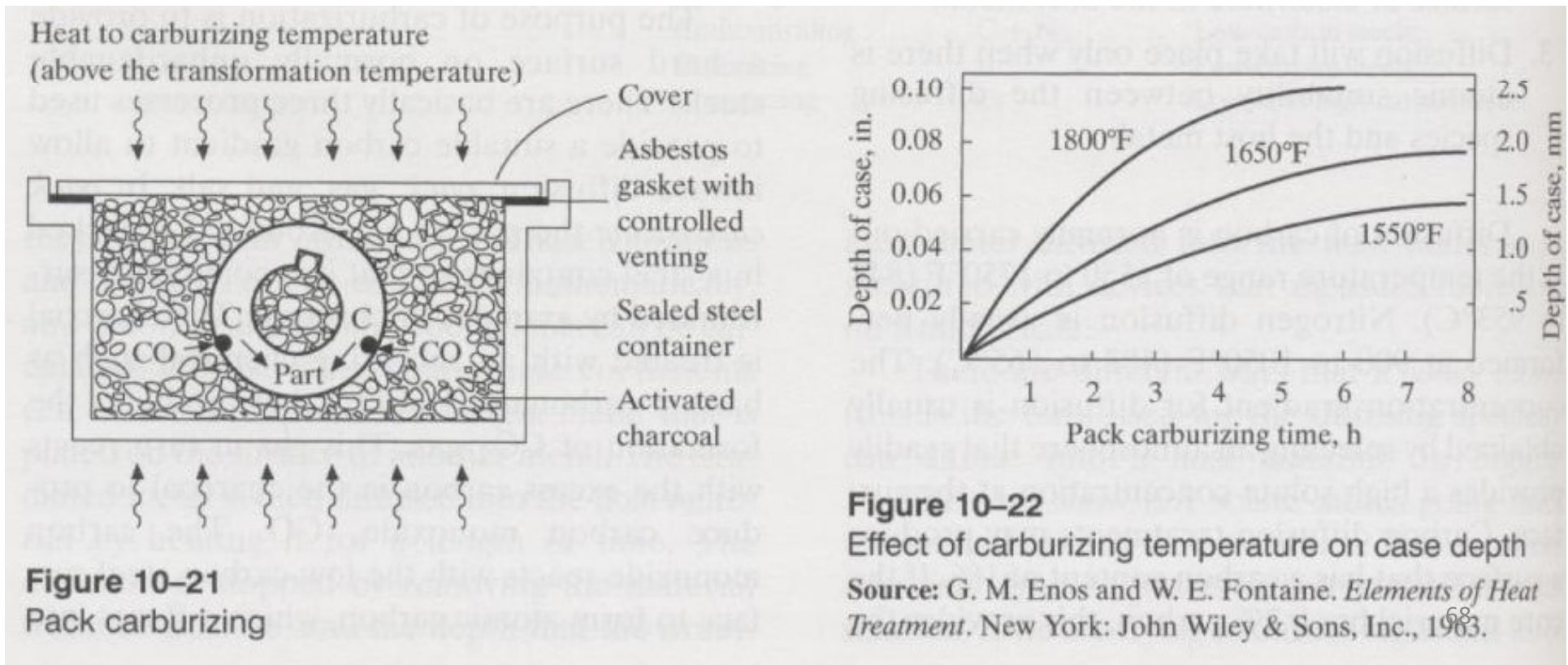
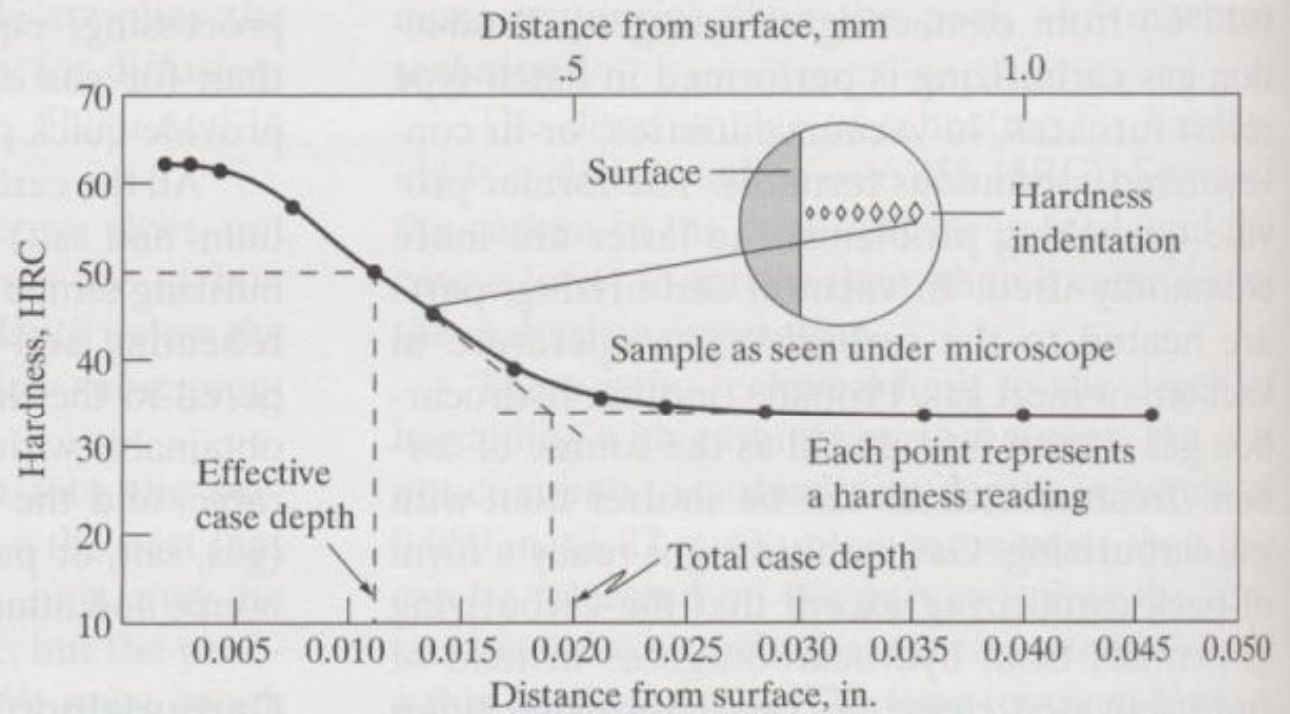
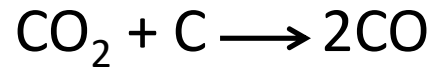
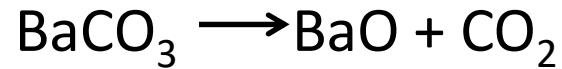


Figure 10-23

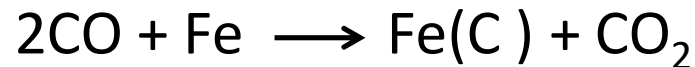
Determination of carburized or carbonitrided case depth by microhardness survey



1. Energizer decomposes to give CO gas to the steel surface with barium carbonate as energiser.

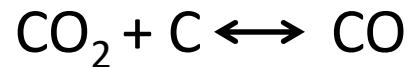


2. Carbon monoxide react with surface of the steel



3. Diffusion of C in steel

4. CO₂ formed in step 2 reacts with C in the coal



Depth of penetration of C at constant temperature depends of diffusion time t

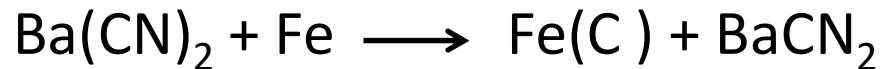
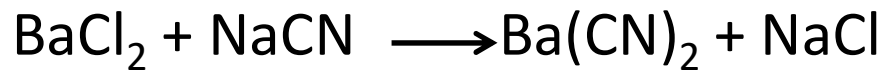
$$\text{Case depth} = k\sqrt{t}$$

Time = 6 to 8 hrs gives case depth 1mm to 2 mm. T and case depth control less than liquid and gas carburizing

- **Advantage:**
 - Cheap and simple method
 - Capital investment less compare to other method
 - No atmosphere controlled furnace and no poisonous cyanide or gas is used
 - Does not require skilled labour
- **Disadvantage**
 - Carburizing time is long
 - Difficult to control the surface C and C gradient
 - Difficult to control case depth exactly and uniformity
 - Maintaining clean atmosphere of work space is difficult
 - Difficult for direct quenching

Liquid Carburizing

- Mixture of salts of NaCN, NaCl+KCl, BaCl (activator)
- Melted in pots to 815- 900°C



- Heating time short and heat transfer is rapid. (work immersed for 5 min to 1 hour)
- This process produces thin and clear hardened layer
- For shallow- 0.08 to 0.25mm; for small parts
- Extensive safety precaution are required to avoid explosions.

- **Advantage**
 - High heat transfer efficiency of liquid bath
 - No decarburization occurs
 - Distortion is minimum
 - Capital cost and labour cost is less than gas or vacuum carburizing
- **Limitation**
 - Safety precaution has to be follow strictly due to handling of cyanide salts
 - Moisture on parts may cause explosions. Preheating is essential
 - Disposal of salts creates pollution problem.

