

THE HEAT TREATMENT OF STEEL

Abstract

The heat treatment behavior of several plain carbon steels and low alloy steels is investigated. In this study the effect of quenching and tempering, iso-thermal annealing and continuous cooling treatments on the formation and decomposition of martensite and other phases of steel are investigated. The tempering experiments demonstrate the kinetics of the decomposition of martensite into a microstructure that yields a softer but tougher material while the iso-thermal and continuous cooling experiments demonstrate the kinetics of the decomposition of austenite. This experiment makes extensive use of the Fe-Fe₃C equilibrium phase diagram and the ITT curves for steel. The results of the heat treatments are evaluated using the Rockwell hardness tests and analysis of the microstructures of each specimen.

Introduction

Pure iron is not suitable for use as a structural material. It is weak, soft, is very ductile and does not respond to heat treatment to any appreciable degree. Steel, which is basically iron alloyed with carbon and a few percent to a few tens of percent of other alloying elements, can be heat treated to a wide range of strengths, toughnesses and ductilities. Carbon is the most important of these alloying elements in terms of the mechanical properties of steel and most heat treatments of steel are based primarily on controlling the distribution of carbon.

Alloying Elements: Alloying provides the basis for the heat treatability of steel. While iron is the chief element in steel, carbon is the most important alloying element in terms of its strengthening effect on iron. Small amounts of carbon increasing to as high as 0.80 - 0.90 w%, steadily increase its strength and hardness. Up to 4.5 w% carbon can be present in plain carbon steels, but the strength and formability deteriorate and it assumes the characteristics of cast irons. Nitrogen is often used to provide the similar strengthening, usually for case hardening (nitriding) while relatively high

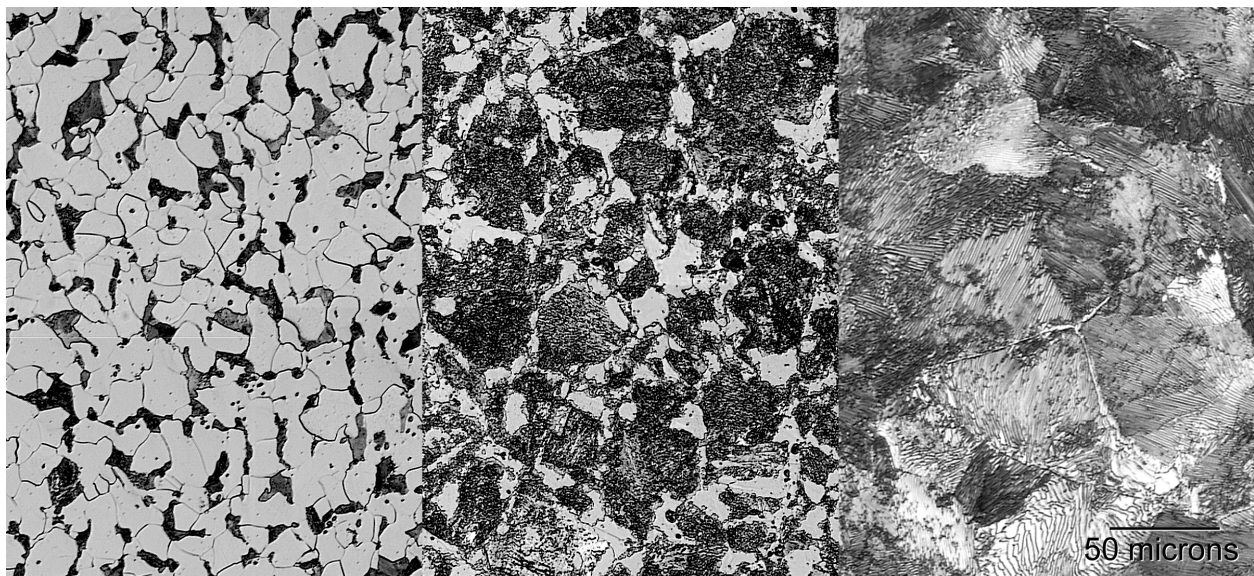


Figure 1. Three plain carbon steels, from left to right: 1018, 1045 and 1095. Increasing the carbon content (from 0.18% to 0.95%) causes the amount of ferrite (light) to decrease and the amount of pearlite (dark, lamellar) to increase.

