CHAPTER 7

Heat Treatment & Phase Transformation

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Introduction To Heat Treatment

Heat Treatment has been defined in handbook as a combination of heating and Cooling operations, timed and applied to a metal or alloy in solid state in a way that will produce desired properties, that are certain predetermined physical and mechanical properties.

As it is expected the desired properties which are needed by heat treatment, are mainly dependent on the microstructure of the alloy, i.e. nature, shape, size and distribution of the phases.

Heat Treatment Steps

According to the definition of the heat treatment by handbook the steps of heat treatment can be determined as given in the following steps:



Heating must be controlled to the desired temperature using a good insulated proper heating furnace under a suitable atmosphere to prevent the heat dissipation and the oxidation process.

Soaking is the essential step to insure that the temperature of component is equated between its surface and its core. This will depend on the dimensions of the heat-treated component.



Cooling will affect the resulted microstructure and then the obtained properties. Accordingly, cooling will be done either in the heating furnace (by switch off the furnace) or by leaving the heat-treated component to be cooled in a certain medium like air, oil or water. Occasionally, salt-bath and low-temperature melted metals are used as a medium for cooling step. 4

Heat Treatment Processes

التخمير 1.Annealing

It is a heat treatment process in which a material is taken to an elevated temperature, kept there for some time and then left to cool, usually, in the furnance.

Purposes of Annealing

Produce specific microstructure:

To relieve (relief) internal or residual stresses.

To increase softness, ductility, toughness and machinability.

Stages of Annealing

Heating to required temperature, which is defined according to the objective of the annealing.

Soaking at the required temperature by leaving the heat-treated material for *soaking of half hour for every 25 mm thickness*.

Cooling step is done slowly inside the furnace by switch off the furnace after the soaking. This will achieve about 50-100 °C/hr cooling rates. 5

Types of Annealing Processes

i. Stress relief annealing

It is used to eliminate and/or minimize stresses from arising plastic deformation during \mathcal{O}^{900} machining or forming processes. Stress relief annealing allows these stresses to relax. Annealing temperatures are relatively low so that useful effects of cold working are not eliminated. The given figure shows that heating temperature for the stress relief annealing is between 625-650 °C



ii. Recrystallisation and grain growth annealing

It is used to eliminate residual and internal stresses and create new 900grains that will be coarse grains. This process will lead to increase the ductility and th workability. As shown in the given figure, heating temperature for recrystallisation is about 650-670 °C



iii. Spheroidisation annealing

It is long soaking time heating below the eutectoid just temperature of the 727 °C 900(below A_1 and $A_{1,3}$) by about $\overline{\varphi}$ 25-30°C. This will produce soft Ξ spheroidite structure that operations. It is erformed formed for could be needed in subsequent forming usually performed for plain carbon steel of more than 0.45 wt%. The heating temperature range and the plain carbon steel composition that used by this process are shown in the given figure.



It has to be noted that in the previous annealing processes types, the heat-treated steel is heated to temperatures lower than A_1 or $A_{1,3}$ which mean that the steel is never reached to austenite region.

iv. Full annealing

It is heat treatment process essentially to produce soft steels suitable for all forming 900 and machining processes by heating up the steel to reach \overline{z} γ -austenite phase region, then \overline{z} the furnace. The resulting are go coarse pearlite and proeutectoid phase according to the carbon content of the steel. The heating temperature range of the full annealing is shown in the given figure as the following:



For hypoeutectoid steel $T = A_3 + (20-40) \circ C$

For hypereutectoid steel $T = A_{1,3} + (20-40) \circ C$

المعادلة 2. Normalizing

It is similar to annealing, except that during cooling stage the material is cooled at a faster rate than annealing usually outside the furnaces in air.

Purposes of Normalizing

• Generally to control more precisely a define grain size to produce finer pearlite mixture and finer proeutectoid phase.

Stages of Normalizing

In order to achieve the objectives of normalizing, heat-treated steel alloys are heated up to austenite region (austenitizing) according to the steel composition as the following

For hypoeutectoid steel: $T = A_3 + (40-80) \ ^{\circ}C$

For hypereutectoid steel $T = A_{cm} + (30-60) \ ^{\circ}C$

Then, the treatment is completed by cooling into air after predetermined *soaking time of 10-20 minutes for every 10 mm material thickness*.



It has to be noted that in case of *full annealing only 20 to 40 °C* above A_3 and $A_{1,3}$ is needed for softening of hypo and hyper eutectoid steels. On the other hand, 40 to 80 °C is needed for normalizing of the hypo-eutectoid steel above A_3 and 30 to 60 °C is needed above A_{cm} for hyper-eutectoid steel. Also, in case of full annealing it is forbidden to increase temperature of heating stage above A_{cm} for hyper-eutectoid steel to prevent the brittle Fe3C to be coarse phase as the result of the very slow cooling stage of the full annealing inside the furnace.