Gas Carburizing

- Gas atmosphere for carburizing is produced from liquid (methanol, isopropanol) or gaseous hydrocarbons (propane or methane)
- Carried out in sealed quench type or continuous pusher type furnaces. Furnaces are either gas fired or heated electrically.
- Gas carburizing temperature: 870 to 950°C
- Carrier gas ($N_2+H_2+CO+CO_2+CH_4+H_2O+O_2$)
- Carrier gas is used to maintain a slightly +ve pressure and prevent oxidation of the steel during heating.
- When material reaches carburizing temperature, propane or methane is introduced to maintain a specific C potential

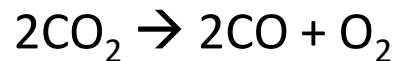
During gas carburizing, the following reactions take places:



Carburizing mainly occur by conversion of CO into CO₂.



Traces of O₂ are also present due to the following reactions:



Limitation:

- Cost of the equipment
- Requires skilled and well trained operators
- Supplying of liquid hydrocarbon in the furnace at 700°C

- **Advantage:**
 - More uniform case depth and accurate case depth
 - Cleaner and more efficient carburizing
 - Time requirement for carburizing less than solid or liquid carburizing
 - Direct quenching reduces the cost of heat treatment and increases productivity
- **Disadvantage:**
 - Capital cost of furnaces and gas generators is high
 - Trays and fixture are expensive
 - Skilled labour is needed
 - Toxic gases and fire hazards are to be carefully handled.

Vacuum Carburizing

- Process of carburizing take place in vacuum or reduced pressure
- It take place in two stages
 - Availability of carbon
 - Diffusion of carbon
- Sample loading → vacuum creation → heating the furnace to 925 -1050⁰C → introduction of hydrocarbon → dissociation of hydrocarbon → Diffusion of C
- Quantity of hydrocarbon depends on
 - Size of the component
 - Surface area of component
 - Depth of case required
 - Concentration of C introduced

- vacuum carburizing is an energy saving process due to
 - Heating carried out by radiation (improved efficiency due to vacuum)
 - Heat zone occupy less volume
 - It is not necessary to keep the furnace on throughout the process(can be heated and cooled rapidly)
 - Absence of atmosphere
 - The quantity of gas required about 1% of conventional process
 - Compared to conventional process surface is free from oxides, micro cracks and decarburization.

- **Advantages**

- Component can be quenched after carburizing
- Components are free from oxides, microcracks and decarburization
- It is an energy saving process
- Close control of case depth

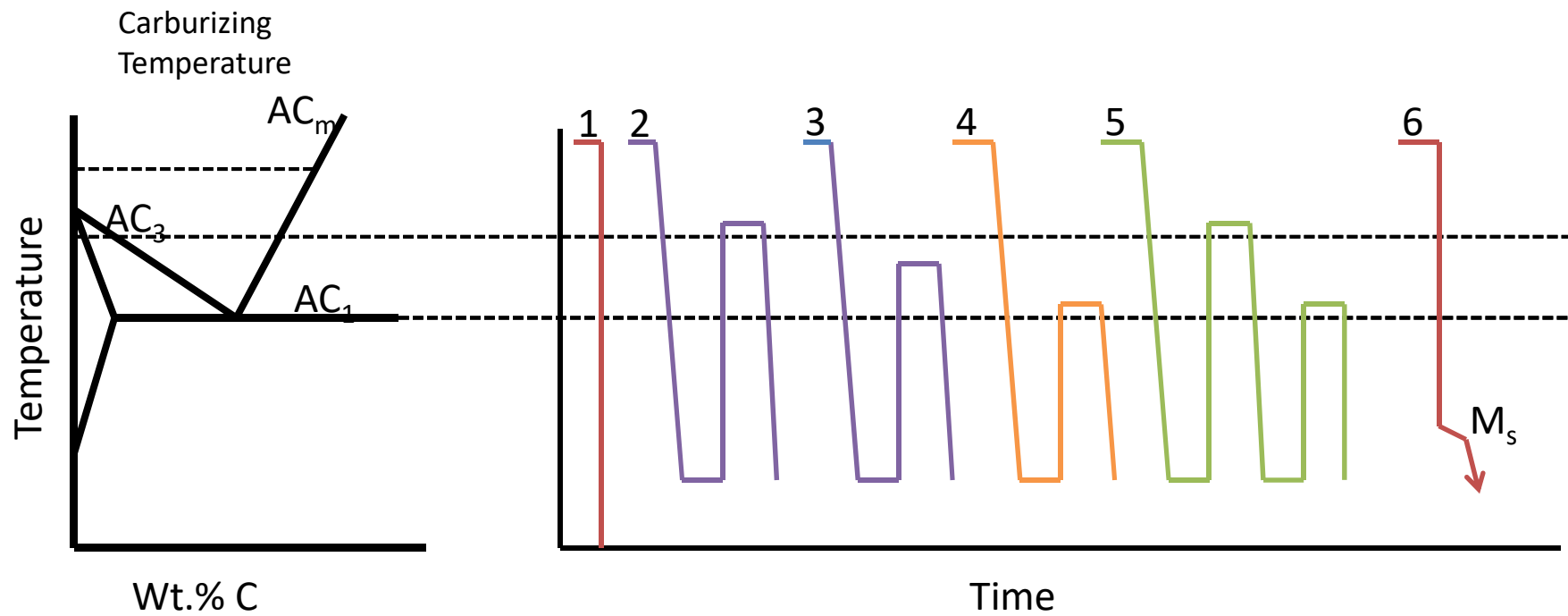
- **Limitation**

- Only batch type process
- Limitation on the size of the component due limited size of vacuum furnace.

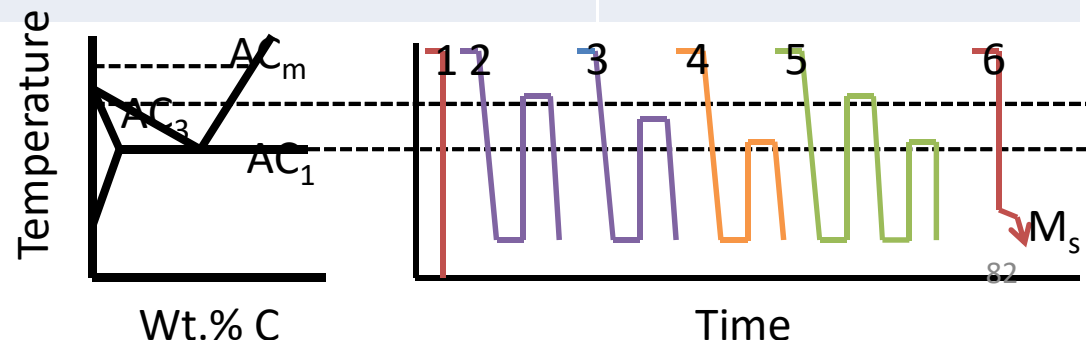
Post carburizing heat treatments

- To reduce the thermal stress on the surface (if martensite is formed on the surface)
- To refine grain size
- If network cementite is formed, it has to be broken

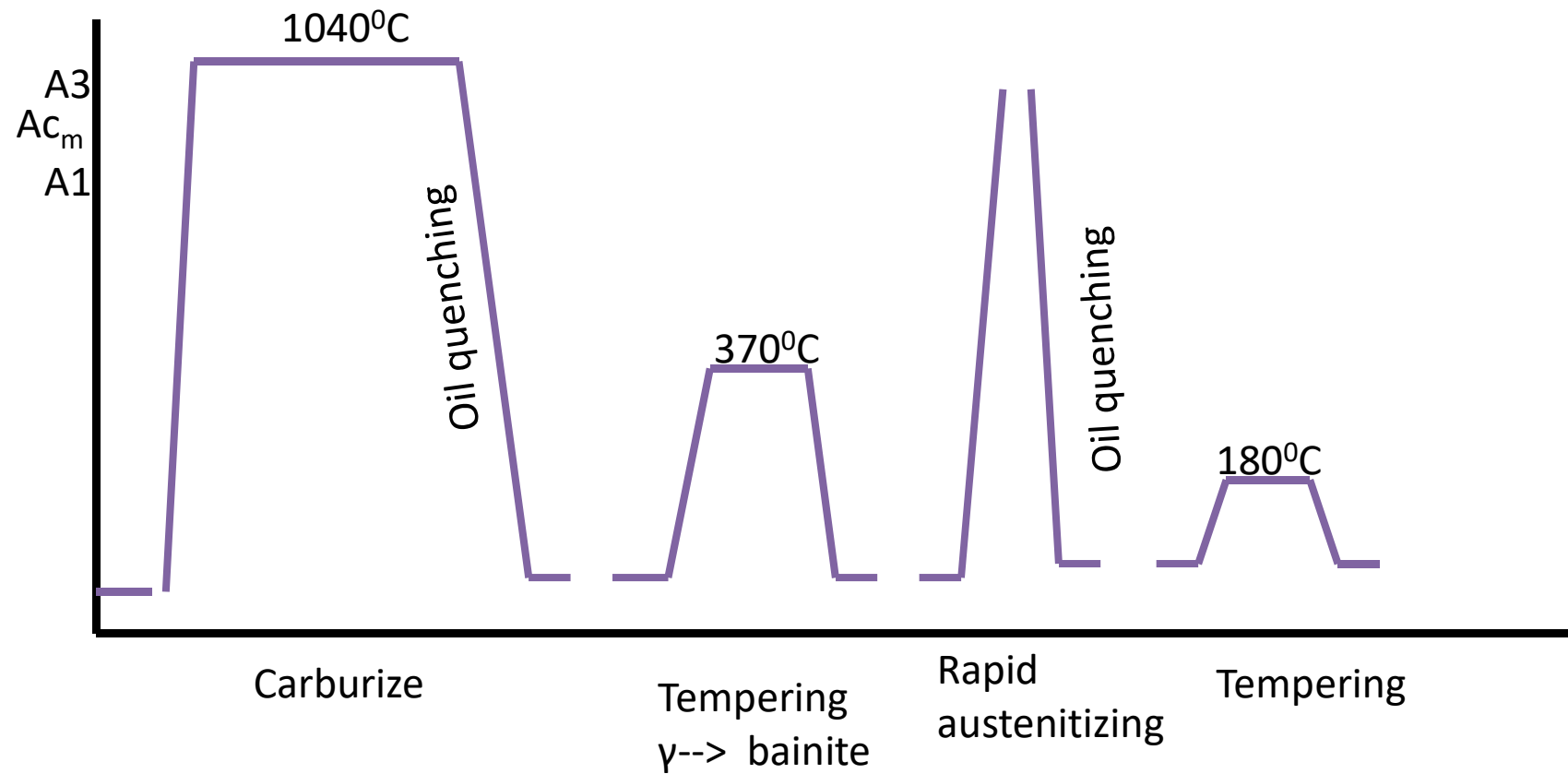
Schematic representation of post carburizing treatments