concentrations of sulfur and phosphorus can improve the machinability of steel. However, sulfur can lead to "hot shortness" during hot forming so small amounts of manganese is often added to scavenge deleterious sulfur impurities from the grain boundaries where it can also contribute to intergranular failure. Small amounts of silicon are usually present. Silicon increases the hardness of ferrite, increases oxidation resistance and is an effective deoxidizer. It is almost universally present in magnet sheet. Aluminum is another strong deoxidizer and is also very effective at refining grain size. In steels with relatively high carbon contents, Cr, Mo, W and V all can form carbides which can both increase the creep resistance of the steel and, when the carbides are present in sufficient quantities and as large particles, the steel may be suitable for use as a cutting tool. There are many other elements that are alloyed with iron to tailor its properties and enhance heat treatability. Aside from the hardening achieved by adding carbon, the most important effect alloying elements can have is improving the hardenability of steel.

Microstructural Dynamics: The heat treating of steels results in a variety of stable and metastable phases. The important equilibrium phases are shown in the Fe-Fe₃C equilibrium phase diagram (figure 3). Most steels contain less than 1% carbon and it can be seen in figure 3 that the Fe-Fe₃C system includes a eutectoid reaction in which the maximum solubility of carbon in the γ phase is only 0.0218 w% at the eutectoid temperature and decreases to 0.008 w% at room temperature. Note that only a minor amount of precipitation hardening can occur below the eutectoid temperature. While the hardening of aluminum alloys is limited to cold working and/or precipitation hardening, the principles behind the heat treatment of steel is much more varied.

A typical heat treatment usually starts with an austenitization treatment where the ferrite (α -Fe, bcc) phase transforms to the austenite (γ -Fe, fcc) and all carbides are dissolved in the austenite. Slow cooling to below the eutectoid temperature results in the formation of ferrite and cementite (Fe₃C). This might appear as proeutectoid ferrite and a lamellar ferrite/cementite structure called pearlite, depending on the carbon content of the steel and the cooling rate. Slower cooling rates produce coarser microstructures and very slow cooling rates, or holding at a temperature just below the eutectoid temperature, can result in a microstructure consisting of spherical cementite grains in a field of ferrite. Heat treatments designed to strengthen steel, however, are based on the formation of bainite, a fine, feathery microstructure consisting of ferrite and cementite, which can be formed by rapid cooling and holding at a relatively low temperature or from precipitation from the metastable martensite phase which is formed by quenching directly from the austenite field.



Mechanisms of Phase Transformations: The grain boundaries of the austenite phase produced during the solution treatment are preferred sites for the nucleation of the new phases formed during

Figure 2. The necklace structure in 1045 steel. As this material cooled from the austenite region the ferrite (light) formed first at prior austenite grain boundaries and pearlite (lamellar) formed later in the remaining material.

subsequent steps in the heat treatment. For the slow cooling of a medium or low carbon steel from the austenite phase field a proeutectoid ferrite would nucleate and grow from the prior austenite boundaries. When the temperature reaches eutectoid temperature the remaining austenite starts to transform into the eutectoid phase. The formation of the pearlite phase is a growth dominated process and requires a combination of high temperature and long times while the formation of bainite is dominated by the nucleation process and results in a fine dispersion of ferrite and cementite.

The formation of martensite also involves nucleation and growth but the mechanism is not controlled by diffusion. Rather, it is a shear process similar to deformation twinning except that it creates a new bcc lattice. But martensite formation is not purely a shear transformation since it also produces some dilatation. And while the nucleation of martensite is thermally activated the growth process is athermal and involves the glide of transformation dislocations which shears the material, forming thin plates. These plates grow along specific crystallographic directions until either they encounter an austenite grain boundary or another martensite plate or when enough martensite has formed to have reduced the free energy sufficiently that the reaction ceases.

