

Austempering AISI 5160

A Major Qualifying Project

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In

Mechanical Engineering

Ву

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This report discusses the result of different austempering holding times and their effect on the material's hardness, ductility and yield strength. AISI 5160 steel samples were heated until they were entirely austenite and then subjected to the austempering process, submerged in a 600°F salt bath for 1, 2, 5, 30, or 90 minutes depending on the sample. Some samples were also heat-treated using water quenching and annealing for comparison. The samples were then cut, ground, and polished using standard sample preparation methods for analysis using optical microscopy, scanning electron microscopy, and x-ray diffraction. Separate samples in the form of tensile testing bars were prepared similarly. Vickers microhardness, Rockwell hardness, and tensile testing was performed. Analysis of the samples and tests showed an increase in bainite as the austempering time increased, reaching 100% composition after 30 minutes. Elongation increased with the holding time, while hardness decreased.

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ABSTRACT:

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1.0 Introduction

American Iron and Steel Institute (AISI) 5160 steel is a widely used material across multiple fields. It is commonly used in the automotive industry to form multiple parts including bumpers, scrapers, and leaf springs [1]. Its properties lead the material to commonly being used for applications requiring a resistance to vibration and shock [1]. Other applications include fasteners, low performance bearings, and high tensile blades [2]. Due to its high carbon content, AISI 5160 welds poorly, something that limits its applications [2]. The composition of AISI 5160 is displayed in the table below.

Table 1 Composition of AISI 5160 [3]

Element	С	Mn	Р	S	Si	Cr	Fe
Nominal	0.56-0.64	0.75-1.00	0.035	0.040	0.15-0.30	0.70-0.90	balance
wt.%			(Max)	(Max)			

AISI 5160 can undergo many different heat treatments to produce a variety of microstructures, allowing for properties to be adjusted to better fill specific needs. The aim of this experiment is to produce the microstructure bainite in AISI 5160, examining how different austempering holding times impact the phase composition of the sample and its mechanical properties. Of importance is how the percentages of bainite and martensite relate to the hardness, elongation, yield strength, and ultimate tensile strength of the metal.

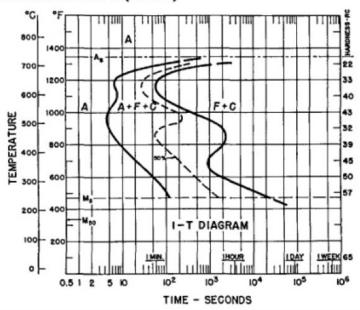
To obtain this information, samples will undergo examination via optical microscopy, scanning electron microscope, x-ray diffraction, Vickers microhardness testing, Rockwell hardness testing, and tensile testing. Tensile testing will be performed by an outside lab due to the metal requiring a stronger tensile testing machine than is available, while all other mechanical tests will be performed at Worcester Polytechnic Institute. Optical microscopy and scanning electron microscope analysis will be used to examine the microstructure of the samples, while x-ray diffraction will be used to identify the phase composition of each sample. The data collected over the course of the project will have further use in continued experimentation with AISI 5160 at Worcester Polytechnic Institute.

2.0 Background

Heat treatment is the process of controlling the heating and cooling rates of a material to obtain specific results. Results include changing the microstructure, chemical structure and grain size, in turn affecting the mechanical properties of the material. Through heat treating, the metal can be altered to better fulfill a purpose. Different types of heat treatments include normalizing, austempering, quenching, annealing and tempering.

This project involves the examination of the properties of AISI 5160 when raised to 1550°F (the austenitization temperature) and then quenched to austempering temperature and held for varying periods of time. The steel is quenched in an austempering salt bath before being left to air cool until it is room temperature. At 1550°F, the microstructure is that of austenite, face centered cubic (FCC). Different rates of cooling lead to the austenite transforming into other forms.

The rate and manner of cooling determines what microstructures will form in the steel. A time temperature transformation (TTT) chart is commonly used in determining how to cool a material to obtain specific results. The TTT chart for AISI 5160 steel is shown below. The y-axis is a linear representation of the temperature of the steel and the x-axis is time in a logarithmic scale. The path the steel takes as it cools over time is plotted to take a certain path to obtain a specific result. The curved lines already present on the chart note the points at which there will be 1% and 99% pearlite or bainite; with the upper curve representing pearlite and the lower curve representing bainite. The dotted line between them represents 50% of the transformation.