S.No.	Treatment	Case Characteristics	Core characteristics
1	Direct quenching	Unrefined, excess carbide gets dissolved; austenite is retained and distortion limited	Unrefined but hardened
2		Refined, solution of excess carbide favoured; austenite retention is promoted in highly alloyed steels	Refined, maximum core strength and hardness
3		Refined, some solution of excess carbide takes place (i.e. carbides get partially dissolved)	Partially refined, stronger and tougher than heat treatment 4
4		Refined, excess carbide is not dissolved	Unrefined, soft and machinable
5		Refined and hardened	Refined, low hardenability
6	Interurpted quenching	Unrefined, solution of excess carbide favoured; austenite is retained and distortion minimized	Fully hardened

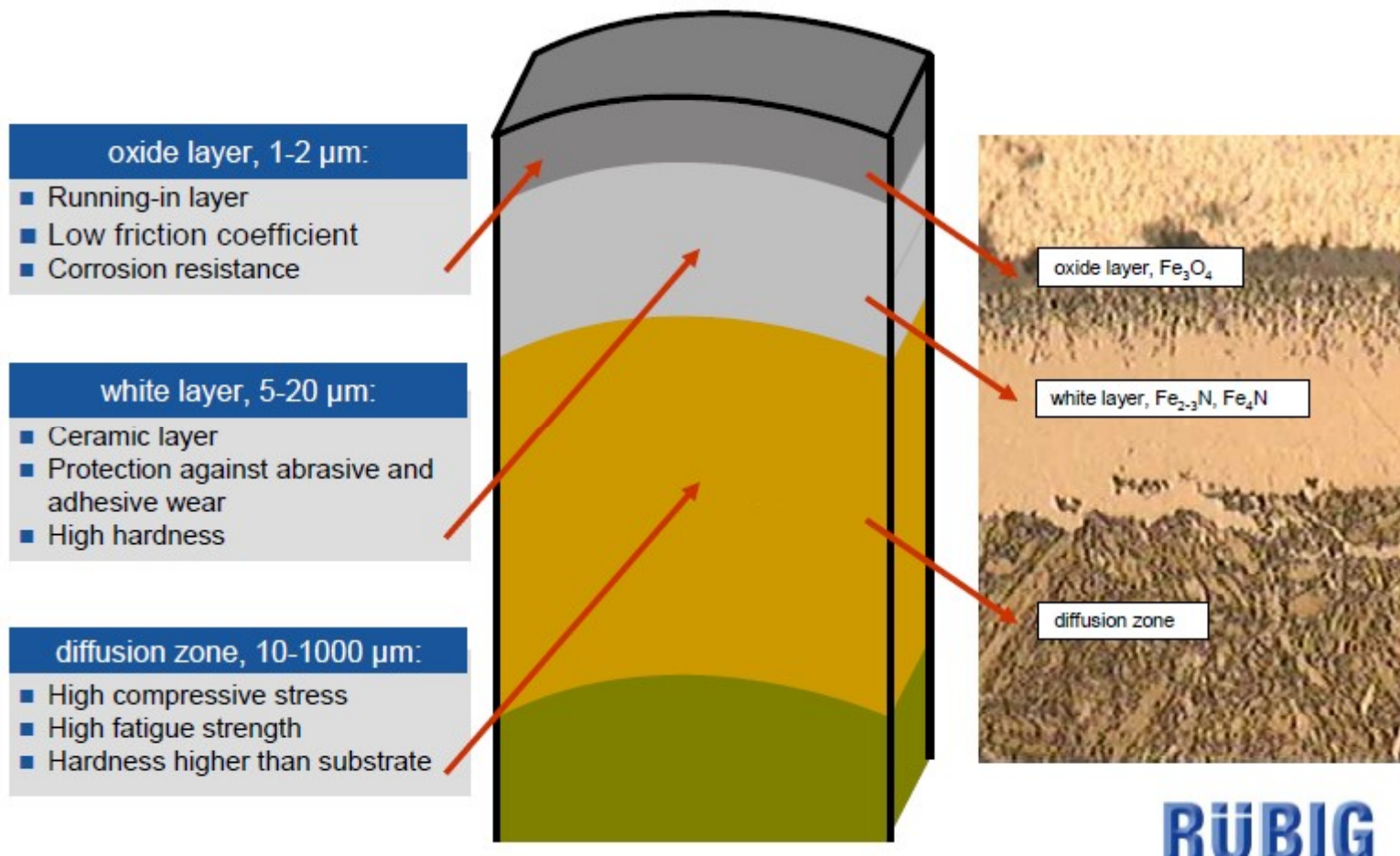


Procedure for maximum case hardness and very fine grain size



Nitriding

- The surface layer of the work piece is enriched with nitrogen by thermo chemical treatment.
- Nitriding preferable for alloy steels (**Al, Cr, Mo, V (these form strong nitrides)**)
- Quenching is not required
- Types of nitriding
 - Gas nitriding - heat in ammonia
 - Liquid nitriding - dip in molten cyanide bath
 - Plasma nitriding
- More expensive than carburizing
- **Hardness retained at 500°C (in carburising falls near 200°C)**



Cyaniding and Carbonitriding

- Steel with 0.3 to 0.4%C are hardened by addition of both C and N₂.
- C and N diffusion take place in liquid NaCN → Cyaniding
- C and N diffusion take place in gaseous atmosphere → carbonitriding
- Hardness → 850VHN

Cyaniding

- Components are heat treated by immersing in liquid bath (800-960°C) NaCN
- Concentration of NaCN → 25% to 90%
- Once temp. is reached, air is passed through the molten bath
- Reactions:
 - $2\text{NaCN} + \text{O}_2 \rightarrow 2\text{NaCNO}$
 - $2\text{NaCNO} + \text{O}_2 \rightarrow \text{Na}_2\text{CO}_3 + \text{CO} + 2\text{N}$
 - $2\text{CO} \rightarrow \text{CO}_2 + \text{C}$
- C and N diffuse into steel → wear resistant layer of **carbonitride (ϵ) phase**
- Process time = 30-90 min.
- Case depth is increased by raising the process temperature
 - 0.13 to 0.35 → T=850°C
 - 0.5 to 2mm → T=950°C
 - If temp. is increased diffusion of C is more than N₂.

- Advantage:
 - Due to liquid bath, bath temp. is uniform which reduces the distortion of the component
 - To improve the hardness, it can be quenched in oil
- Limitation
 - Not suitable for parts which required shock, fatigue and impact properties due to properties of the steel

Carbonitriding

- Used to improve the plain carbon and low alloy steels
- Temp: 800-870°C
- Gas mixture: 15%NH₃+5%CH₄+80%neutral carrier gas
- Diffusion of C and N in austenitic and ferritic condition
- Case depth → 0.05 to 0.75mm
- After carbonitriding, quenched in oil to avoid cracking followed by tempering (150-180°C)
- Surface hardenability, wear resistance and corrosion resistance are better than carburising
- Time required is more than carburizing