The martensite plates form at approximately the speed of sound, much too fast for carbon to diffuse out of the fcc lattice, and therefore the martensite has the same composition as the austenite. This means the martensite phase is supersaturated with carbon, is metastable, and will have a tendency to decompose into equilibrium ferrite and cementite. This is the basis for the tempering of steel. By raising the temperature to a few hundred degrees the martensite will decompose into a hard but much tougher, bainite. The decomposition of martensite starts by forming bainite at the boundaries of the martensite needles. If the temperature is high enough this bainite can coarsen into lower (fine) pearlite and eventually upper (coarse) pearlite. In fine pearlitic structures the carbide lamellae can be as thin as 125 angstroms while the coarsest pearlite structures can have carbide lamellae as thick as 2 microns.

Heat Treatability: Hardenability is one measure of the heat treatability of steel. The typical heat treatment begins with an austenitizing treatment followed by quenching to form martensite. The martensite is then tempered. This softens the material somewhat but increases the toughness considerably. Ideally, the quench would produce a 100% martensitic structure so that the tempering treatment will produce a part with uniform hardness. In plain carbon steels, however, only the first $\frac{1}{8}$ - $\frac{3}{8}$ inches near the surface is quenched rapidly enough to form 100% martensite. The deeper sections contain progressively softer microstructures. A more severe quench might produce 100% martensite to a greater depth but it can also lead to warping and cracking, especially in large parts or ones in which the thickness varies abruptly.

To improve the hardenability the steel must be modified so that martensite can be formed at greater depths at lower cooling rates. While carbon is required for the formation of martensite and determines the hardness of this martensite, a number of other elements are known for their ability to improve the hardenability of steel. Apparently they achieve this by retarding the nucleation rate of bainite. Molybdenum is very effective at increasing hardenability and resisting softening during tempering. Chromium is only slightly less effective at increasing hardenability and it is also very effective at improving corrosion resistance. Nickel has only a moderate effect on hardenability. However, it increases the toughness and tends to render high chromium, low carbon steels austenitic. Also, alloying with these elements tends to lead to the retention of a minor amount of austenite.

In this experiment the heat treatment behavior of several plain carbon and low alloy steels is investigated. This experiment demonstrates the interesting and important kinetics of phase transformations in steel. It illustrates how to use an equilibrium phase diagram, TTT curves, ITT curves and CCT curves to specify a heat treatment, understand the development of a microstructure and, to a lesser extent, the relationship between microstructure and properties.

Four different heat treatments are performed. The first two deal with the formation and decomposition of martensite. Specimens are austenitized and quenched to form martensite then tempered to form a mixture of ferrite and carbide. The third heat treatment involves isothermal anneals and the fourth involves the use of different cooling rates after the austenitizing anneal. The effects of these heat treatments is evaluated by measuring the hardness and examining the microstructure.

Preparation

Before attempting this experiment one should be familiar with the certain technological and theoretical aspects of the experiment. One should also start thinking about possible outcomes of the experiment. The following questions should help get you started.

1. AISI uses a 4 digit numbering system for designating the compositions of steels. The first two digits represent the major alloying elements used while the last two numbers tell how much carbon is in the steel. What are the major groups designated by the first two numbers? What does it mean when a B, E, H or L is included with the alloy designation?
2. Describe the following iron based alloys in terms of the range of compositions (particularly carbon) and typical mechanical properties.

a. Plain carbon steel	e. Gray cast iron
b. Low, medium and high carbon steel	f. Stainless steels: ferritic, austenitic, martensitic
c. Cast iron	g. Hypo-eutectoid steel
d. White cast iron	h. Hyper-eutectoid steel
3. Describe, in general terms, the composition, structure, mechanical properties and formation of the following phases found in steels:

a. Ferrite	d. Martensite
b. Pearlite	e. Austenite
c. Cementite	f. Graphite
4. Describe, in general terms, the following standard types of heat treatments in terms of the processing, microstructure produced, and mechanical properties for steel:

a. Normalize	e. Martemper
b. Temper	f. Austemper
c. Quench	g. Stress relieve
d. Austenitize	h. Anneal
5. Obtain copies of the ITT curves for each of the steels studied in procedures 2 through 4 of

this experiment. Sketch each heat treatment on the ITT curves and note the hardness and microstructure that these treatments should produce.