Type: 5160 Composition: Fe - 0.61% C - 0.94% Mn - 0.88% Cr Grain size: 7 Austenitized at 843°C (1550°F)

Figure 1. AISI 5160 TTT Chart [1]

Austenite is a common starting point for heat treatment as it can be transformed into many other forms and is obtained by raising the steel to a temperature above the eutectoid point. It can also be referred to as γ -Fe, or the gamma phase of iron. From this point, different cooling rates can produce different materials, including martensite, pearlite and bainite. Austenite as a final result is generally not desirable; retained austenite in martensite results in poorer physical properties.

Martensite is a body-centered tetragonal (BCT) phase formed when the heated iron-carbon alloy is rapidly cooled down to a low temperature. It is a single-phase structure that results from the transformation of austenite. The rapid cooling required to form it prevents the carbon diffusion in the alloy which results in a formation of ferrite and cementite, but can also cause some austenite to be retained, unable to transform fast enough. Pearlite is a phase containing ferrite and cementite [4]. Ferrite and cementite form in a lamellar structure from the redistribution of carbon in austenite. The material that loses carbon becomes ferrite, while the now carbon-rich material forms cementite. Cementite forms when the weight percentage of carbon to iron is over 6.67%. The lamellar structure comes from the ferrite and cementite forming in layers which can vary with the type of pearlite. Steels with this structure can be referred to as pearlitic steels. Pearlite is more ductile than austinite.

Bainite is the microstructure that forms during the Austempering heat treatment, it consists of ferrite and cementite, Bainite possess a higher yield strength and more ductility than the other microstructures which makes it good to be formed in different shapes. In another word it is a decomposition of austenite above the Ms (temperature where austenite begins to transform to martensite) and below the highest temperature point where the displacive transformation occurs.

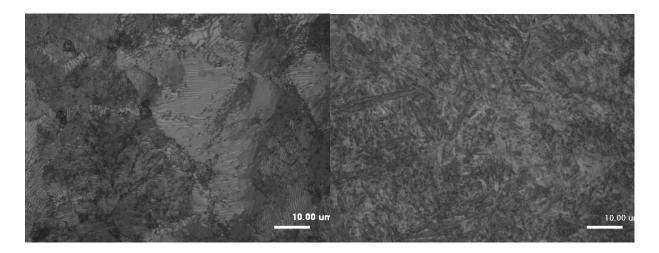


Figure 2. Pearlite (left) and Martensite (right). Captured on a Nikon Epiphot inverted microscope

3.0 Experimental Plan

The goal of this report is to examine the properties of AISI 5160 after various heat treatments including quenching, annealing, and austempering. For austempering, different austempering times were carried out to investigate the effect of martensite/bainite percentage on the mechanical properties of the austempered samples.

3.1 Quenching

To start the quenching process, the sample must be heated above the austenitic temperature for a period to ensure that the whole sample is turned into austenite. It is then removed from the furnace and submerged in a quenching medium. Common mediums are water and oil, with the main requirement being that the quenchant rapidly drop the temperature of the material being quenched. The quenchant can also be at any temperature the quenchant can support while maintaining a liquid state, but as the temperature change needs to be rapid, it is usually a much lower temperature than the furnace. Room temperature water is a common quenchant. The quenchant can also be stirred or agitated, which causes an even more rapid transition in temperature due to the increased interaction between the material and cool particles of the quenchant. Agitated baths, however, can lead to defects in the product, meaning the speed of quenching sometimes needs to be weighed against the tolerance for defects.

For the purpose of this report, quenching was done in room temperature water with an unagitated bath.

Type: 5160 Composition: Fe - 0.61% C - 0.94% Mn - 0.88% Cr Grain size: 7 Austenitized at 843°C (1550°F)

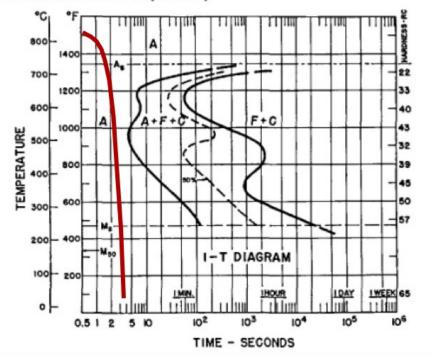


Figure 3 (original TTT [1])

3.2 Annealing

The process of annealing requires the material cool gradually and consistently. With metals, this is often achieved through leaving the material inside of the furnace that heated it. With AISI 5160 steel, the furnace is used to raise the internal temperature of the metal high enough that the entirety of the material turns to austenite. Once this is achieved, but before grain boundaries become too established, the cooling portion of the process begins. The furnace is turned off with the metal still in it, and both cool naturally until room temperature. This provides plenty of time for the carbon to diffuse, creating distinct regions of ferrite and cementite in the annealed metal. As such, a common result of this process is pearlite.