التصليد 3.Hardening

Hardening is the process of heating the steel to temperature sufficient to produce an austenite condition (not essentially austenitizing) depending on the carbon content of steel followed by rapid cooling at rate fast enough to prevent the transformation to any product phase(s) differ than martensite. This cooling type called quenching process.

Purposes of Hardening

The main objectives of steel hardening process are inducing high hardness and achieving high wear-resistance for steels. Hardening is done to all heavy-duty carbon steel machines parts and almost all machine parts made of all steel types.

Stages of Hardening

In order to achieve the objectives of hardening, plain carbon steels are heated up to temperature depending on the steel composition as the following:

For hypoeutectoid steel: $T = A_3 + (20-40) \ ^{\circ}C$

For hypereutectoid steel $T = A_{1,3} + (20-40) \ ^{\circ}C$



Introduction to phase Transformation

Phase transformations (change of the microstructure) can be divided into three categories:

- Phase transformations with no change in phase composition or number of phases present (e.g. melting, solidification of pure metal).
- Phase transformations with changes in phase compositions and/or number of phases (e.g. eutectic, peritectic and eutectoid reactions transformations).
- Phase transformations produced due to small displacements of all atoms in structure (e.g. Polymorphism and Allotropic Phenomena).

Objectives and Remarks

- The present studying is devoted to discuss the transformation involving in solid phases.
- Emphasis will be toward the phase transformation involved in the Fe-Fe₃C phase diagram due its familiarity and because it contains wide varieties of microstructures for Fe-Fe₃C alloys.
- Phase transformations do not occur instantaneously, the progress of the *phase transformation is depending on time*.
- The final structure often depends on *the rate of cooling/heating*. Therefore, it is required to consider *the time dependence or kinetics of the phase transformations*

Fe–Fe₃C Phase Diagram



Phase Transformation Stages (kinetics) Rates of solid state reactions

- Often phase transformation => Nucleation + Growth
- Nucleation =>formation of very small particles of a new phase
- Growth =>nuclei increase in size



Time-Temperature-Transformation Diagram (TTT Diagram) (Isothermal Transformation Diagrams) Introduction





The S-shaped curves are shifted to longer times at higher temperature ²⁰ indicating that the transformation is dominated by nucleation

In order to represent both the time and temperature dependence of the phase transformation, the bottom portion of the following figure is given. The vertical and the horizontal axes are, respectively, temperature and



- •At temperatures above the eutectoid and for all times, only γ -austenite will exist.
- •γ-phase to *pearlite transformation* will occur only if an alloy is *supercooled to temperature below eutectoid temperature of 727°C*.
- •The time necessary for the transformation to begin and then to end depends on temperature.
- •The *start and finish curves are nearly parallel*, and they approach the eutectoid temperature line.
- •At the left of the transformation start curve, only *unstable* γ *austenite* will be present, whereas at the right of the finish curve, only *stable pearlite* will exist.

•Between the start and the finish curves, the γ-austenite is in the process of transformation to pearlite, and thus both microstructures will be presented.



Several constraints are imposed on the figure:

- •First, this particular plot is valid only for Fe-Fe₃C alloy of eutectoid composition (0.77%C), for other composition the curves will have different configuration.
- In addition, these plots are accurate only for transformation in which the temperature of the alloy is held constant throughout the duration of the reaction. This Condition is termed isothermal transformation and the diagrams is called timetemperature-transformation (or T-T-T) plots.

T-T-T Diagram of the Eutectoid Fe₃C Alloy



•An actual isothermal heat treatment curve (*ABCD*) is superimposed on the isothermal transformation diagram for eutectoid Fe-Fe₃C alloy of 0.77% C.

•Very rapid cooling of γ austenite to a temperature is indicated by near-vertical line *AB*, and the isothermal treatment at this temperature is represented by the horizontal segment *BCD*.

•The transformation of γ -austenite to pearlite begins at the intersection of point *C* after approximately 3.5 seconds .

• γ -austenite is completed to pearlitic transformation after about 15 seconds, corresponding to point **D**.

Pearlite structures formed at different temperatures



Near eutectoid temperature Slow diffusion => coarse pearlite



Much below eutectoid temperature Fast diffusion => fine pearlite





Bainite Formation



- In addition to pearlite, other microstructure can be produced from the transformation of the γ-austenite. One of them is called bainite (acicular).
- As shown in the figure the time-temperature dependence of the bainite transformation may also be represented on the TTT diagram
- The following sign were used for each of the following phases: P for Pearlite and B for Bainite.



- The figure indicates that all transformation curves (beginning, half reaction and end) are Cshape with a nose at point N.
- As it may be noted from the figure, pearlite forms above the nose N in the temperature range of about 540 to 727 °C for the eutectoid alloy of Fe-0.77% C.
- Whereas for isothermal treatment at temperatures between about 215 and 540 C, bainite is the transformation product.