6. Repeat question 5 for part 5 using CCT curves.
7. Why do alloys possessing high hardenability often have poor weldability (usually due to weld cracking)?
8. What role does carbon play in the formation of martensite? Can pure iron be austenitized then quenched to form martensite?
9. How does austenite grain size effect hardenability?
10. What is the A_3 temperature and why is it different for the different alloys being investigated?

Safety Considerations

This experiment involves heat treating several steels, hardness testing them and polishing them for metallographic observation. Extreme care should be exercised during the heat treating phase of the experiment as the temperatures are quite high (900°C) and therefore poses severe burn hazards to personnel and fire hazards to the building. Slight hazards are associated with polishing and grinding the specimens. Novices invariably end up having a specimen or two snag the polishing cloth, sending their specimen flying across the room. The etchant which is used, nital, is not particularly aggressive but should be respected.

Chemical Hazards

Moderate. Etchants containing nitric acid are used. Appropriate personal protective equipment should be worn and the etchants should only be used in a safe out-of-the-way part of the laboratory which is set up for only etching the samples (preferably the fume hood). Proper technique should be used for etching the specimens, washing the specimens and disposing of the etchant. MSDS's for each of the etchants or the components of each etchant are available. MSDS's are also available for the polishing and grinding materials.

Physical Hazards

1. The potential for very serious burns exists. Temperatures approaching 900°C are used during these experiments. At these temperatures one can easily be burned while loading and unloading specimens from the furnaces, even if the hot specimens and furnace are not touched. It will be important to wear heat resistant gloves and to use long tongs. One should also take care to prepare a clear area to work, have an emergency procedure in place in case hot specimens are dropped on the floor, etc. It would be a good idea to rehearse the procedures for handling hot specimens.

2. Hardness testing poses very little hazard if proper testing procedures are followed. Using the proper anvil and indenter and a clean specimen will minimize the chance of damaging the equipment or injuring personnel.

3. The polishing wheels can spin at several hundred rpm. Take care during the grinding and polishing phases of the experiment that your fingers or the specimen do

not get trapped between the spinning wheel and the bowl. Also, take a moment to bevel both the tops and bottoms of the specimens to remove sharp edges that might snag the polishing cloth or grinding paper, get ripped out of your hand, cutting your fingers and sending the specimen flying across the room.

Biohazards

None.

Radiation Hazards

None.

Protective Equipment

Recommended: safety glasses are recommended during the polishing/grinding and hardness testing phases of the experiment. The use of protective coverings for the floor and counter tops is also recommended.

Required: safety glasses, heat resistant gloves and long tongs for the heat treatment phases of the experiment. Safety glasses and chemical resistant gloves must be used when etching the specimens.

Waste

1. Pour used etchant back into the original bottle. Dilute nital etchant which was washed from the specimen can be poured down the drain. Other disposal procedures may be required if etchants other than nital are used.

2. Used specimens can be recycled as scrap steel.

Materials

The alloys studied in this experiment are 1018, 1045, and 1095 plain carbon steels and 4140, 4340, and 8620 low alloy steels. All alloys were received as hot rolled rod stock. These materials were cut from this into $\frac{5}{8}$ to $\frac{3}{4}$ inch thick specimens and then painted to color code them for alloy composition.

The following are a few comments on the alloys being studied. In them, the following abbreviations are used: HR=hot rolled, OQ=oil quenched, WQ=water quenched, HRB and HRC are Rockwell hardness numbers on the "B" and "C" scales respectively. For more information refer to references [1] and [2].

1018 Excellent forgeability, reasonably good cold formability, excellent weldability, relatively poor machinability. Tensile strength from 69 (HR) to 92 (WQ) ksi., elongation from 38% to 20%, hardness from 45 to 64 HRB.

1045 Popular medium carbon steel, excellent forgeability, fair machinability, responds well to heat treatment. As-quenched hardness greater than 55 HRC. Tensile strength from 98 (HR) to 120 (WQ) ksi., elongation from 24% to 18%.