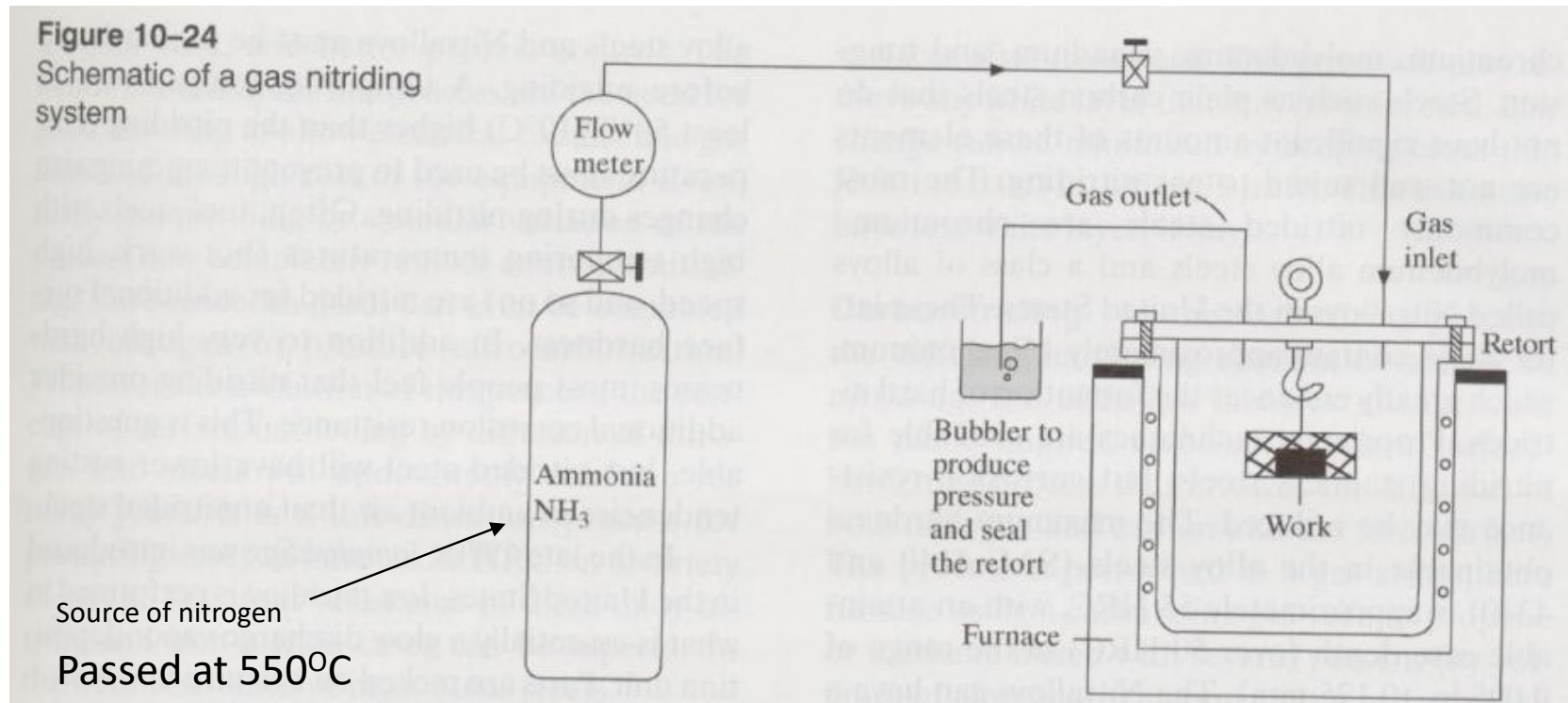
Gas Nitriding

- Temp: $<590^{\circ}\text{C}$
- No phase change after nitriding
- Before nitriding, Component (heated to 930°C and quenching in oil followed by tempering at 650°C) to bainitic structure which increases the strength and toughness of the core.
- The portion which does not require nitriding covered with thin coating of tin deposited by electrolysis

Gas Nitriding Furnace



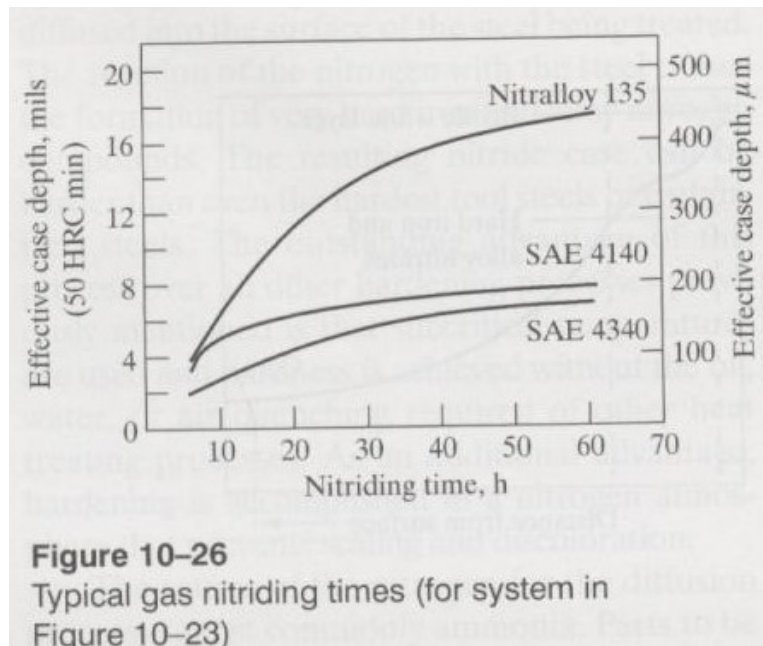
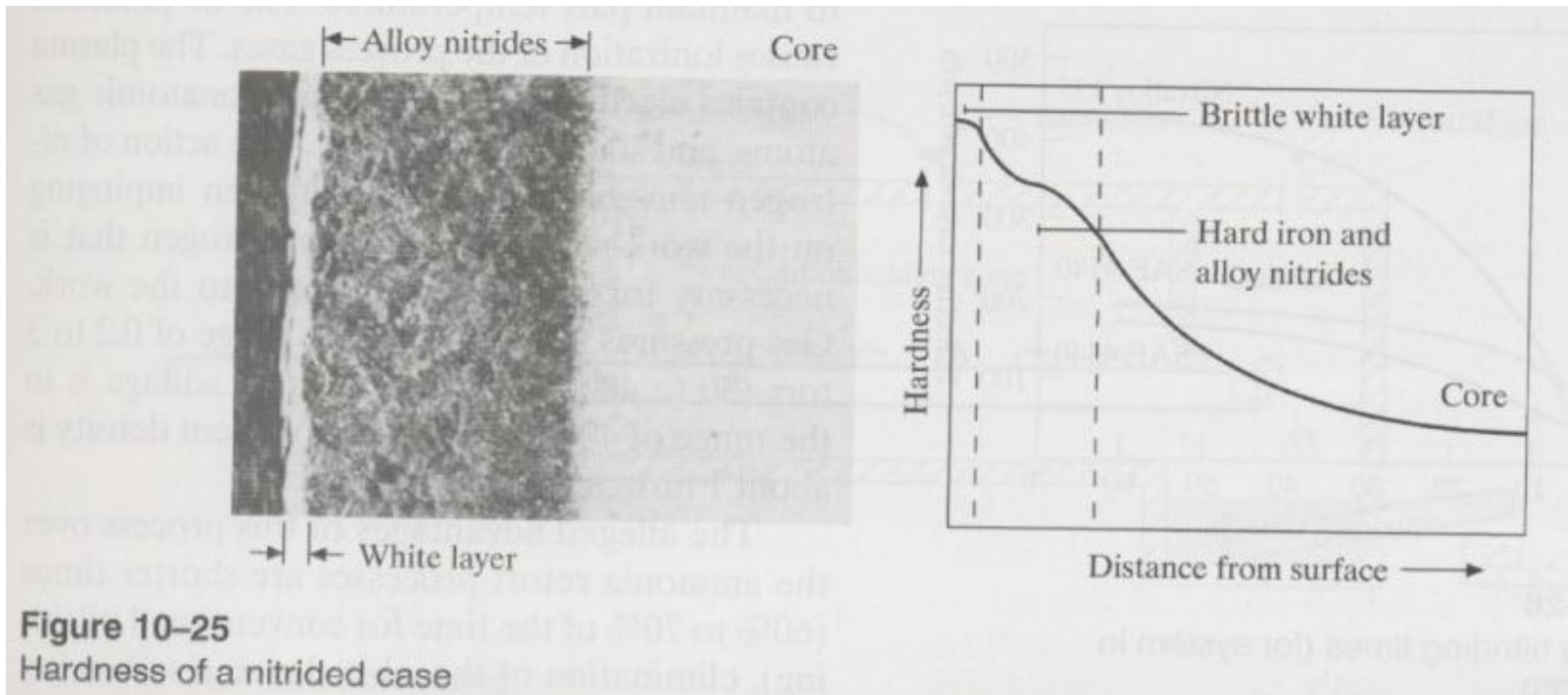
Figure 10-24
Schematic of a gas nitriding system



Reduction process: $2\text{NH}_3 \rightarrow 2\text{N} + 3\text{H}_2$

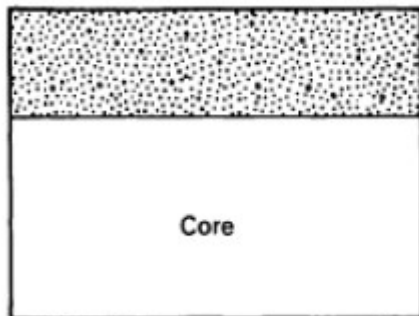
Time: 21 to 100 hours (for 0.5mm case depth require 100hours)

After nitriding, component cooled in the furnace itself in presence of NH_3



- Nitriding process produces
 - Fe_4N (γ' nitride)
 - Fe_2N (ϵ nitride)
- White layer [Fe_4N (γ' nitride) and Fe_2N (ϵ nitride)]
 - Very brittle
 - Formed due to high N_2 potential
 - Keeping desired level N_2 this layer can be avoided
 - Removed by mechanical polishing or by diffusion polishing
 - This layer can be suppressed by
 - Floe process or double stage nitriding
 - Plasma Nitriding (Ion nitriding)

