Experiment: Heat Treatment - Quenching & Tempering

Objectives

- 1) To investigate the conventional heat treatment procedures, such as quenching and annealing, used to alter the properties of steels. SAE 1040 steel standard Charpy type impact specimens will be used.
- 2) To study the effects of heat treatment on the microstructure and mechanical properties of steels; impact strength and hardness will be measured for heat treated specimens.
- 3) To quantitatively evaluate the hardenability, or depth of hardness, of a steel rod through application of the standard (ASTM A255) Jominy End-Quench Test.

Abstract

Conventional heat treatment procedures for producing martensitic steels generally involve continuous and rapid cooling of an austenitized specimen in some type of quenching medium, such as water, oil, or air. The properties of a steel that has been quenched and then tempered depends largely on the rate of cooling and tempering times and temperatures. During the quenching heat treatment, the specimen can be converted to a variety of microstructures including soft and ductile spheroidite to hard and brittle martensite. The production of pearlitic and bainitic steels is lower in cost and suffices for most applications. Martensitic steels must be tempered prior to use due to their extreme brittleness. A range of heat treatments producing a variety of microstructures and mechanical properties will be investigated in this experiment beginning with a set of initially equivalent samples of SAE 1040 steel. Pearlite, Bainite and Martensite will all be produced through variations in the cooling rates of initially austenized samples.

The second experiment involved in the study of the heat treatment examines "Hardenability". The Jominy End-Quench Test is a widely utilized standard test procedure for determining the hardenability of ferrous alloys. The term "Hardenability" is not the same as hardness, "Hardenability" refers to an alloy's ability to be hardened by the formation of Martensite as a result of a given heat treatment. High "Hardenability" alloys are those that not only harden on the surface, but to a great extent harden throughout the part's interior. Hardenability may also be thought of as a measure of the depth to which a specific alloy may be hardened.

Background

Heat treatment is a combination of timed heating and cooling operations applied to a metal or alloy in the solid state in such ways as to produce certain microstructures and desired properties. Annealing, Normalizing, Quench Hardening, Tempering, and Austempering are five of the important heat treatments often used to modify the microstructure and properties of steels. The microstructure produced by any of the above heat treatments can be deduced using Continuous Cooling Transformation (CCT) diagrams, which are directly related to the Time Temperature Transformation (TTT) diagrams for the specific steel being treated. Such a TTT diagram for eutectoid steel is shown in Figure 1. The microstructures that result from various heat treatments are dependent on the cooling rate from the austenite range; they are predicted using the TTT diagram with superimposed cooling curves for the selected material.

Time Temperature Transformation (TTT) and Continuous Cooling Transformation (CCT)

Recall that phase diagrams indicate the microstructures that should develop if equilibrium conditions are attained. Engineering materials often involve phase transformations that result in non-equilibrium structures. These non-equilibrium structures are Metastable – that is they're stable in time but will transform into the equilibrium structure over time. Phase transformations, especially solid-solid transformations as in an eutectoid reaction, do not occur instantaneously; phase transformations require time! TTT and CCT diagrams summarize the percent completion of a given phase transformation and are similar to phase diagrams except that time is included.

TTT and CCT diagrams generally contain 3 distinct lines, which represent:

Nucleation Line - Beginning of the Phase Transformation Process.

50% Completion of the Phase Transformation.

~100% Completion of the Phase Transformation.

Iron – Iron Carbide (steel) TTT and CCT diagrams involve the transformation of Austenite. Looking specifically at eutectoid composition steel (0.77wt% C)

Pearlite is composed of alternating layers of Ferrite and Cementite. Pearlite layer thickness is a function of quenching temperature (and Rate):

- 1. Thick layers (Coarse Pearlite) are produced at a quench temperature of ~650°C.
- 2. Thin layers (Fine Pearlite) are produced at a quench temperature of ~500°C.
- 3. A particulate structure (Bainite) is produced at a quench temperature of ~350°C.

