



Heat Treatment Process – An Overview

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The term heat treatment is understood to mean a change in the structure & consequently in the properties of an alloy accomplished by heating it to a definite temperature, holding at this temperature & subsequent cooling at a specified rate. Although pure metals can be hardened or softened by mechanical working or heating at high temperatures, the term heat treatment is applied to attain the properties in alloys only. Alloys are able to exhibit the change on heat treatment due to a phenomenon known as phase transformation particularly in “solid solution” phase. “Solid solution” is the phase in which one component of the alloy (A) retains its crystal lattice and hence called as ‘Solvent or Matrix’ while the other component (B), gives up its atoms to 1st one & called as “solute”. Two types of solid solutions are – ‘Substitutional Solid solution (SSS)’ & ‘Interstitial solid solution ISS’. In Substitutional SS, part of the A atoms are replaced by those of B, whereas in Interstitial SS the solute atoms are accommodated in the inter-atomic spaces of the solvent atoms.

Difference in the atomic sizes of solvent & solute atoms causes distortion of the lattice, causing change in mechanical properties. The process of acquiring atoms of 2nd component is influenced by the temperature and forms the basis of heat treatment process.

This article has been written with focus on Interstitial SS. Hence the heat treatments based on Substitutional SS, namely solution annealing & precipitation hardening are not described.

The Iron Carbon System

Heat processing of interstitial solid solution of carbon (C) in iron (Fe) two most important elements of steel has evolved an entire heat treatment industry. Heat treatment can be best understood through a phase diagram. Phase diagram is a graphical depiction of inter-relationship between phases, temperature and alloy composition under equilibrium condition. The Iron Carbon phase diagram given in fig. 1 depicts the existence of different phase as a function of % C in Fe at different temperatures.

Two important points in the Fe – C system have greatly influenced the heat treatment process namely:

- Change in the crystal structure of iron (Allotropy) with temperature &
- Large variation of Carbon solubility in Iron (from 0.016% at RT to 2.00% at 10950C)

Although plain carbon steel contains typically Manganese, Silicon, Sulphur & Phosphorus besides carbon, their presence in limited quantity has minimal effect on the phase diagram. However, phase diagram changes substantially with alloying additions.

During cooling of pure iron (with 0% carbon) the phase changes from

Liquid → BCC structure → FCC structure → BCC structure
 (Known as δ - ferrite) (known as γ- austenite) (known as α ferrite)

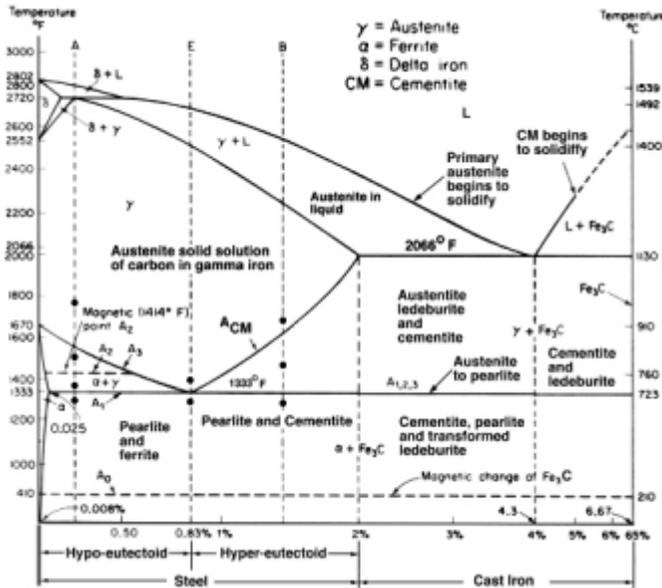


FIG 1 : IRON CARBON PHASE DIAGRAM

Structural changes which take place during the slow cooling are known as equilibrium transformation are reversible as during the process of slow heating, the changes take place in reverse direction. Alloying of Fe & C is a two step process. In the initial step Fe combines with 6.67% Carbon forming carbide called as cementite. Thus at room temperature a mixture of 2 phases exists in conventional steel – cementite & ferrite, which is essentially pure iron. On heating above 725 0 C, cementite dissolves in the matrix to form a new phase – austanite. On cooling the austanite throws out excess carbon. The process of discarding the excess carbon results into alternate layers (lamellae) of ferrite & cementite – the combination being known as pearlite.

