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Microconstituent Formation Analysis

Abstract

This experiment heat treated eleven samples of W1 Tool Steel in four different methods to analyze the physical properties and microconstituents inside each resulting from cooling. Out of the eleven, two were quenched in water, two rested in sand, two reposed in open air, three cooled within the furnace as it cooled, and two were inspected as received without heating. The furnace heated these nine samples to 870^{o}C, then removing material, polishing and etching revealed the microconstituents within each sample. Isothermal transformation diagrams for steel concurred with the microconstituents found in each sample due to the different cooling methods used. A Rockwell C hardness test identified each material based on relative hardness, and this matched the isothermal transformation diagram prediction as well. The water quenched samples had a Rockwell C hardness of up to 66, agreeing with transformation diagrams and images to indicate Martensite. Air cooled, sand cooled, and furnace cooled samples contain both fine pearlite and bainite, hardness measurements close to 30. A cooling curve created by temperature taken from the cooling furnace agrees that in the 2.2 hours, pearlite and bainite formed. The lowest Rockwell C hardness belonged to the as received samples, indicating spheroidite.

Introduction

The design of this experiment provided exploration into micro-constituent formation within eleven samples of W1 tool steel. Each sample underwent a different method of cooling from an austenitic composition. Analysis of each sample provided insight into the use of heat treating techniques and how phase structures alter material properties. Temperature and amount of time cooling determines the micro-constituents present in each sample. Each cooling technique's effect on a sample can be guessed using figure one, a continuous cooling transformation diagram (CCD). Figure one provides a guide for material microconstituents that form as the material cools within different time intervals if cooling from austenite.

Although the as received samples were untreated, meaning that a microstructure due to heat treating is unknown, the CCD predicts the nine treated samples' compositions. Based off the CCD, specimens cooled in the furnace required the most time to do so, denoting a likelihood of fine or course pearlite within those samples. Within the two sand cooled trials, the sand would insulate the heated material to some extent, raising the cooling time. This establishes fine pearlite or bainite's presence within sand cooled steel samples. The next fastest cooling technique was cooling in air, meaning that the samples develop a greater amount of bainite. Water quenching is definitely the fastest cooling

method, so martensite formation was predicted. The hardest structure, martensite, will have the highest Rockwell C hardness value while spheroidite will have the lowest.

The different arrangement of phases in the samples are used for their material characteristics. Table one describes characteristics of each microconstituent. Due to its corrosion resistance and durability, martensitic steel is used for blades, machine screws, springs, surgical equipment, and many other applications that involve high stress or corrosive conditions [1]. Martensite is the hardest and most brittle microconstituent of steel, with Bainite being the next hardest, known to be slightly more resistant to creep. Bainite is easier to make than martensite, so it is often used in the automotive industry for structural supports [2]. Pearlite has similar characteristics with more ductility, and thermal resistance. Its uses lie in creating nails, chisels, refrigeration equipment, or wires [3]. Spheroidite is the softest microconstituent of steel, useful to soften higher carbon steels and while cold working metal [4].