Coarse Pearlite is relatively soft and ductile but is often utilized as a low-cost structural material. Its' properties depend largely on carbon content. Fine Pearlite is generally a mid-range steel with good strength and ductility characteristics; actual properties are a function of carbon content. Bainite is stronger and slightly less ductile than Fine Pearlite. Overall Bainite is a relatively cheap steel which combines good strength with moderate ductility.

If Austenite is quenched (cooled rapidly) at a rate such that the knee of the TTT/CCT diagram is avoided (rapid quench), a diffusionless transformation into Martensite occurs where:

MS -> Start of the Transformation.

M₅₀ -> 50% Transformed into Martinsite.

M₉₀ -> 90% Transformed into Martinsite.

The Martensite transformation involves sudden reorientation of the carbon atoms in the FCC Austenite structure to a Body Centered Tetragonal (BCT) structure without allowing time for the diffusion of atoms to their lowest-energy equilibrium positions. The result is a high internal energy material that is very strong, hard, brittle and susceptible to rapid crack growth.

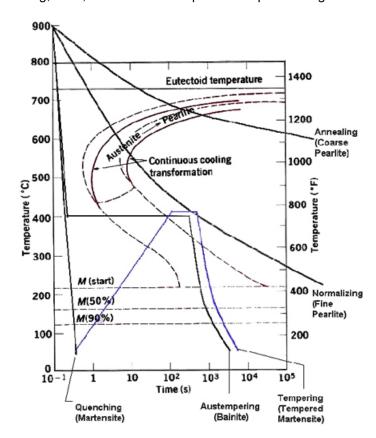


Figure 1 - TTT Diagram for Eutectoid Steel. [Shackelford, 1996]

Continuous Cooling Transformation (CCT) Diagrams

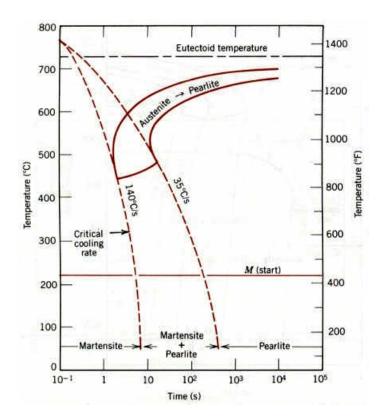


Figure 2 - CCT Diagram for a Eutectoid Steel. [Callister, 1991]

In practice, most heat treatments for steels involve the continuous cooling of a specimen to room temperature. TTT diagrams are valid only for constant quenching temperatures. For Continuous Cooling, the required time for a reaction to begin and end is slightly delayed and cooling does not occur at a constant temperature.

Hardness Testing

Hardness tests measure the resistance to penetration of the surface of a material by a hard object. The depth of penetration is measured by the testing machine and converted to a hardness number. C-Scale Rockwell Hardness (HRC) numbers can be converted to Brinell Hardness (HB) numbers and then used to approximate the tensile strength in steels.

Impact Testing

When a material is subjected to a sudden intense blow in which the strain rate is extremely rapid, it may behave in a much more brittle manner than is observed in the tensile test. The Charpy Impact test, also known as the notch toughness test, is often used to evaluate the brittleness of a material under these conditions. The test specimen should be notched, because V-notched specimens better measure the resistance of the material to crack propagation. The ability of a material to withstand an impact blow is often referred to as the toughness.

Hardenability: Jominy End-Quench Test

Hardenability refers to the relative ability of a steel to be hardened by the formation of martensite. The curves shown in Figure 3 are Hardenability Curves produced from Jominy End Quench Tests where water is sprayed on the end of a heated steel bar. Note the following:

- the 5140 steel is only hard near the quenched end (low hardenability)
- the 4340 steel is relatively hard all along the bar (high hardenability)

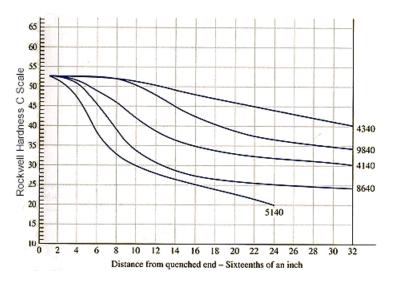


Figure 3 - Hardenability Curves for some Common Alloy Steels. [Shackelford, 1996]