- The microstructure of bainite consists of a matrix of ferrite contains a distribution needles of cementite, Fe₃C, phase
- The following figure illustrates schematically the mechanism of bainite formation



(a) Upper Bainite



⁽b) Lower Bainite

- As shown in the figure bainite is nucleated by ferrite, which is followed by precipitation of cementite, Fe₃C. This process leads to a dispersion of iron carbide in a matrix of ferrite in the form of platelike or needles
- Higher transformation temperature below 540 °C leads to the so-called upper bainite, where coarse Fe₃C is distributed over a ferrite matrix.
- Lower transformation temperature below 540 °C near to 215 °C leads to finer distribution of Fe_3C

The following figure shows the microscopic graph of both upper and lower bainite.





Upper Bainite

Lower Bainite

Microscopic Photos of Upper and Lower Bainite



Upper Bainite



Lower Bainite

Microscopic Photos of Upper and Lower Bainite

Spheroidite Formation



Heating pearlite or bainite below eutectoid temperature for almost 18 to 24 hours, will lead to a transformation called spheroidite.



phase

Martensite Formation

In addition to pearlite and bainite other microstructure called martensite is formed when any austenitized $\text{Fe-Fe}_3\text{C}$ alloys are rapidly cooled (quenched) to relatively low temperature.

Martensite results from a diffusion-less transformation of γ -austenite, where carbon diffusion is prevented due to the rapid quenching rate.

Therefore, martensite is metastable phase, which it can persist indefinitely at room temperature, but will transform to equilibrium phases if it is heated at an elevated temperature.

In martensitic transformation, large numbers of atoms are encountering co-operative movements, in that there is only a slight displacement of each atom relative to its neighbours.

This occurs in such a way that the FCC γ -austenite is transforming to body-centred tetragonal (BCT) martensite



- As shown in the figure, all the carbon atoms remain as interstitial impurities in martensite phase.
- Martensite can coexist with other phases and/or microstructures like α-ferrite, pearlite and γaustenite in the Fe-C system.
- Martensite is metastable phase, it does not appear in Fe-Fe₃C phase diagram.







12 µm

Being the metastable lacksquarephase dose martensite not appears on the Fe-Fe₃C phase diagram, the γ -austenite to martensite transformation is represented on the isothermal transformation diagram (TTT diagram) of the eutectoid alloy (Fe-.077% C) as shown in the given figure



- Martensite transformation can be depicted in Figure .
- The beginning of the martensite transformation is represented by horizontal line designed M (start) at about 215 °C for • the eutectoid alloy.
- Two other solid horizontal and dashed horizontal lines, labeled M (50%) and M (90%), indicate percentages of the austenite to martensite transformation.
- The temperatures at which these lines are located vary with alloy composition as shown in the figure.
- The horizontal and linear character of these lines indicates that the martensitic transformation is independent of time; it is a function only on the temperature to which the alloy is quenched or rapidly cooled.



Effect of Carbon Percentage on TTT Diagram

1. TTT diagram for hypoeutectoid Fe-0.5% C alloy



2. TTT diagram for hypereutectoid Fe-1.1% C alloy



Application of TTT Diagrams

TTT diagram can be used to specify the nature of the final microstructure and approximate percentages of the phases that are existed at room temperature according to certain heat treatment path

Consider three case of heat treatment of Fe-0.77% C eutectoid steel rapid cooled from preheated temperature of 760 °C (>727°C) as follows:

- a) Rapidly cool to 350 °C, hold for 10^4 s and quench to room temperature;
- b) Rapidly cool to 250 °C, hold for 100 s and quench to room temperature;
- c) Rapidly cool to 650 °C, hold for 20 s, rapidly cool to 400 °C, hold for 10³ s and quench to room temperature;

In each case, the initial cooling is rapid enough to prevent any no required transformation



a) At 350 °C γ transforms to **B**; the reaction starts after 10 s and ends at 500 s. By 10⁴ s, 100% of the specimen is bainite and no more transformation is possible.

- b)At 250 °C, at 100 s the specimen is still 100% γ. As the specimen is cooled, M is started at 215 °C. Transformation is complete by the time until room temperature is reached at 100% martensite.
- c)At 650 °C, P begins after 7 s; after 20 s 50% of γ is transformed to P. During rapid cool to 400 °C little of the remaining γ transforms to either **P** and/or **B**. At 400 °C and after 10³ s, the remaining 50% γ will have completely transformed to bainite

Continuous Cooling Transformation Diagrams (CCT)



- Heat treatment using isothermal transformation diagrams is not rather practical especially for bulk samples.
- Most heat treatments involve continuous cooling
- For continuous cooling, the time required for a reaction to begin and end are delayed as shown in the figure
- Isothermal curves are shifted to longer times and lower temperatures.
- Normally, bainite will not form during continuous cooling of plain carbon steel



The given figure indicates two cooling curves for eutectoid steel corresponding to:

slow rate

moderately fast rate

As shown in the figure, the transformation starts period after time corresponding the to intersection of the cooling curves with the beginning reaction curve and ends crossing the upon completion transformation curve.