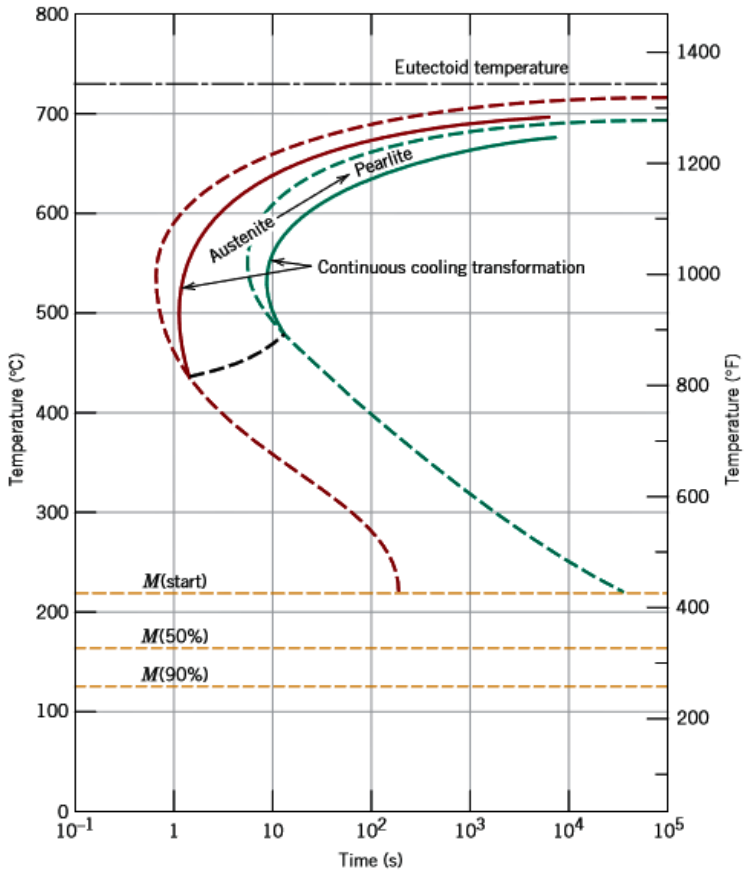

Figure 1: Continuous Cooling Transformation Diagram [5]

Microconstituent	*Phases Present*	*Arrangement of Phases*	*Mechanical Properties (Relative)*
Spheroidite	α-Ferrite + Fe_3C	Relatively small Fe_3C spherelike particles in an α-ferrite matrix	Soft and ductile
Coarse pearlite	α-Ferrite + Fe_3C	Alternating layers of α-ferrite and Fe_3C that are relatively thick	Harder and stronger than spheroidite, but not as ductile as spheroidite
Fine pearlite	α-Ferrite + Fe_3C	Alternating layers of α-ferrite and Fe_3C that are relatively thin	Harder and stronger than coarse pearlite, but not as ductile as coarse pearlite
Bainite	α-Ferrite + Fe_3C	Very fine and elongated particles of Fe_3C in an α-ferrite matrix	Harder and stronger than fine pearlite; less hard than martensite; more ductile than martensite
Tempered martensite	α-Ferrite + Fe_3C	Very small Fe_3C spherelike particles in an α-ferrite matrix	Strong; not as hard as martensite, but much more ductile than martensite
Martensite	Body-centered, tetragonal, single phase	Needle-shaped grains	Very hard and very brittle

Table 1: Describes common features of each microconstituent [5]

Materials and Methods

The experiment started with eleven cylindrical samples dimensioned as portrayed in figure two. Of the eleven samples of W1 Tool Steel, nine of them were initially placed in a Lindberg Boxfurnace. The other two were left unaltered, to be inspected as received from the manufacturer. The nine samples entered the boxfurnace set at 870^0C and remained within it for about one hour each. Then, each was subsequently removed (with exact exit time noted) to be cooled in different methods. Of the nine furnace heated steel samples, three remained in the furnace as it cooled, two were buried in sand, two were left in the room temperature air (22.1^0C), and two were quenched in water for 30 seconds.

Two days later after each sample reached room temperature, seven mils of material was evenly removed from opposite faces of the cylindrical samples. A Wilson Instruments Hardness Tester indented one face of all eleven sample to collect six measurements of Rockwell C hardness, and the opposite face provided evidence of microconstituent formation via polishing. This polishing consisted of smoothing each sample with silicon carbide sanding paper, thoroughly utilizing grits 240, 320, 400, and 600. In that order the increasing grits denoted a finer and finer sanding gradient. Each sample's sanded face then contacted two Ecommet III polishing wheels, employing alumina particles of $10\mu m$ and $3\mu m$. When the majority of the scratches dissipated, the eleven samples were then etched using the 2% Nytal solution containing methanol and nitric acid. Etching lasted from five to twelve seconds on each surface, depending on Rockwell C Hardness values. The harder the sample, the more time the etching solution remained on the surface. An Olympus BX51 optical microscope photographed each sample at 1000 times magnification, revealing microconstituents based on patterns of phases seen in the samples.