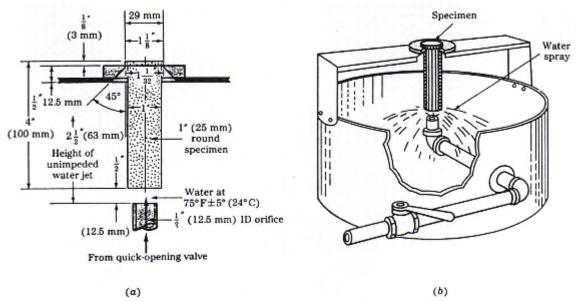


Figure 4 - Specimen and Fixture for a Jominy End-Quench Hardenability Test. [Shackelford, 1986]

The Jominy End-Quench test consists of selectively cooling a standard size bar of steel, as is illustrated in Figure 4. The specimen consists of a cylindrical bar with a 1-in. diameter and 4-in. length. After the sample has been austenized, it is placed in a fixture, as shown in Figure 4, and a jet of water is quickly splashed onto one end of the specimen. Note that only the end of the specimen is actually quenched, and as a result, the transformation of Austenite begins at the water-cooled end and progresses "up" the bar. The cooling rate decreases with increasing distance from the quenched end and thus microstructure and material properties are functions of the distance from the quenched end. After cooling, a flat surface is ground on the test bar and Rockwell C hardness measurements are made along the surface up to 3 inches from the quenched end. The results are plotted in the form illustrated in Figure 3 that indicates the hardenability of a given steel.

Note the Following:

- There is a rapid decrease in the hardness of steels with relatively low hardenability as a function of the distance from the water quenched end.
- There is a gradual decrease in the hardness of steels with relatively high hardenability as a function of the distance from the water quenched end.

For steel, ultimate strength is related to Rockwell C hardness and thus the strength of a heat-treated steel can be approximated from the Rockwell C hardness values. Figure 5 presents a conversion for Brinell, Vickers and Rockwell tests.

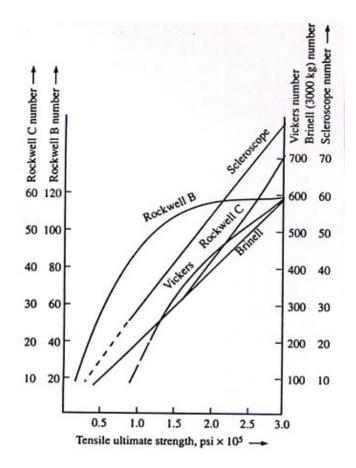


Figure and Ultimate Tensile Strength for Steel Parts. [Metals Handbook - Desk Edition, 1992]

5 - Relationships Between Hardness

Experimental Procedure

- 1. Immerse five SAE 1040 specimens (and the Jominy Bar) in charcoal powder contained in a ceramic crucible. Austenitize these at 900°C for 1 hour. Allow adequate time for the crucible to heat to the temperature of the furnace (about 10 minutes). A total of 1 hour heating time should be adequate allowing roughly 45-50 minutes of soaking.
- 2. **Normalizing** Rapidly remove one specimen and allow it to cool in air on a ceramic base.
- 3. **Quench Hardening** Rapidly remove two specimens and quench them in water at room temperature.
- 4. **Tempering** Reheat one of the water quenched specimens to 400°C in another furnace for 30 minutes, then remove it and allow it to cool in air to room temperature.
- 5. **Austempering** Remove one specimen and quench it in a fused salt bath (mixture of Na-nitrate and Na-Nitrite in equal proportion) that is maintained at 400°C. Hold the quenched specimen in the salt bath inside the furnace for 30 minutes then remove it and quench it in water or allow it to air cool to room temperature.
- 6. **Annealing** Allow the fifth specimen to cool in the furnace itself after switching the power to the furnace off. When the temperature of the furnace drops to between 650 600°C, remove the specimen and quench it in water at room temperature or allow the furnace to cool down slowly to room temperature and remove the specimen thereafter.