In case of continuous cooling transformation diagram, critical cooling rate for martensite formation of the eutectoid steel can be defined to be 140 °C/s for 0.76% C.

Also the figure indicates cooling rates slower than 35 °C/s are essentially to produce only pearlite microstructure

Critical cooling rate depends on steel composition. Lower carbon content shifts the nose to the left. Therefore, lower carbon steels require higher cooling rates for martensite formation

Mechanical Behavior of Fe-C Alloys (Structure and properties)

Cementite is much harder but more brittle than ferrite. Therefore, increasing the fraction of Fe_3C in a steel alloy while holding other microstructural elements constant will result in a harder and stronger material as well as less ductile and less tough (toughness) (المتانة) or impact energy material.



The layer thickness of each of the ferrite and cementite phases in the pearlite microstructure also influences the mechanical behavior of the material. Fine pearlite is harder and stronger than coarse pearlite.

Coarse pearlite is more ductile than fine pearlite.



Bainitic steels microstructure has smaller dispersed fine cementite needles particles in ferritic matrix. Therefore, the finer structure steels with bainite structure are stronger and harder than pearlitic ones



Martensite and Tempered Martensite Properties

Of the various microstructures that may be produced for a given steel alloy, martensite is the hardest.



The given figure plots the hardness of martensite and fine pearlite, for comparison, as a function of carbon weight percent and Fe₃C cementite fraction percent.

- Because martensite is so brittle, it has negligible ductility. Therefore, it needs to be modified for practical applications. A process called tempering ((a = b = b)) does this modification. Martensite is heated below A₁ (lower than eutectoid temperature) to usually about 250-650 °C and then it is kept at the chosen temperature for predetermined time period.
- Therefore, it needs to be modified for practical applications. A process called tempering (A = A = A) does this modification. Martensite is heated below A_1 (lower than eutectoid temperature) to usually about 250-650 °C and then it is kept at the chosen temperature for predetermined time period.
- This will produce the so-called tempered martensite, which is an extremely fine dispersed cementite grains in a ferrite matrix as shown in the figure according to the following reaction equation:







Martensite (BCT, single phase) Tempered martensite (α +Fe₃C)₄₇



The dependence of the tensile and yield strength and ductility (reduction in area %) on tempering temperature for martenstic oil-quenched plain carbon steel is shown in the following figure.



Exercise 2

How to plot log scale?

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1. Using the TTT diagram of the 0.45 wt% C steel alloy shown in Fig. 1, determine the final microstructure developed during the following time-temperature treatment which are carried out on small 0.45 wt% C steel specimens. Note that all specimens are heated up to 845°C, and held at this temperature long time enough to achieve a complete and homogeneous γ -austenitic structure, then they are cooled according to the following sequences:

a) Rapidly cool to 250°C, hold for 10³ s and then quench to RT



b) Rapidly cool to 700°C, hold for 30 s and then quench to RT



c) Rapidly cool to 400°C, hold for 500 s and then quench to RT



d) Rapidly cool to 700°C, hold for 10⁵ s and then quench to RT



e) Rapidly cool to 650°C, hold at this temperature for 3 s, rapidly cool to 400°C, hold for 10 s and then quench to RT



f) Rapidly cool to 450°C, hold for 10 s and then quench to RT



g) Rapidly cool to 625°C, hold for 1 s and then quench to RT



h) Rapidly cool to 625°C, hold at this temperature for 10 s, rapidly cool to 400°C, hold for 15 s, then quench RT



- 2. For the alloy of Fe-0.45 wt% C, sketch and label on the diagram of Fig. 1 the time-temperature paths to produce the following microstructures:
 - (a) 50% proeutectoid ferrite and 50% coarse pearlite
 - (b) 50% fine pearlite and 50% bainite
 - (c) 100% martensite

- (a) 50% proeutectoid ferrite and 50% coarse pearlite
- (b) 50% fine pearlite and 50% bainite
- (c) 100% martensite



3. Using the CCT diagram of the eutectoid steel given in Fig. 2, define and name the microstructural products of eutectoid Fe-Fe₃C alloy of 0.77 wt% C specimens that are first completely transformed to austenite, then continuously cooled to RT at the following rates: (a) 200° C/s (b) 100° C/s (c) 20° C/s

(a) 200°C/s

(b) 100°C/s

(c) 20°C/s