1095 Easily forged, hardness up to 66 HRC in full quenched condition. Contains free carbide. Slightly lower hardenability than the 1090 alloy. Tensile strength from 142 (HR) to 200 (WQ) ksi.,

elongation from 18% to 12%.

4140 Most widely used alloy steel. Inexpensive considering its high hardenability. Very good forgeability and fair machinability but poor weldability due to susceptibility to weld cracking. Tensile strength from 103 (HR) to 156 (OQ) ksi., elongation from 27% to 19%.

4340 A high-hardenability steel, higher than any standard AISI grade. Because of its high hardenability it has poor weldability except when using sophisticated processes, ie. electron beam welding. Tensile strengths from 101 (HR) to 182 (OQ) ksi., elongation from 21% to 15%.

8620 Extensive use as a case hardening steel, excellent forgeability and weldability, reasonable hardenability, fair machinability. Tensile strength from 89 (HR) to 127 (WQ) ksi and elongations from 25% to 21%.

Procedure

1. Preliminary

Measure hardness of the original specimens, before any heat treatments are performed. Clean and deburr if necessary. Next, devise a numbering system to identify each specimen by composition and heat treatment number. Finally, prepare a specimen of each as received alloy for metallographic examination.

2. Quenching

Austenitize all specimens at 50°C above the A_3 temperature ($\pm 10^\circ\text{C}$) for thirty minutes. Upon removal from the furnace quench one of each alloy in oil, and the others in water. Measure the hardness of each specimen.

Note: proper austenitizing and quenching are very important in all parts of this experiment. It is one of the procedures that we seem to have the most difficulty with. Don't skimp on the austenitizing treatments and make sure the quenches are quick and as effective as possible.

Table 1. Hardness before and after austenitizing anneals and quenching in oil and water.

Alloy	Austenitizing Temperature	Rockwell Hardness, HRC		
		As Received	Oil Quenched	Water Quenched
1018	880°C			
1045	840°C			
1095	750°C			
4140	845°C			
4340	845°C			
8620	885°C			

3. Tempering

Austenitize specimens as in part 2, water quench, then temper for ½ hour at specified temperatures ($\pm 10^\circ\text{C}$) listed in table 2. Water quench to conclude the tempering process. Measure the hardness of each specimen.

Table 2. Hardness after austenitizing, water quenching, and tempering.

Alloy	Rockwell Hardness, HRC				
	As Quenched	200°C	400°C	600°C	700°C
1018					
1045					
4140					
4340					
8620					

4. Continuous Cooling Transformations

Perform this experiment using only the 1045 and 4140 steels. Austenitize as in part 2. When removing the specimens from the furnace, cool specimens at different rates as indicated in table 4. To furnace cool, simply turn off the furnace and leave the door closed. It might take 6 to 12 hours for it to cool to a temperature that you could handle the specimen.

Table 3. Hardness following austenitizing anneals and cooling at different rates.

Treatment	Rockwell Hardness, HRC	
	1045 Alloy	4140 Alloy
Furnace Cooled		
Air Cooled		
Blowing Air Cooled		
Oil Quenched		
Water Quenched		

5. Metallography

Prepare all 1045 alloy specimens and a selection of the other specimens for metallographic evaluation. The basic procedure is:

- C Wet grind using 240, 320, 400 and finally 600 grit SiC paper.
- C Rough polish using 6 micron diamond on a nylon cloth.
- C Fine polish using 0.05 micron alumina on a flocked cloth.

C Etch in a Nital solution.

Refer to experiment 3 and the appendix for more detailed instructions on preparing specimens for metallography.

Sketch or take photomicrographs of each specimen. Note the main features of each microstructure, trends in microstructure with heat treatment, etc. As there are four different parts to this experiment, metallography will produce four sets of micrographs. Each set should clearly show how the different heat treatments influence the microstructure.

Analysis

The following questions should help you analyze the results and to begin to develop the ideas you will put into your report.