- 7. Develop a Data Sheet that includes the following:
 - a. Determine the **Rockwell** hardness values for each of the heat treated specimens after cleaning one of the surfaces with sand paper to remove any hard carbon deposits that may have formed on the surface. Take 5 readings on each sample.
 - b. Determine the **Charpy** impact strengths of all the heat treated specimens (make sure the Impact Testing Machine is rezeroed after each test!).
 - c. Examine the fracture surfaces of each specimen and note the salient features.

Hardenabilty: Jominy End-Quench

Apparatus

- 1. High Temperature Oven (Capable of up to 1000°C)
- 2. Controllable Flow Water Source of Room Temperature (24°C) Water
- 3. Standard 4-Inch Jominy Test Bar with Appropriate Mounting Fixture
- 4. Heat Resistant Gloves
- 5. Heat Resistant Face-Mask
- 6. Oven Tongs for Removal of the Jominy Test Bar from the Oven
- 7. Metallographic Grinding Equipment
- 8. Hardness Tester (Rockwell Type)

Procedure

- 1. Preheat the oven to 1500°F (816°C).
- 2. Check that the Jominy Test Set-Up tank is full of room-temperature water.
- 3. Adjust the water spout height on the Jominy Test Set-Up to 2 and 1/2 inches above the final resting position of the Test-Bar bottom when placed in the specimen fixture.
- 4. Properly place the specimen fixture directly over the water spout of the Jominy Test Set-Up with the fixture center placed directly above the water spout center. Practice rapidly placing a cool sample into the fixture before proceeding.
- 5. Remove the Jominy Sample from the oven with the oven tongs and place it in the Jominy Fixture <u>as</u> <u>quickly as possible.</u>
- 6. Let the Test Bar cool to room temperature (approximately 20 to 30 minutes) then turn off the water.
- 7. Each group of 2-3 students must check out the lab session's Jominy Test Bar from the lab instructor and grind a flat onto the curved surface of the lab session's Jominy Test Bar. All Scale (oxide) on the flat should be removed such that a shinny, smooth flat results. Try to remove the minimal amount of material to obtain a shinny flat!
- 8. Using a pencil, clearly mark the positions along the bar on the ground flat at the distances from the Test Bar End as indicated on the blank Jominy End-Quench Data Sheet.
- 9. Take a single set of Rockwell C hardness measurements along the bar on the ground flat at the distances marked in Step 8; record the values on the data sheet.
- 10. Return the Jominy Test Bar to the lab instructor.

Jominy End-Quench Data Sheet

Specimen Description:			
D . 0 T			
Date & Time:			
Daalassall Oaala asad	La da atam		
Rockwell Scale and	indentor:		
Test Number	Spacing from Preceding Measurement (in.)	Distance from Quenched End (in.)	(RC)
	T		
1	1/16	1/16	
2	1/16	2/16	
3	1/16	3/16	
4	1/16	4/16	
5	1/16	5/16	
6	1/16	6/16	
7	1/16	7/16	
8	1/16	8/16	
9	1/8	5/8	
10	1/8	6/8	
11	1/8	7/8	
12	1/8	1	
13	1/8	1 & 1/8	
14	1/8	1& 2/8	
15	1/8	1& 3/8	
16	1/8	1& 4/8	
17	1/4	1& 6/8	
18	1/4	2	
19	1/4	2 & 1/4	
20	1/4	2 &2/4	
21	1/4	2 & 3/4	
22	1/4	3	
Data Collected	Ву:		

Lab Requirements- Quenching

Follow the steps outlined in the Procedure section for this experiment!

- 1. Discuss the expected microstructures and properties for each of the specimens based on the appropriate TTT and/or CCT diagrams for the cooling sequences applied.
- 2. Develop and complete a data sheet showing the results of the hardness and impact tests for each sample. Include brief comments on the fracture surfaces observed.
- 3. Discuss the relationship between heat treatment and the resulting hardness and impact energy values obtained in this experiment.
- 4. Compare and comment on the experimental results obtained with respect to the expected impact energy and hardness results.
- 5. When Austempering steel, after the 30 minutes in the 400°C bath the specimen is removed from the bath and can be either quenched in water or allowed to air cool at room temperature. Will these two procedures produce different results? Explain.