As indicated by the austanite area, carbon content can vary from approximately 0% to 2%. On heating a steel with specific carbon content, the BCC structure transforms to FCC structure with the process of transformation starting from a critical temperature A1 and continuing up to other critical temperatures A3 or Am with intermediate temperatures showing mixtures of BCC + FCC. As mentioned above the phase from RT to A1 is a mixture of pearlite & excess ferrite. However, the mixture composition changes gradually, with increasing carbon % resulting in increasing pearlite content & decreasing ferrite – highest pearlite content of 100% being at 0.77% carbon. Increasing the carbon content beyond this point, results in a mixture of pearlite with balance being cementite. Composition of steel with 0.77% carbon is known as Eutectoid steel. The corresponding temperature called as eutectoid temperature is a single transformation temperature unlike a range of transformation temperature for other compositions. Accordingly, steel with lower & higher % carbon are known as ‘Hypo eutectoid’ & ‘Hyper eutectoid’ steels.

High temperature austanite phase with higher dissolved carbon is unstable at RT & will revert back to RT phase of Pearlite + ferrite / Pearlite / Pearlite + Cementite. This process of transformation can however be halted by arresting the dissolved carbon in the structure which in turn can be achieved by quenching the steel at a rate higher than



a critical cooling rate. The resultant structure known as ‘Martensite’ is hard & brittle phase with a BCT (body centre tetragonal) crystal structure, which has an elongated lattice dimension and about 4% higher volume.

Heat Treatment Processes

Based on this phenomenon of martensite phase formation, the following heat treatment processes have evolved:

Hardening : In this, the steel is heated to austanizing range and quenched rapidly to get hard martensite phase. The process does not give 100% conversion and approximately 5 to 8 % can remain as ‘retained austanite’ which is detrimental, as it affects the mechanical properties. However, 100% conversion can be achieved through sub-zero quenching or multiple post treatments.

Tempering : The hardened structure due to meta-stable martensite phase is not usable because of its brittle nature. A second stage heating below the critical temperature A1 (in the range 2500 to 7000C) gives a more stable tempered martensite structure. This, although lower in hardness, has better toughness. Depending on the end requirement, tempering temperature can be adjusted with high temperature tempering giving lower hardness & vice-a-versa.

Stress relieving : In addition to the formation of the meta-stable martensite, the sudden cooling, in hardening process, introduces large amount of thermal stresses which must be removed. This is achieved through a low temperature heating after hardening (at 1500 to 2500 C). This is known as stress relieving, as it gets rid of the thermal stresses without changing the hardness value. This treatment is typically applicable to tool steels.

Normalizing : If the austanized steel is allowed to cool in air, it gets a structure called as ‘bainite’ with properties similar to those of pearlite.

Annealing : Allowed to get cooled in the furnace itself, the steel gets back its original combination of phases, the process being known as annealing.

Quenchants

Many different types of quenching media such as – water, oil, brine solution, caustic solution, polymers, molten salts are used in the hardening process with the 1st three of the list being most common choice. Quenching power can be greatly increased through use of agitation. Typical cooling power of common quenchants is given in the following table. Cooling power of non-agitated water is arbitrarily taken as ‘1’ other media evaluated with the reference of this.

Circulation or Agitation	Quenching Power		
	Oil	Water	Caustic Soda / Brine
None	0.25 - .30	0.9 – 1.0	2
Moderate	0.35 0.40	1.2 – 1.3	==
Violent	0.8 – 1,10	4	5

Air quenching is also resorted to very high hardenability steels.

Furnace Classification Based on Heat Treatment Media

Heat treatment media are classified in 3 different categories namely – gaseous, liquid & solid. Various media under these categories are:

Gaseous : Conventional (air), fuel fired furnace (having specific combustion products of fuel), exothermic & endothermic gases, vacuum, cracked ammonia, neutral atmospheres e.g. argon, reducing atmosphere like hydrogen.

Liquid : Molten salt baths (in the temperature range 1750 to 12000C) help in complete protection from oxidation of parts when submerged.

Solid : Typical example of this is fluidized bed furnace where the part is immersed in the bed of inert material particle like alumina. These particles are suspended by passing gas generated by combustion of fuel & air. As the hot gases move up-ward, part appears to be on fluidised bed. Heating rate achieved in this can be 10 times higher than conventional direct firing.