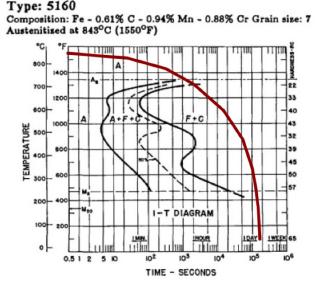


Figure 4 (original TTT [1])

3.3 Austempering

The process of austempering has the same start as annealing and quenching; converting the sample into austenite through heating in a furnace. The goal of austempering, however, is to hold the material at a specific temperature with minimal time spent in the transition from furnace temperature to the goal temperature. Both the holding temperature and time have a large impact on what phases form in the metal. As with quenching, the initial rapid temperature change prevents the diffusion of carbon and holding at a specific temperature encourages specific phase growth. A common result of austempering is bainite. A bath is used to get the material to the target temperature and hold it there. As the desired temperature is often too high for water and most oils, liquid salt is used. After the bath, the metal is left to cool in the air until it reaches thermal equilibrium.

Austempering for this project was done at 316°C in a sodium and potassium nitrate (NaNO3 and KNO3) bath.

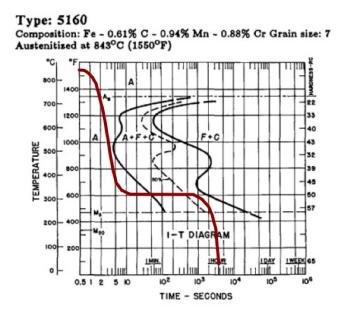


Figure 5 (original TTT [1])

4.0 Experimental Procedure:

4.1.0 Quenching and Annealing

4.1.1 Procedure:

The samples were heated in a Thermo Scientific Thermolyne Benchtop Muffle Furnace until the temperature throughout was above the austenite point at 843°C. After heating, the quenching sample was submerged in room temperature water until equilibrium was reached. During the quenching process, the water was left unagitated. The annealing sample was left to cool inside the now turned-off furnace until they both reached room temperature.

4.1.3 Sample preparation for test

The samples were cut into slivers using a Mark V 600 Series cutter, exposing both the rolling and drawing directions on broad surfaces suitable for analysis. These samples were mounted using a Buehler SimpliMet 3000 Mounting Press and ground using silicon/carbide resin paper with progressively finer grit on a Pace Technologies Nano 2000T Grinder-Polisher, finishing at 600 grit. Grinding was followed by a polishing process with Century Eplus AC Adjustable Speed Control polishing wheels. Samples were placed in an ultrasonic cleaner until the polishing solution was removed. An etchant of HNO₃ was applied to enhance structure visibility.

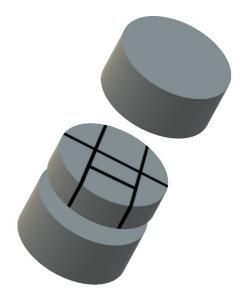


Figure 6: Sample Cut

4.1.4 Tests

These samples were used for microstructure comparison, and as such only underwent examination using optical microscopy using a Nikon Epiphot inverted microscope at magnification levels of 50x, 100x, 200x, 500x, and 1000x.

4.2.0 Austempering

4.2.1 Procedure

Two different types of samples were prepared for testing. Cylindrical coupons were used for optical and hardness testing, while specifically shaped tensile testing bars were used for tensile testing and scanning electron microscope (SEM) analysis. The samples were austempered at FCA, and spent either 1, 2, 5, 30, or 90 minutes submersed in an austempering salt bath at 600°F.

4.2.2 Sample Preparation for Tests

Tensile testing samples were received already formed from the company. After tensile testing, the fractured portions were preserved for SEM examination, and slices removed with the Mark V 600 Series cutter for optical microscopy analysis.

Cylindrical coupons were first cut into slivers and mounted, with different samples displaying grains in wither the drawing or rolling dimensions. Samples were cut, mounted, ground, and polished using the same equipment and processes used for the quenched and annealed samples. Mounted, ground samples were prepared for x-ray diffraction (XRD) and hardness tests.

4.2.3 Tests

As the focus of the project, multiple tests were used to characterize the different austempering samples.