Lab Requirements- Hardenability: Jominy End Quench

- 1. Based on the data collected in the laboratory, plot Rockwell C Hardness verses the Distance from the Quenched End; the result is a "Hardenability Curve".
- 2. Discuss the Hardenability of the steel tested. Does the material appear to have relatively high or low hardenability? Compare Hardenability Curve to published results for various steels.
- 3. Although a CCT diagram is actually necessary to explain the measured hardness values, discuss the measured hardness numbers in terms of a TTT diagram.
- 4. Speculate on what you would expect in terms of the variation in hardness through-the-thickness of the Jominy Test Bar one inch from the quenched end. Would it be hardest in the center or near the surface? Explain.

Homework

- 1. Using the TTT diagram for Eutectoid Steel, specify the constituents and their approximate percentages of a small specimen subjected to the following time-temperature treatments. All begin with a completely Austenized specimen:
 - Rapidly cool to 650°C, hold for 100 seconds, then quench to room temperature.
 - Rapidly cool to 500°C, hold for 100 seconds, then quench to room temperature.
 - Rapidly cool to 350°C, hold for 100 seconds, then quench to room temperature.
 - Rapidly cool to 100°C, hold for 100 seconds, then quench to room temperature.
 - Rapidly cool to 500°C, hold for 4 seconds, quench to 300°C, hold for 200 seconds, then quench to room temperature.
- 2. Briefly explain the steps involved in the following heat treatments of low alloy steels. Discuss the expected microstructure for each treatment and how if affects the mechanical properties.
 - a.) Normalizing
 - b.) Quenching
 - c.) Tempering
 - d.) Austempering
 - e.) Annealing
- 3. What is decarburization? What causes it and how is it prevented? How does it affect the properties of heat treated steels? (Check sources outside the modules)

Samples



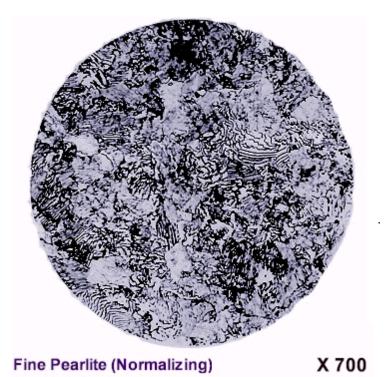
Process Annealing

Austenitized at 900°C, a previously cold-worked SAE 1040 specimen, held for 1 hour, then allowed to cool in the furnace itself by turning the oven off thus facilitating a very slow cooling of the sample.

Process Annealing is used to relieve stresses, increase ductility and modify the microstructure.

Process annealing involves recovery, recrystallization and grain growth.

Quenching is often conducted following equiaxed grain formation to facilitate the production of a fine-grained microstructure.



Normalizing

SAE 1040 specimen, Austenitized at 900°C for 1 hour then allowed to cool in air.

Similar to a Process Anneal, Normalizing is applied to reverse the embritling effects of cold work. By heating the sample into the austenite range and allowing recrytallization, the grain structure is refined and relatively small grains are formed by allowing the sample to slow-cool in air.



Quench Hardening

SAE 1040 specimen, Austenitized at 900°C for 1 hour, then rapidly quenched in cold water.

Of the various microstructures that may be produced for a given steel alloy, martensite is the hardest and strongest and, in addition, the most brittle.



Tempering

SAE 1040 specimen, Austenitized at 900°C for 1 hour then water quenched. The specimens are reheated to 400°C in another furnace for 30 minutes and are then removed and allowed to cool to room termperature in air.

Martensite in the as-quenched state, in addition to being very hard, is so brittle that it cannot be used for most applications. The ductility and toughness of martensite are enhanced, and the internal stresses relieved, through the Tempering process.



Austempering

SAE 1040 specimen, Austenitized at 900°C for 1 hour, then the specimen is quenched in a fused salt bath (mixture of Na-nitrate and Nitrite in equal proportion) that is maintained at 400°C. After 30 minutes, the specimen is removed from the bath and quenched in water or allowed to air cool at room temperature.

Austempering facilitates the formation of a Bainitic structure that exhibits an excellent balance between high strength and ductility.