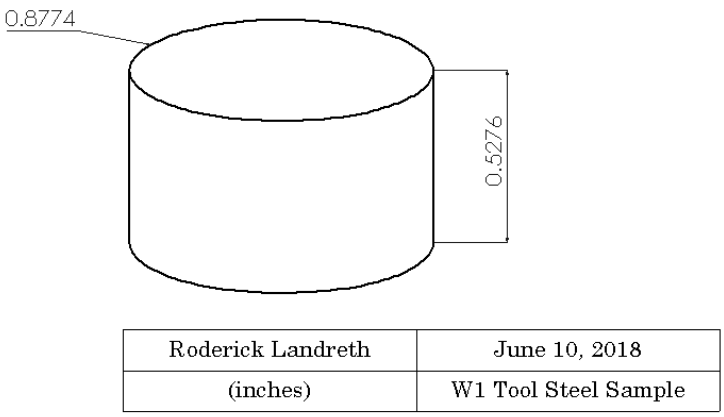

Figure 2: Dimensions of each Steel Sample

Results

Several samples placed in the Boxfurnace became attached because of the heating. Affixed samples comprised the three furnace cooled samples and the two sand cooled samples, each of which were detached after cooled to room temperature in their respected methods. Time spent in the furnace varied between each sample, because removing samples could only happen one by one (unless stuck together). Exact removal time is presented in Table two.

Although the furnace was set to 870^oC, samples were observed entering at 792^oC, and exiting at 901.5^oC. Each sample glowed red as removed, darkening as they cooled. The black oxidation cracked and shelled off the samples during quenching, and the two quenched samples developed cracks. The Rockwell C Hardness test produced six values for each sample, the average of which is presented in table two. In addition, the rate of cooling of the furnace and the samples within gave a time estimate for when the furnace samples reached room temperature. The cooling curve generated from the log plot in figure three compared with the CCD accurately predicted the phase structure of both furnace samples.

From the images collected from the optical microscope, the microconstituents were strikingly visible even though the etched surface looks dark. Figure four displays a non-etched, unpolished sample, demonstrating the necessity to process each sample. Figure five depicts the as received sample's microstructure, the small circles denoting a clear example of spheroidite, as table one describes. The next image, figure six, shows intermingled sections of bainite and pearlite, recognizable because of pearlite's layered pattern and bainite's darker, denser patterning. While this image portrays the furnace samples, supposedly the samples that cooled the slowest, both figures seven and eight show a similar combination of microconstituents. The water quenched samples produced clear examples of martensitic structure in figure nine's needle-like phase patterning.

Sample	Time in Furnace	Rockwell C Hardness
As Received 1	N/A	12.3
As Received 2	N/A	7.0
Water Quenched 1	1:02:24	58.4
Water Quenched 2	1:06:13	66.1
Air Cooled 1	1:08:08	34.7
Air Cooled 2	1:10:42	29.5
(Affixed) Sand Cooled Sample 1	1:09:31	30.9
Sand 2	1:09:31	31.8
(Affixed) Furnace Cooled Sample 1	1:11:28	27.0
Furnace 2	1:11:28	29.4
Furnace 3	1:11:28	30.0

Table 2: Furnace heating times (in removal order)