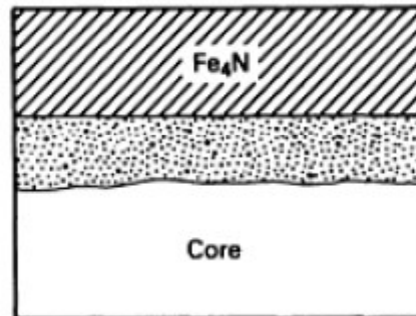
1-5% nitrogen
Balance carrier gas



Diffusion layer

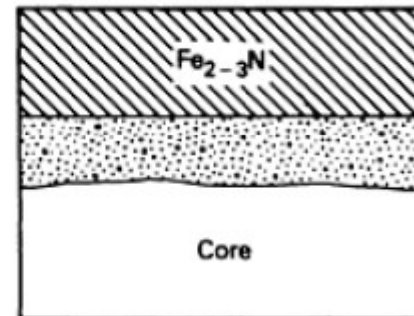
Gas composition

15-30% nitrogen
Balance carrier gas



γ' layer +
diffusion layer

60-70% nitrogen
1-3% methane
Balance carrier gas



ϵ layer +
diffusion layer

Floe Process or Double Stage Nitriding

- In this process, white layer first produced and then decomposed
- Carried out in two stage
 - Stage 1: Heat the component between 495 to 525⁰C for 15 to 20 hours.
 - White layer is formed in the first stage
 - 20% of NH₃ dissociated in this stage
 - Stage 2: Heat the component between 550 to 765⁰C
 - 75 to 80% NH₃ dissociated in this stage
 - Low N₂ potential is maintained
 - White layer formed in the first stage undergoes dissociation where it reduced from 0.05mm to 0.01mm or completely eliminated
 - Case hardness decreased compared to first stage but case depth increased

- Decomposition of white layer take place by three fundamental processes
 - Diffusion of N_2 out the white layer into the vacuum of the iron nitriding vessel
 - Diffusion of N_2 from white layer into the diffusion layer
 - Sputtering of the white layer by bombardment with inert gas

Plasma (or Ion) Nitriding

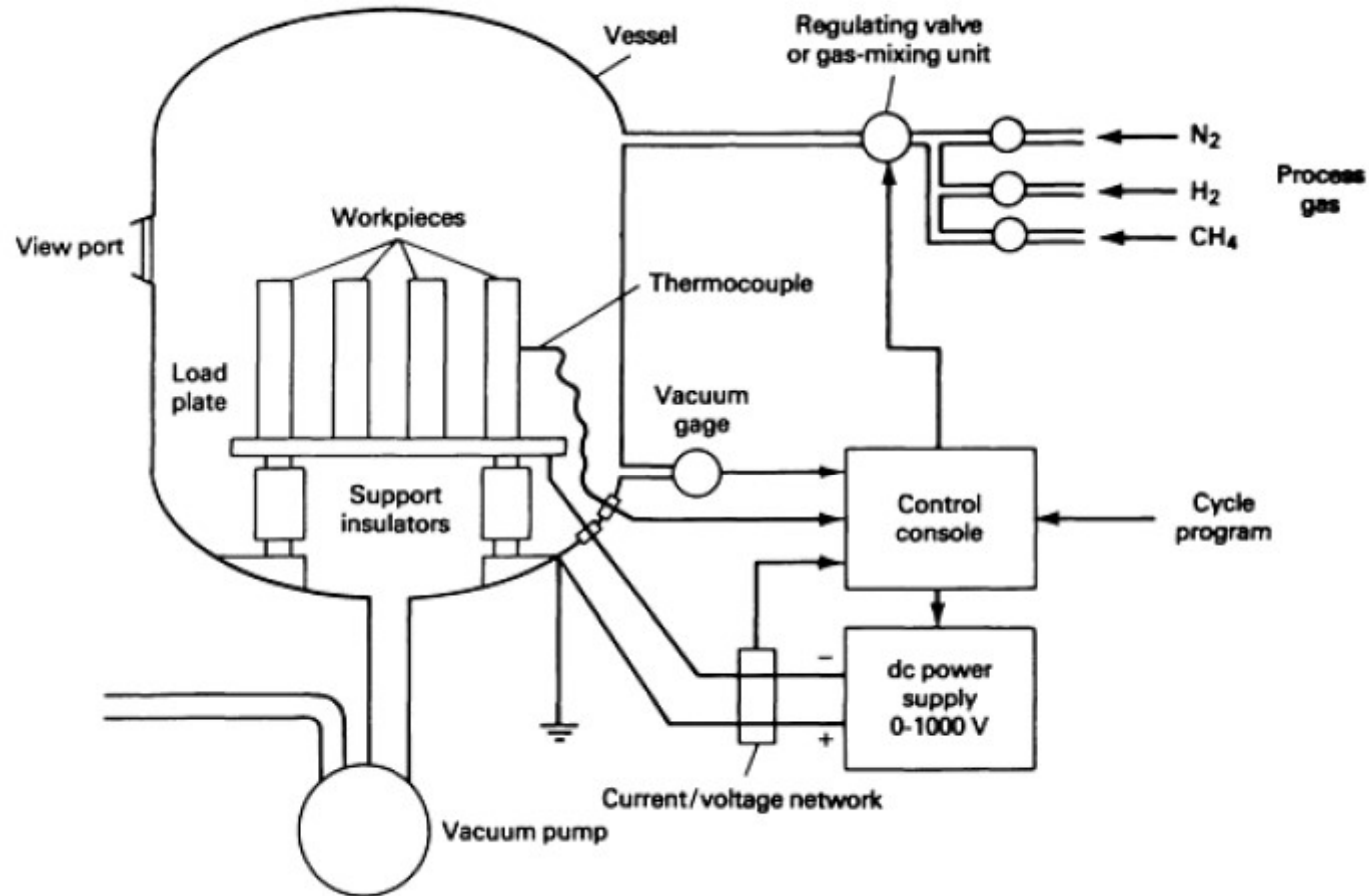


Fig. 5 Typical ion-nitriding vessel

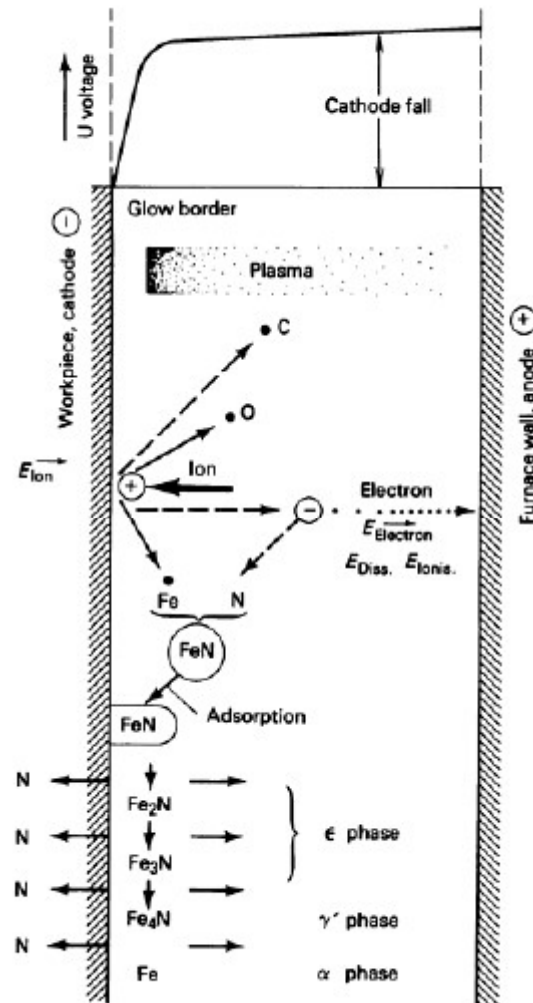
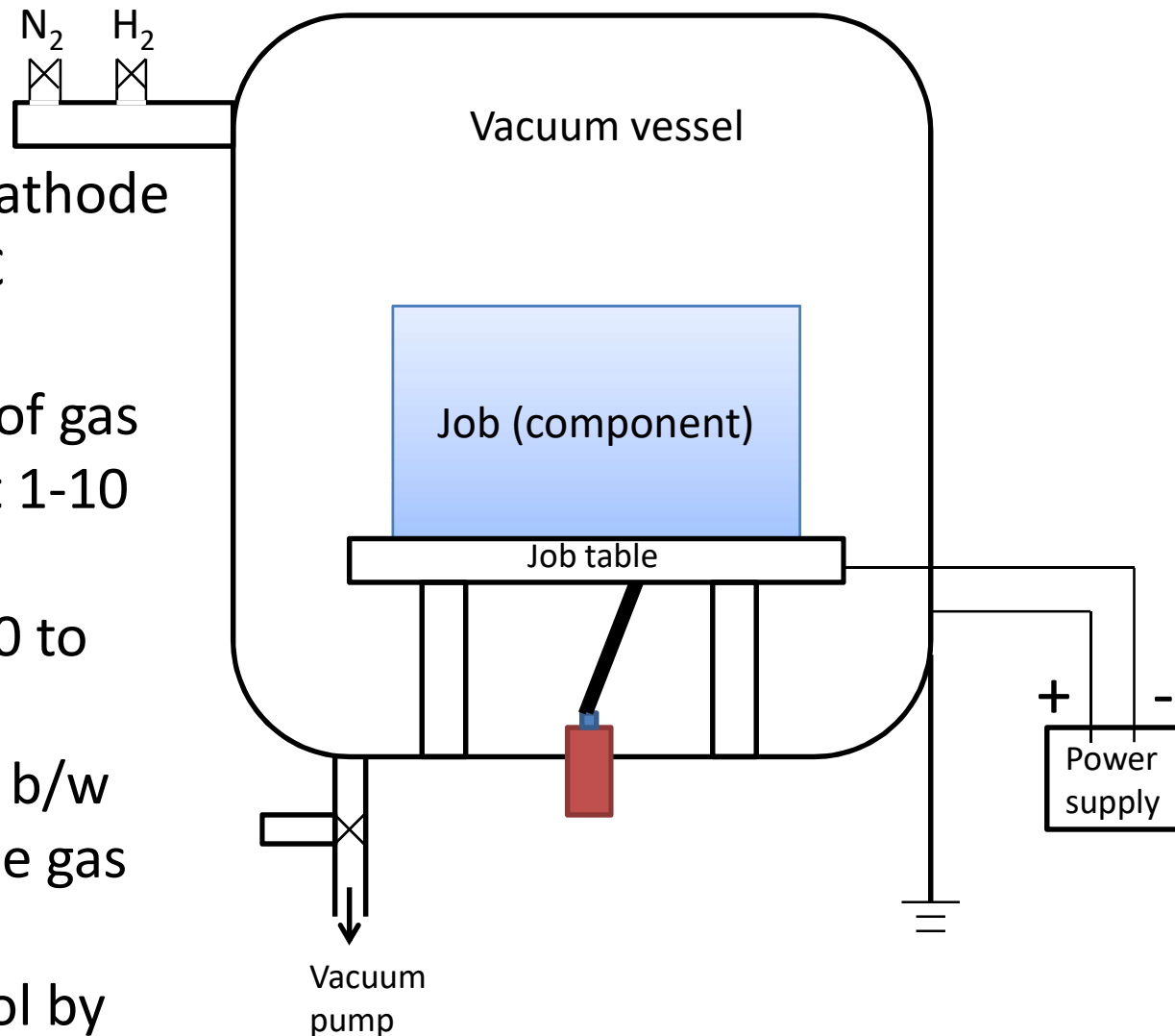