Case Hardening

In many applications of metals, strength of metal is lesser importance compared to the wear resistance. In such cases, the surface of the part needs to be hard & wear resistant. In other cases, along with a hard surface, core is also required to be tough. These two conditions of metal are obtained by a specialised technique called “case hardening”. Surface hardening of steel is broadly divided into 2 types –

(A) Surface hardening technique without altering the surface composition &

(B) Surface hardening achieved through alteration of surface chemistry

A. Surface Hardening without Altering Surface Chemistry

Two methods of heat treatment practised are (a) Induction hardening & (b) flame hardening

(a) Induction Hardening : It is accomplished through thermal effects of high frequency current by placing the job in an alternating magnetic field. The job / work is placed in an inductor (called as heating coil) comprising of several turns of copper tubing. Alternating current passing through the inductor sets up alternating magnetic field which induces an eddy current in the work piece. This in turn gets heated by the joule effect. As the current mainly passes through the surface of the work piece, it is called as skin effect.

Thickness of the skin which gets heated is inversely proportional to square root of the frequency. With this heating, pearlite – austenite transformation is easily achieved. In fact heating rate in induction process (2000 – 2500 C) is substantially higher than that achieved in conventional heating process (30 – 50 C). This leads to quenching temperature range going 500 – 600C higher. In spite of this, near absence of holding time, results in a finer grain size.

This process is easily amenable to automation.

(b) Flame Hardening : This process is used for large sized & irregular shaped work pieces and is accomplished by heating the surface with high temperature gas flame. Typically acetylene, natural gas or kerosene is used as fuel for the flame. It requires strict control on the heating time, fuel & oxygen consumption. This process too can be automated.

B. Surface Hardening Achieved through Alteration of Surface Chemistry

This type of process is also known as chemical heat treatment. There are 4 basic processes in this category.

Carburising

Surface of the steel becomes chemically very active at elevated temperatures and readily absorbs elements in the atmosphere. Carburising or introduction of carbon in the surface is achieved by placing the work piece at high temperature in carbonaceous environment, with sufficient carbon potential. High temperature (in the austenite range) and the carbon potential difference between work piece & environment results in the diffusion of carbon in to the surface. Carbon diffusion can take place in any of the 3 media namely – gaseous, liquid or solid.

Gaseous Media : Natural gas with a carrier gas like endo-gas or nitrogen gas is used in this method of case hardening.

Liquid State : This method of carburising involves carbonaceous atmosphere achieved through molten salt baths, cyanide or non-cyanide baths.

Solid State : Also known as pack carburizing, this method uses atmosphere obtained by packing the job in solid compound that decomposes to give active carbon.

Vacuum Carburising : This is an advanced version of gas carburising wherein pressures lower than atmospheric are used (by pumping out the air & creating low vacuum) followed by filling the atmosphere with carburising gas.

Choice of the media is decided by size & shape of the job, accuracy required of the end result, production volumes and most importantly the economics.

Process temperature, difference in carbon potential of atmosphere & job and processing time are the important variables (in that order) of carburising process.

Carbonitriding

It is a modified gas carburising process wherein ammonia (up to 10 %) is introduced along with the carbonaceous gas. Ammonia dissociates at high temperature, releasing nascent nitrogen which diffuses into the surface. This process is used for jobs requiring shallower case depths compared to carburising as well as lower transformation temperature.

Nitriding

In this case hardening process, nascent nitrogen derived from cracking of ammonia is introduced in the surface. The process is carried out at much lower temperature (5000 – 5500C) and does not involve quenching. It has multiple advantages like low distortion, improved corrosion resistance in addition to increased wear resistance. As aluminium & chromium are strong nitride formers, steels containing these elements are most suitable for the application. Care should be taken while nitriding a quench hardened steel in that the tempering temperature should be at least 300 – 500C higher than the nitriding temperature. A variation of nitriding process includes – ion nitriding, gas nitriding & salt bath nitriding.

Nitrocarburising

The process involves simultaneous diffusion of nitrogen & carbon at temperatures below 6750 C. This being the ferrite region, the process is also known as Ferritic nitrocarburising. The process is available in many proprietary names. This process results in a thin top layer of iron carbonitride & nitride, which improves resistance to corrosion & wear along with an underneath diffusion zone which substantially improves the fatigue strength.