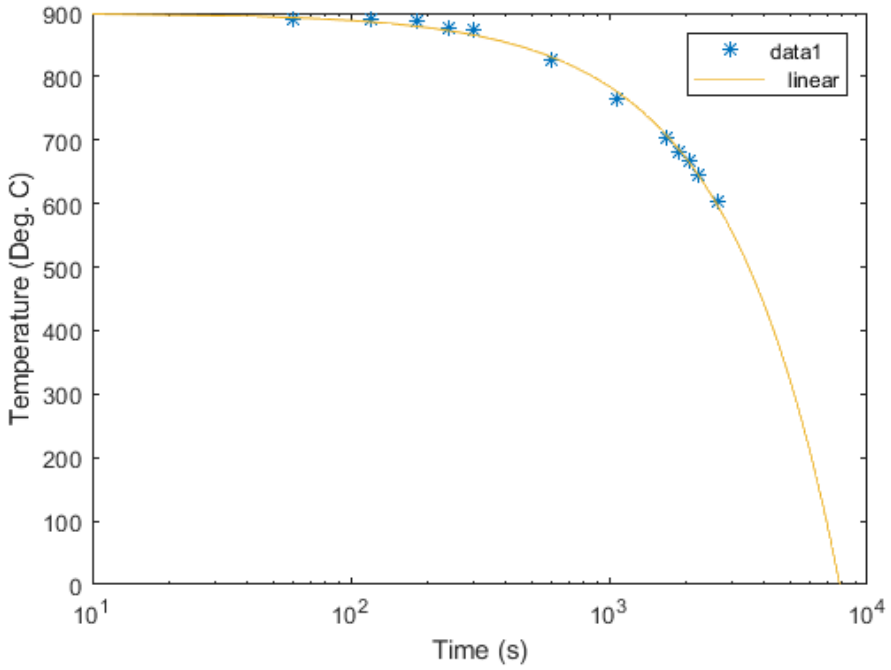

Figure 3: Log Plot of Furnace cooling time with linear fit for CCD analysis

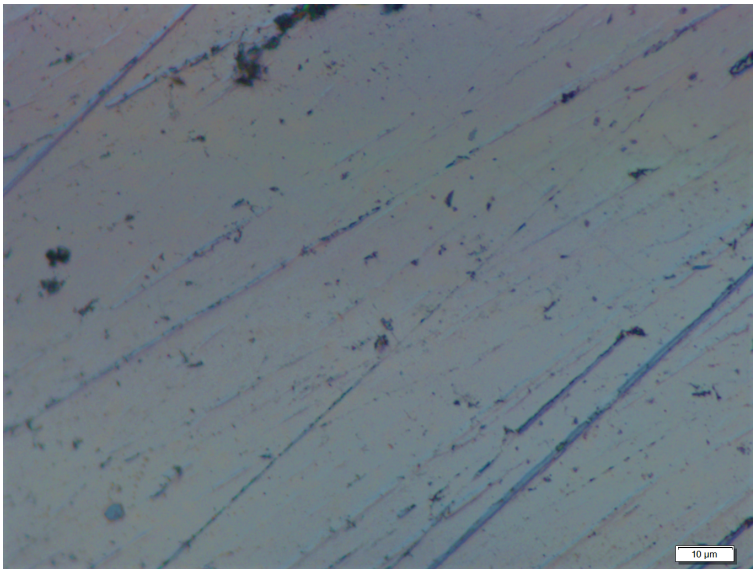

Figure 4: Unpolished, Unetched metal surface