Fig. 7 Glow-discharge ion-nitriding mechanisms (Koelbel's model)

Plasma (or Ion) Nitriding

- Component act as cathode
- Temp.: 370 to 650°C (electrically heated)
- Followed by supply of gas mixture (N₂ and H₂) at 1-10 torr.
- High DC voltage (500 to 1000V)
- When current flows b/w cathode and anode the gas mixture gets ionized.
- The anode is kept cool by circulating water around it



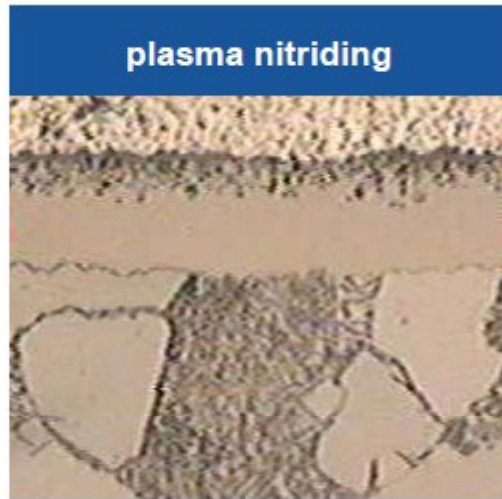
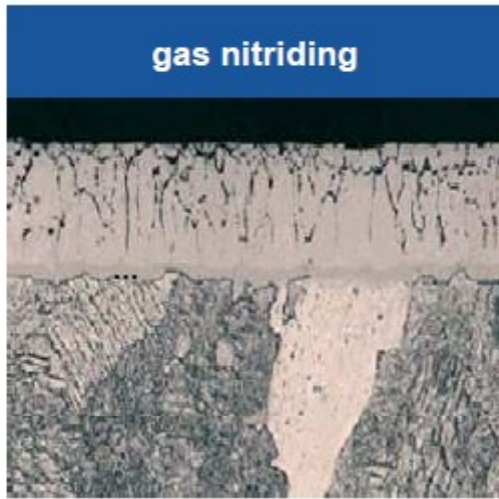
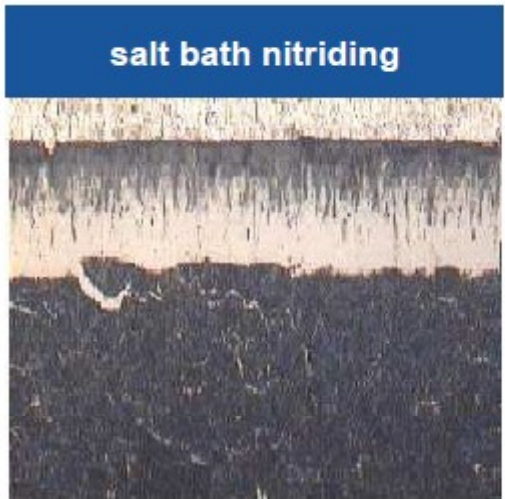
- When the part subject to ionized discharge then the energy
 - N ion bombard to the surface of the component
 - Heats the cathode
 - Diffusion of N take place
 - Displacing the secondary electron the surface which produce a glow over the component. (it indicates nitrides starts)
- Nitriding case depth depends on current, temperature and time of nitriding.
- After nitriding component is cooled in N₂ atmosphere.
- No white layer formation in plasma nitriding
- This process applicable for low alloy steel, tool steel and high alloy steel

Advantages:

- Different shape and size of the component can be nitrided
- Excellent dimensional stability with no distortion
- White layer formation can be Completely avoided
- Fatigue properties is very good
- Nitriding can be done at low temperature which is useful for cold work steel also
- Process is completely non-toxic and environmentally clean
- Low energy consumption and single step process

Limitations:

- Equipment is complex which require skilled labour
- Different size and shape of component can not be plasma nitrided in the same batch
- Rate of growth of case is less than salt bath nitriding
- Initial investment cost is high.



No GO
(for comparison purposes only)

- high friction due to rough porous zone
- post polishing necessary
- very good corrosion resistance

- low friction due to smooth porous zone
- no post polishing
- good corrosion resistance

gas nitriding

process temperature 530 – 600 °C

process gases for nitriding: NH₃, N₂

Carbon precursor for nitrocarburizing: CO₂

Gas consumption in an average furnace size: 4000 l/h

high influence of cleaning on nitriding result

NH₃ as Nitrogen precursor

plasma nitriding

process temperature 400 – 600 °C

process gases for nitriding: N₂, H₂

Carbon precursor for nitrocarburizing: CH₄

gas consumption in an average furnace size: 400 l/h

final cleaning by „sputtering“

N₂ as Nitrogen precursor

decreased energy demand

used gases not relevant for the environment

no greenhouse gas emissions

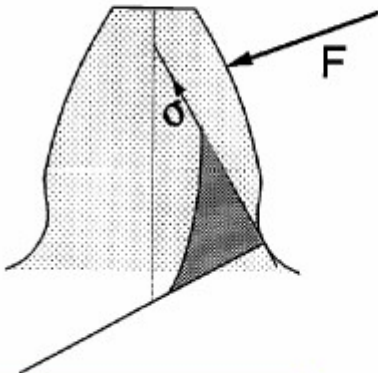
reduced pollutant emissions

reduced cleaning expenses

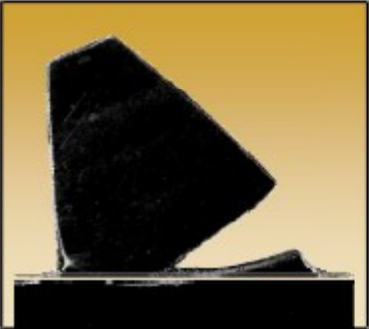
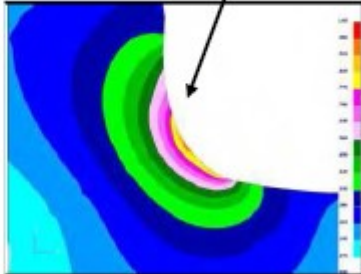
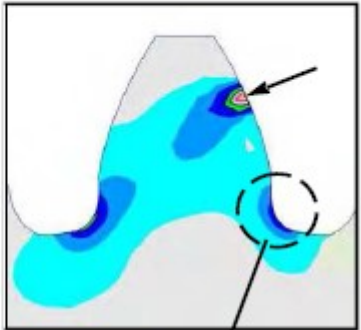
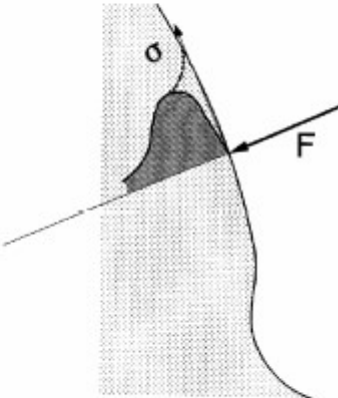
used gases not relevant for the health