1. Organize the data into tables and prepare graphs as you feel are appropriate.
2. In general, how does the hardness data compare to that which is typical for these steels and heat treatments? Do the trends follow what you expect from the carbon content and hardenability of these steels?
3. Examine the microstructure. Are they similar to those predicted by the ITT diagrams?
4. Correlate properties and microstructure. Briefly, describe the types and approximate amounts of phases present, the hardness of each phase and the overall hardness of the specimen.
5. Correlate microstructure and heat treatments. Describe the kinetics of the phase transformations as shown by your data.

References and Further Reading

1. Heat Treating Guide: Standard Practice and Procedure for Steel, eds. P.M.Unterweiser, H.E.Boyer and J.J.Kubbs, ASM, Metals Park, Ohio, (1982). (UCD PSL: TN751.H4 1982 (REF))
2. Atlas of Isothermal Transformation and Cooling Transformation Diagrams, ed. H.E.Boyer, ASM, Metals Park, Ohio, (1977). (UCD PSL: TN693.I7A871)
3. Smithells Metals Reference Book, 6th edition, ed. E.A.Brandes, Butterworths & Co., Ltd., London, 1983. (UCD PSL: TN671.S5 1983 (REF))
4. Samuels, L. E., Optical Microscopy of Carbon Steels, ASM, Metals Park, Ohio, 1980. (UCD PSL: TN693.I7S26)