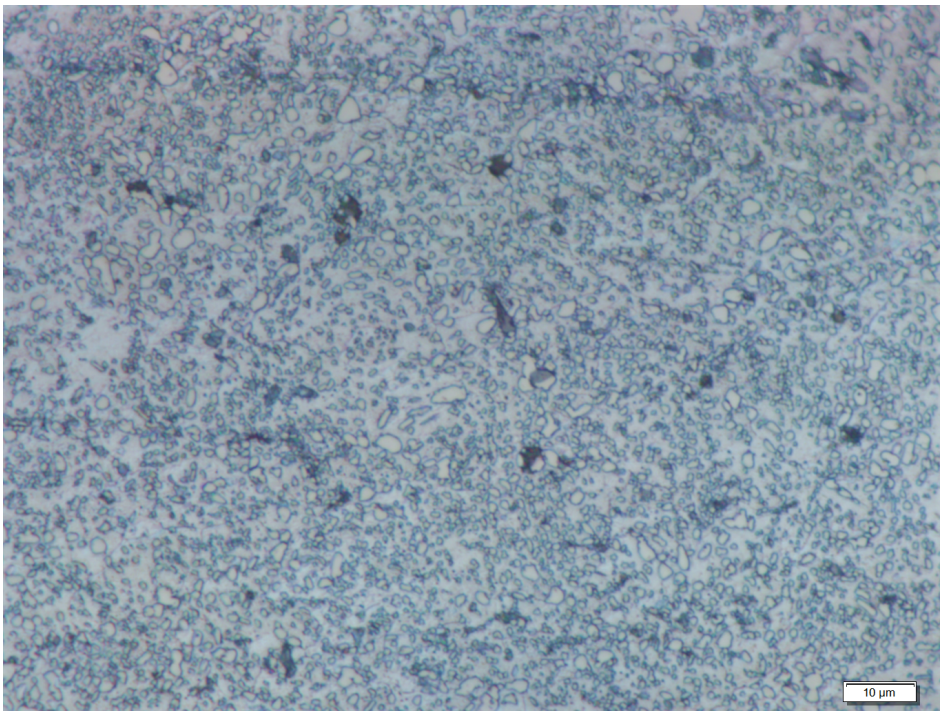

Figure 5: As Received Sample Two, Spheroidite etched with 2% Nytal

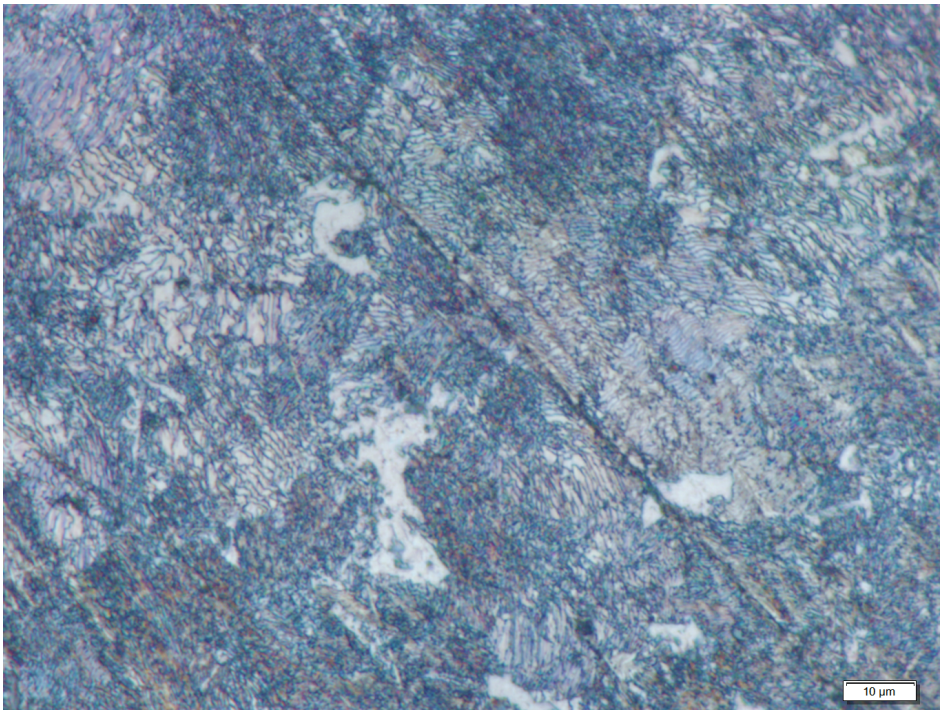

Figure 6: Furnace Cooled Sample Three, depicting Pearlite and Bainite etched with 2% Nytal