LOAD AND STRESS PROFILE ON A GEAR

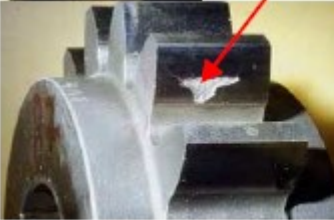
Tooth root



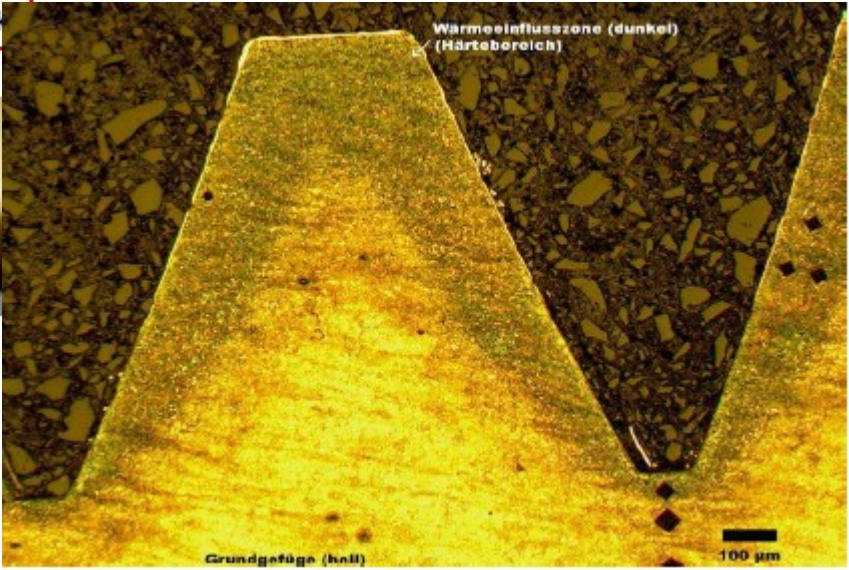
Tooth flank



Micro Pitting



Pit Mark

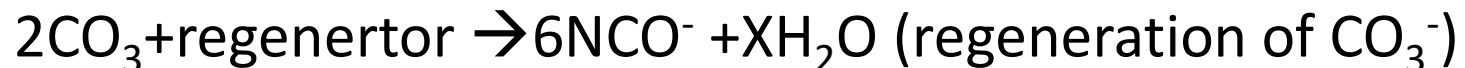
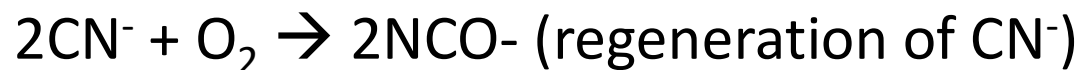
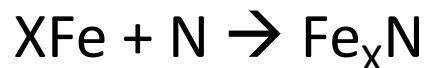
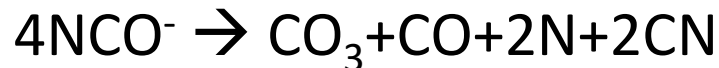


Salt Bath Nitrocarburizing/ ferritic nitrocarburizing

- Temp.:570-580⁰C in salt bath

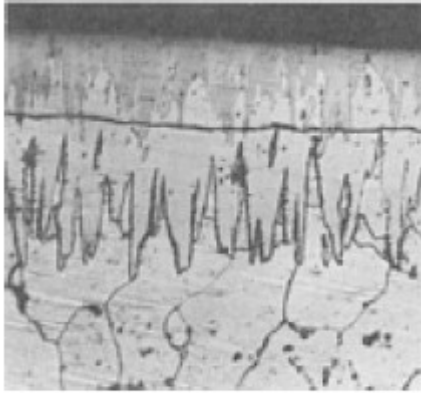


- With Cyanide:
 - Saltbath: NaCN+KCN+carbonates
 - Time: 1.5 hours →10-15μm (low carbon steel)
 - Reactions



- Advantages
 - Treatment time is short
 - Low temperature process (→ low risk for distortion)
 - No need of costly finishing work
 - Improves the surface hardness, fatigue strength and corrosion resistance at elevated temperature
 - Service life of the component is more
 - Applicable for any ferrous materials
- Limitation
 - Case depth is very less (very thin layer)
 - Salts are difficult to handle safely
- Applications
 - Gears, pinion shafts, piston rings, gun barrels, springs, crank shafts etc.

Boronizing



Separation of two phase boride layer on low carbon (St37) steel (borided at 900°C for 4 hr) caused by grinding with cutting-off disk. 200x.
FeB layer on the top followed by Fe₂B layer.

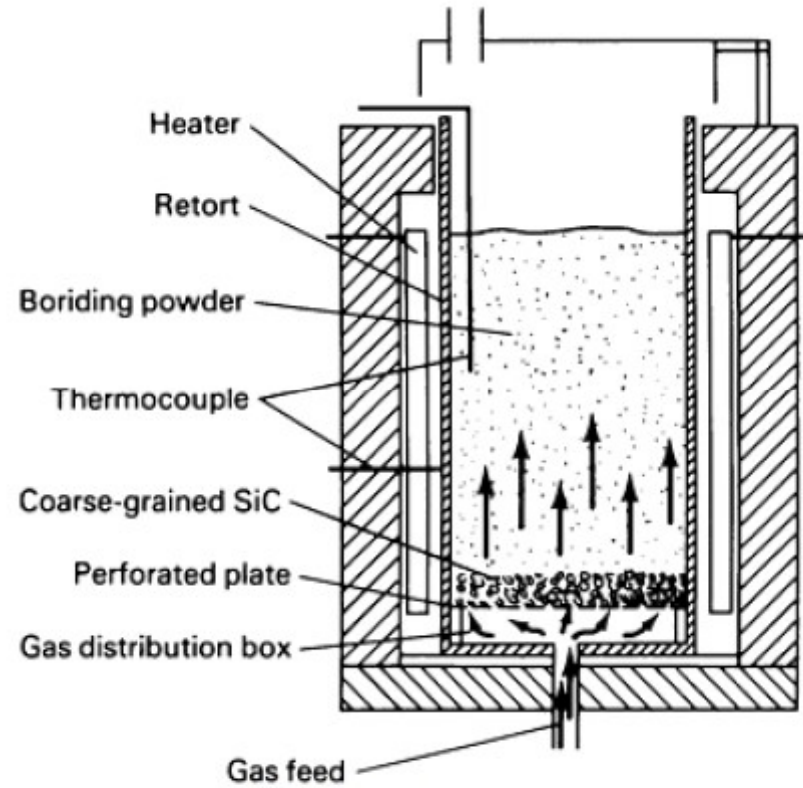


Fig. 12 Diagram of a fluidized bed for boriding. Source: Ref 4

Boronizing

- Diffusion of B
- Applicable for ferrous materials (carbon steels and tool steels)
- Processing types
 - Pack boronizing
 - Gaseous boronizing
- Hardness of boride layers is more than hard chrome electroplate, hardened tool steels and WC.
- Hardness 1500 to 2100VHN
- Case depth – 0.012mm to 0.127mm
 - 0.127mm → thick layer for abrasive wear
 - 0.025mm → for adhesive wear and friction reduction
- Boronizing increases the resistance to acids
- Boronizing increases abrasive, sliding and adhesive wear
- Improve the tools life

Pack Boronizing

- Temp: 900 to 1000°C
- Granules /paste of Boron carbide + activators + diluent
- $B + Fe \rightarrow FeB + Fe_2B$
- Fe_2B – inner layer
- FeB – outer layer which is brittle.
- High temperature, longer treatment time and high alloy steels favours the formation of FeB phase
- 0.15mm \rightarrow 900°C for 6 hours

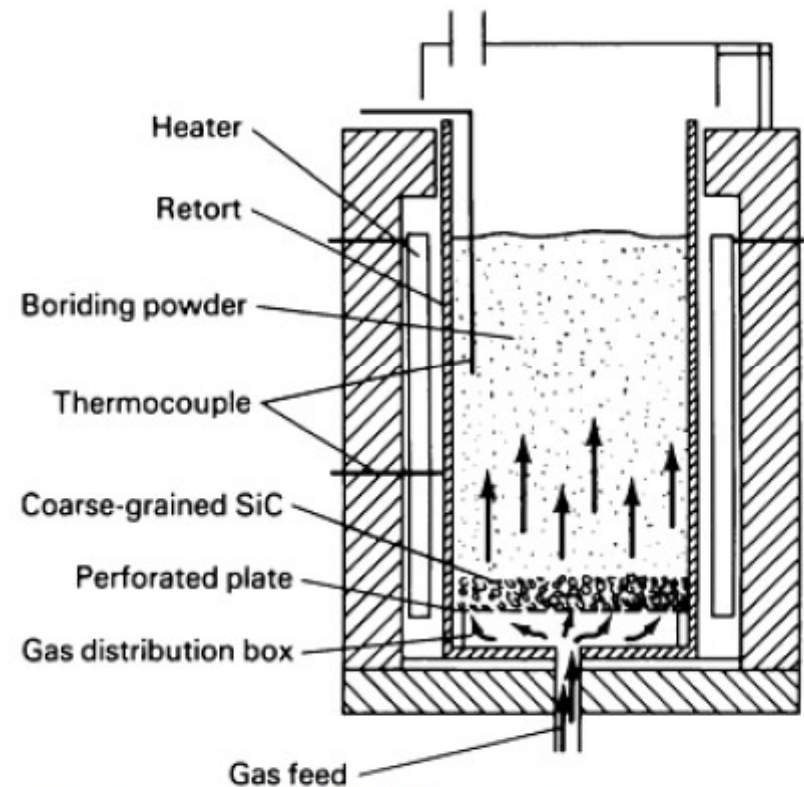


Fig. 12 Diagram of a fluidized bed for boriding. Source: Ref 4

Pack Boronizing

Chromizing

- Diffusion of Cr
- Two types
 - Pack chromizing
 - Gaseous chromizing
- Pack chromizing
 - Fine chromium powder (Ferro chrome)+ additives (ammonium iodide and Kaolin powder)
 - Temp:900-1020°C
 - Formation of chromium carbide on the surface
 - 0.02 to 0.04mm → 900 to 1020°C for 12 hours
 - Hardness 1500VHN
- Hard Chromizing
 - $C > 0.35\%$ (for steel)
 - Corrosion and wear resistant chromium layer is formed
- Soft chromizing
 - $C < 0.35\%$ (for steel)
 - Cr diffusion (NO formation of chromium carbide)
 - Case depth 200 μ m
 - Corrosion and oxidation resistant