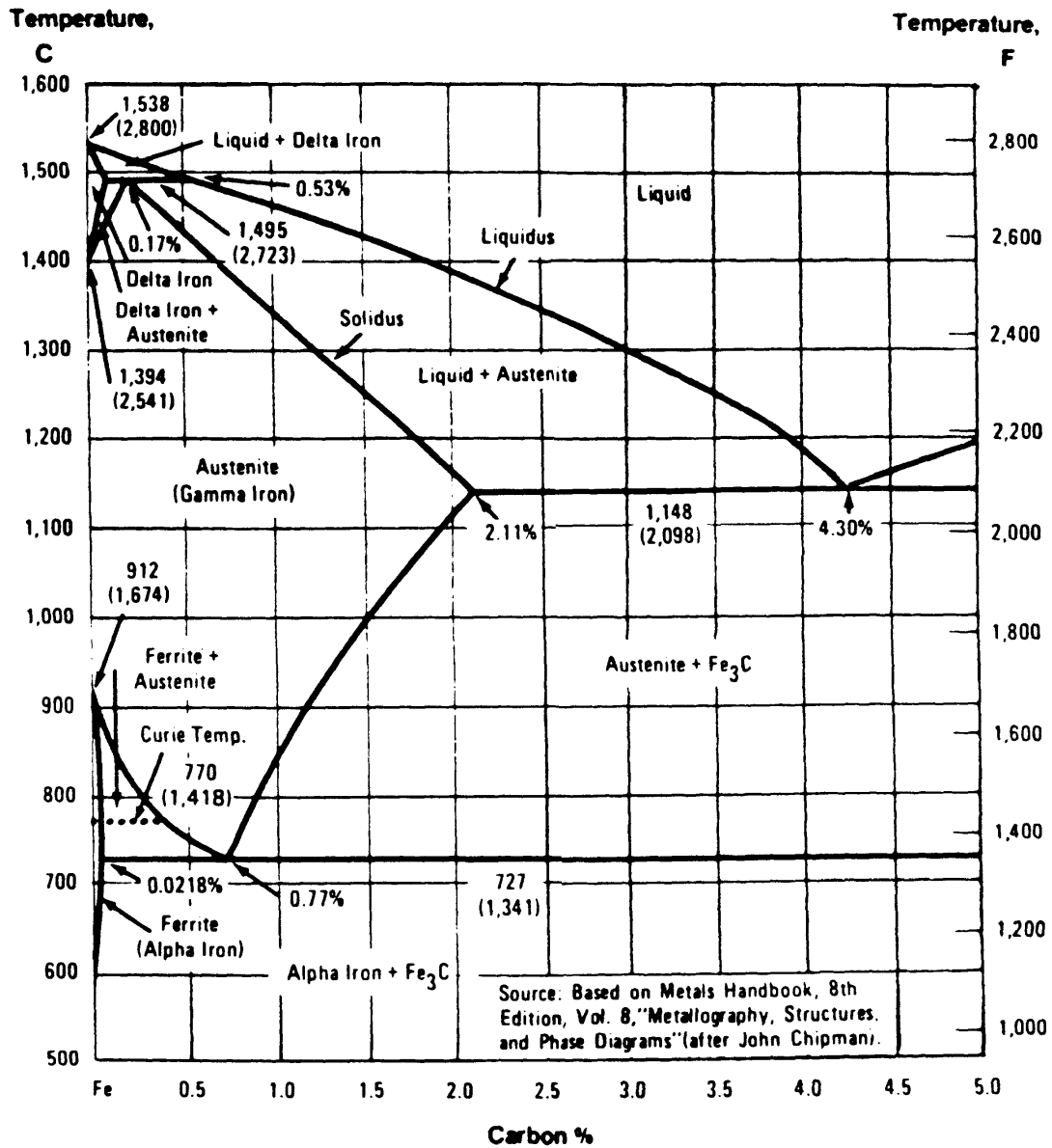


Figure 3. Fe-Fe₃C equilibrium phase diagram, from Alloy Steel Data Book, Copperweld Steel, Warren, OH.

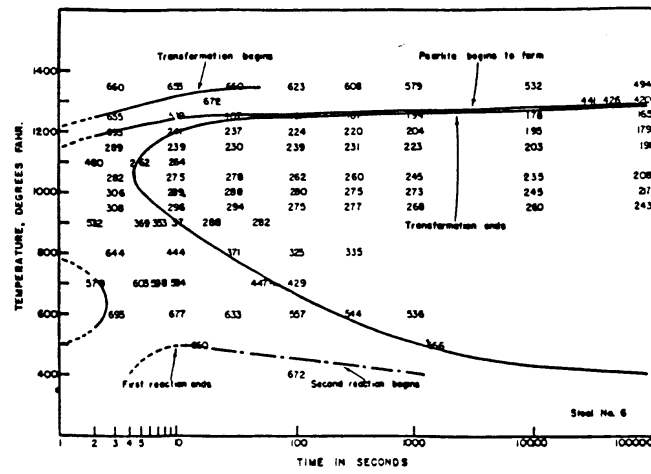


Figure 4. ITT Curve for 1045 Steel [2].

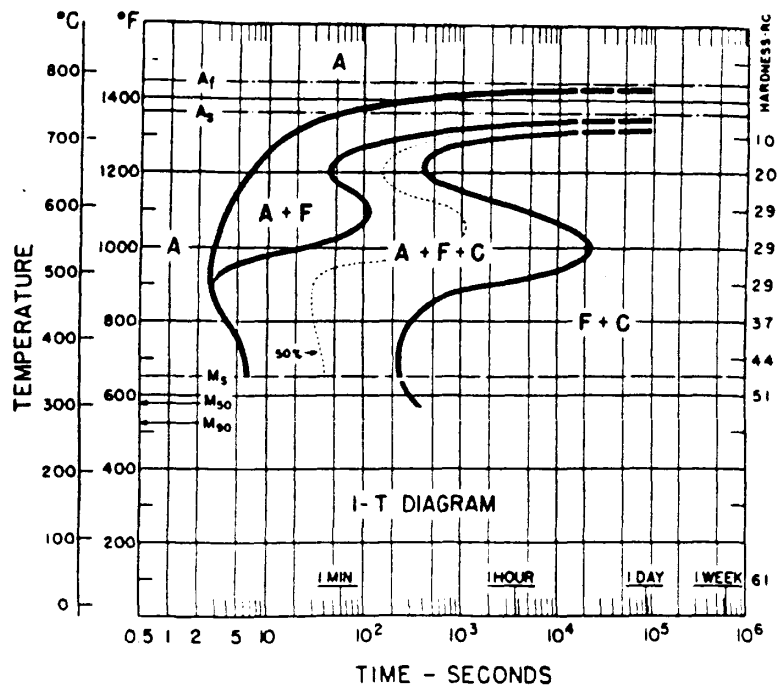


Figure 5. ITT Curve for 4140 Steel [2].