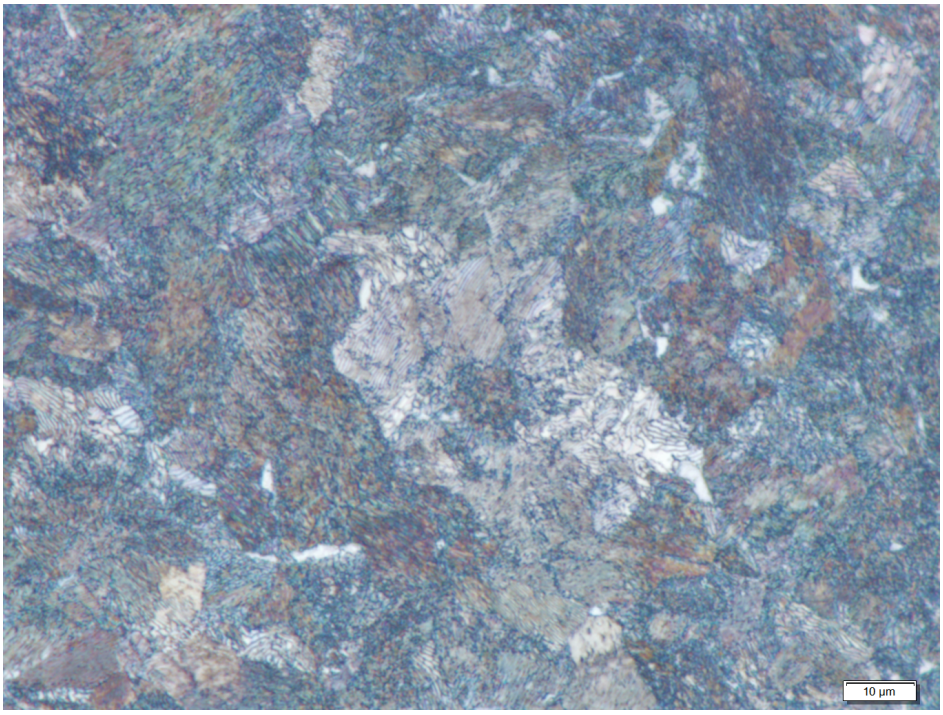

Figure 7: Sand Cooled Sample Two, depicting Pearlite and Bainite etched with 2% Nytal

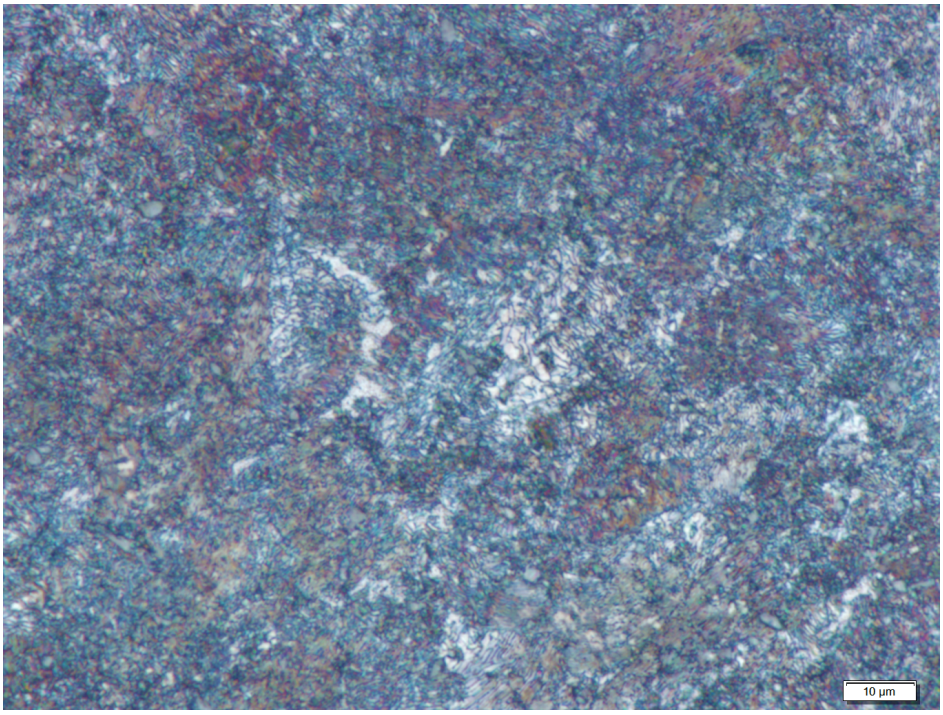

Figure 8: Air-cooled Sample Two, depicting Pearlite and Bainite etched with 2% Nytal