Table 1 Melting point and microhardness of different boride phases formed during boriding of differer substrate materials

Substrate	Constituent phases in the boride layer	Microhardness of layer, HV or kg/mm ²	Melting point	
			°C	°F
Fe	FeB	1900-2100	1390	2535
	Fe ₂ B	1800-2000
Co	CoB	1850
	Co ₂ B	1500-1600
	Co ₃ B	700-800
Co-27.5 Cr	CoB	2200 (100 g) ^(a)
	Co ₂ B	~1550 (100 g) ^(a)
	Co ₃ B (?)	700-800
Ni	Ni ₄ B ₃	1600
	Ni ₂ B	1500
	Ni ₃ B	900
Inco 100	...	1700 (200 g) ^(b)
Mo	Mo ₂ B	1660	2000	3630
	MoB ₂	2330	~2100	~3810
	Mo ₂ B ₅	2400-2700	2100	3810
W	W ₂ B ₅	2600	2300	4170
Ti	TiB	2500	~1900	3450
	TiB ₂	3370	2980	5395
Ti-6Al-4V	TiB	
	TiB ₂	3000 (100 g) ^(a)
Nb	NbB ₂	2200	3050	5520
	NbB ₄	
Ta	Ta ₂ B		3200-3500	5790-6330
	TaB ₂	2500	3200	5790
Hf	HfB ₂	2900	3250	5880
Zr	ZrB ₂	2250	3040	5500

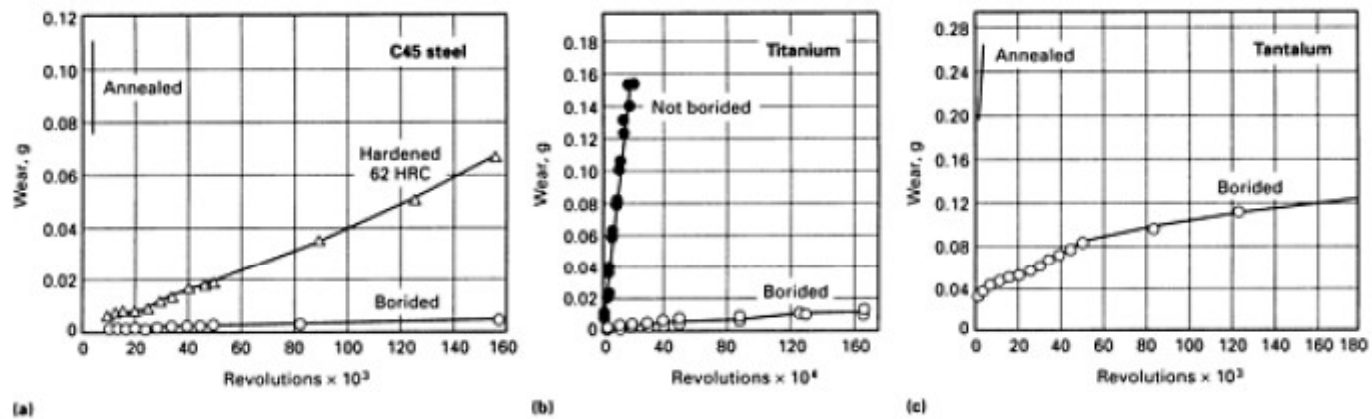


Fig. 2 Effect of boriding on the wear resistance (Faville test). (a) 0.45% C (C45) steel borided at 900 °C (1650 °F) for 3 h. (b) Titanium borided at 1000 °C (1830 °F) for 24 h. (c) Tantalum borided at 1000 °C (1830 °F) for 8 h. Source: Ref 11

Toyota Diffusion (TD) Process

- Carbide forming elements such as V and Nb diffusion take place in steel
- Salt bath used for diffusion of V and NB
- Temp.:1000°C
- Case depth 5 to 12μm
- Hardness 3000VHN
- Dimensional changes are possible during the process
- Advantage
 - High hardness, impact resistance and seizure resistance
 - Tool life increases
 - Lubricant requirements decreases
 - Applicable for automotive press tools and engineering components shafts, screws, bushes, blades, taps, pins and plugs

Process	Diffusion of interstitial atom	Material	Surface hardness (VHN)
Carburizing	C	Wide range of steel	850
Carbonitriding	C and N	Carbon steel	850
Nitriding	N	Cr-Mo steel	650
	N	Co-Mo-V steel	900
	N	Cr-Mo-Al steel	1100
	N	High alloy tool steel	1100
Induction and Flame hardening	-	0.4% C steel	500-600
	-	0.5% C steel	600-700
Bronizing	Br	Mild steel, Tool steel	1500
Chromizing	Cr	Carbon steel, tool steel	1500
Toyota diffusion process	V and Nb	Tool steel	3000-4000

Case depth Measurements

- **Case depth** (actual) is defined as “the perpendicular distance from the surface of the steel to the point at which the change in hardness, chemical composition or microstructure of the case and the core cannot be distinguished.
- Effective case depth is defined as the perpendicular distance from the surface of a hardened case to the farthest point at which a specified hardness value is obtained.
- The methods used for measuring case depth can be classified as:
 - Chemical method
 - Hardness method
 - Macrostructure method
 - Microscopic method

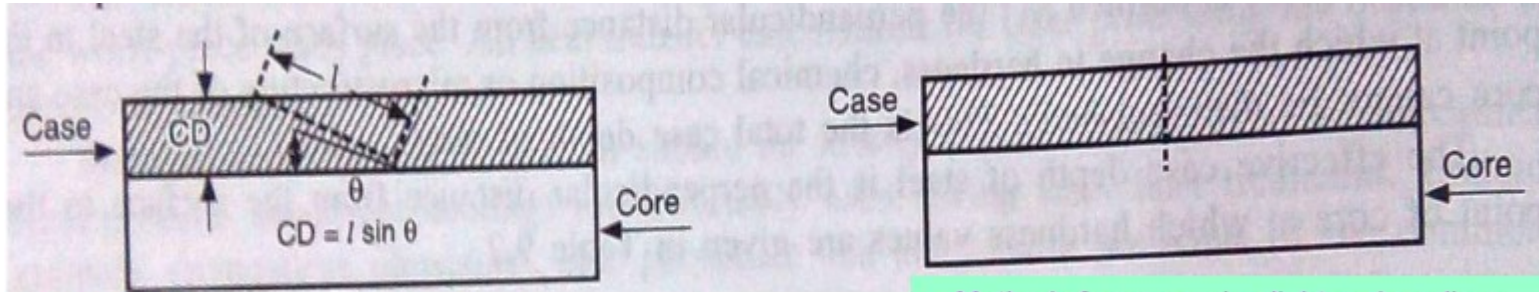
Chemical Method

- It is a usual practice to apply this method to carburized cases, nitriding and carbonitriding etc.
- For carburised cases, C content is determined at various depths below the surface of case hardened specimen, where hardening is due to formation of nitrides, N₂ content is estimated.
- This is the most accurate method of determining case depth.

Hardness Method

- In this method, hardness values are taken along the case and core.
- It is also a very accurate method since sharp change in hardness across case and core region can be measured.
- Specimens for this method are prepared by
 - Cross section procedure
 - Taper-grind procedure
 - Step-grind procedure

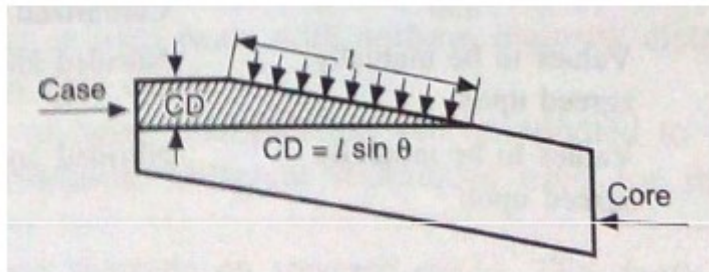
- Cross section procedure



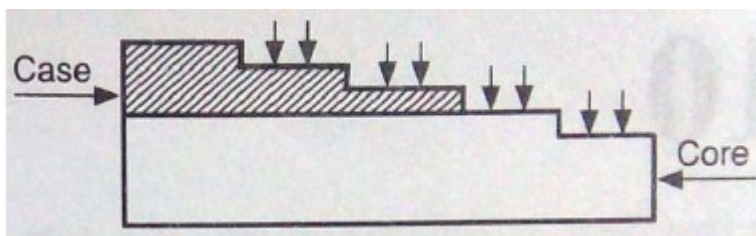
Methods for measuring light and medium cases

Methods for measuring light and medium and heavy cases 239

- Taper-grind procedure



- Step-grind procedure



Macrostructure Method:

- The test specimen is cut perpendicular to the hardened surface. After polishing and etching, the macrostructure of the sample is observed by 20x magnification.

Microstructure Method:

- Similar to macrostructure method, after sample preparation the case depth is measured from surface to the modified microstructure.