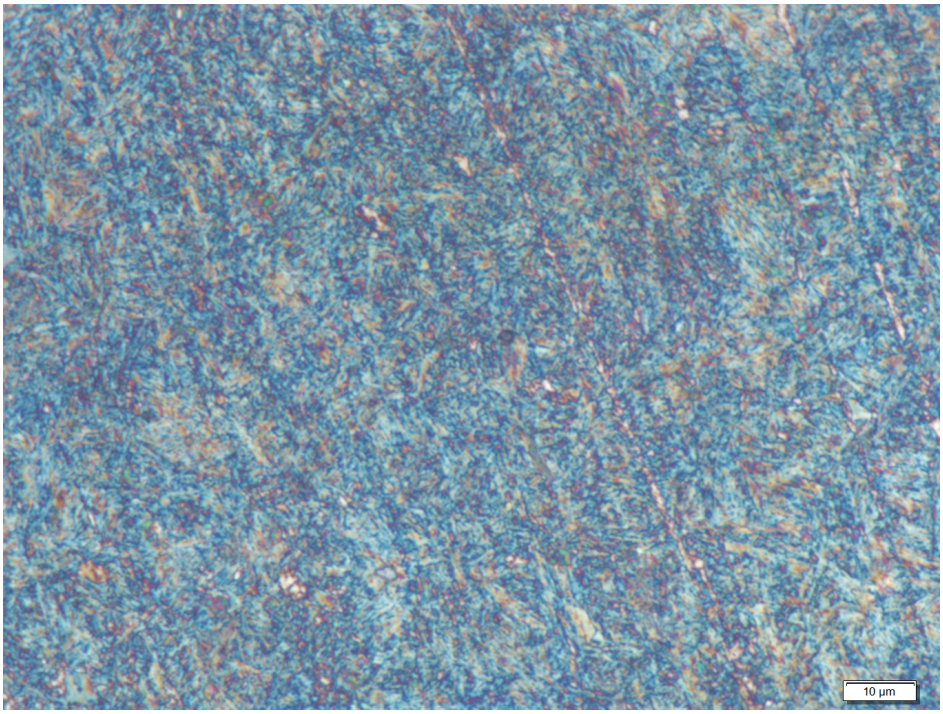

Figure 9: Water Quenched Sample Two, depicting Martensite etched with 2% Nytal

Discussion

Although the range of predicted microconstituents varied much more than the results, each microconstituent formation does make sense. Referring to figure 1, in order to reach the course pearlite that was the prediction for furnace cooling, the samples would have had to slowly cool over more than $3 * 10^4$ seconds. Temperature taken every few minutes from the furnace revealed that the cooling process for those two samples lasted from two to three hours, about $7.8 * 10^3$s. The furnace, Air exposed, and sand exposed samples must have all cooled within a few hours because their structures are very similar, judging both from the photos and the Rockwell C hardness values. The log plot in figure three predicts a cooling time for the furnace samples, but also gives a cooling curve that can be overlaid on the CCD to accurately predict a combination of pearlite and bainite formation. Water quenched samples cooled very rapidly, entering the water for thirty seconds and exiting almost at room temperature. Their Rockwell C hardness values, optical microscope images, and the continuous cooling diagram concur with the predicted outcome of martensite formation. The specimens that lacked information pertaining to heat treatment, the unaltered, as received specimens, contained spheroidite. The manufacturer shipped steel in this fashion probably because spheroidite is the easiest for the customers to form or mold into different shapes without treating.

Although there were no substantial sources of error in the experimentation that made the information unusable, there are several ways to improve the precision of this experiment. Heating the samples with a constant temperature (instead of the boxfurnace that varied over 100^oC), and touching the samples within the oven causing them to stick together introduced error into the experiment. There was also a ten minute range of time within which the samples were removed from the furnace, equivalent to 15% of the total heating time. Removing these error sources may result in more clear or regular formation of microconstituents. Handling every sample the exact same way until cooling avoids inconsistencies due to handling after heating. Improving the images of the microconstituents means removing error due to inconsistent etching time and pressure because of human error, and improved polishing.

Conclusions

Evident from this lab, the cooling time greatly alters mechanical characteristics of a metal. The shortest cooling time produces the hardest material, while the longest cooling time produces a softer material. Cooling time has to vary significantly for physical change, more than the few hours it took to cool the heated samples out in the open.

References

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[5] Callister, W. D. and Rethwisch, D. G., 2014, *Materials Science and Engineering An Introduction,* 9th ed., John Wiley & Sons, Inc., Hoboken, NJ.

I affirm that I have carried out my academic endeavors with full academic honesty. Signed, Roderick Landreth.