4.2.3.1 Optical Microscopy

The austempering samples underwent the same analysis as the annealing and quenching samples.

4.2.3.2 X-Ray Diffraction

X-Ray diffraction through small-angle x-ray scattering was performed using a PANalytical Empyrean X-Ray Diffractometer using a chromium tube. Through the results, the phase composition of the samples was determined.

4.2.3.3 Mechanical Tests

4.2.3.3.1 Tensile Test

Tensile testing was performed by Touchstone Testing Laboratory, as the campus equipment could not complete the test. Testing provided information of the yield strengths, ultimate stresses, and percent elongation for the different times.

4.2.3.3.2 Hardness Test

Both Vickers microhardness and Rockwell hardness testing were performed on the austempered samples. Samples was placed in the hardness testing machines apparatuses and test for each sample with regarding to time. Vickers microhardness testing was performed using a Clark CM-400AT microhardness tester, and the Rockwell hardness testing used BJR The Stanley Rockwell Co. Hardness Test.

5.0 Experimental Results

5.1 Microstructure

5.1.1: As Received

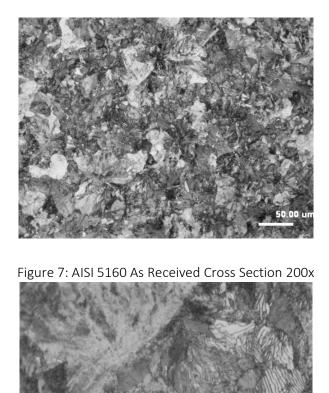


Figure 8: AISI 5160 As Received Cross Section 1000x

5.1.2 Quenched

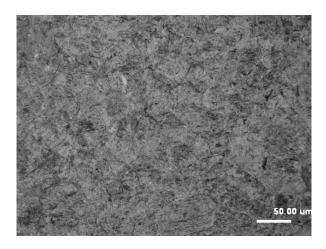


Figure 9: Quenched 200X under the microscope (Cross Sectional Center)

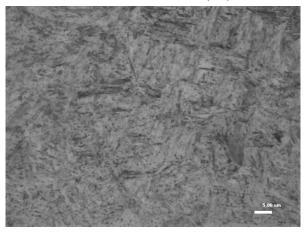


Figure 10: Quenched 1000X under the microscope (Cross Sectional Center)

5.1.3 Annealed

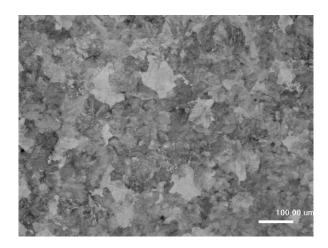


Figure 11: Annealed 100X under the microscope (Cross Sectional Center)

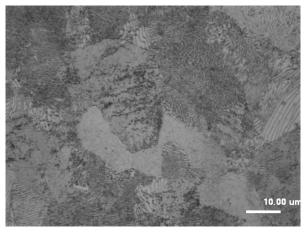


Figure 12: Annealed 1000X under the microscope (Cross Sectional Center)

5.1.4 Austempered

5.1.4.1 1 Minute Austempering Holding Time

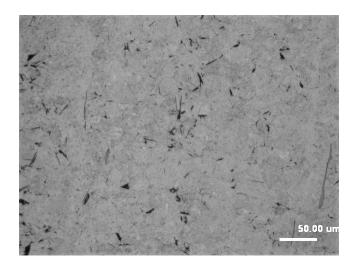


Figure 13: Austempered 1 Min 200X under the microscope (Center)

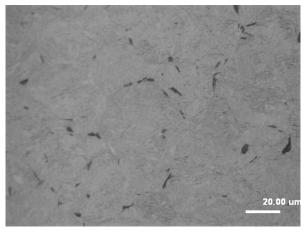
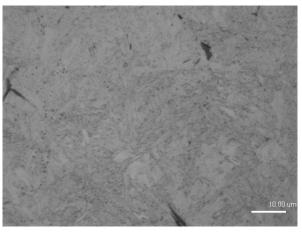


Figure 14: Austempered 1 Min 500X under the microscope (Center)



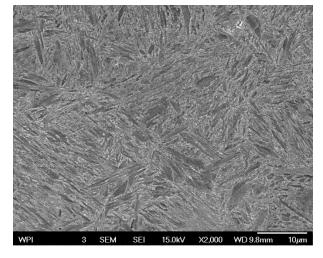


Figure 15: Austempered 1 Min 1000X under the microscope (Center)

Figure 16: Austempered 1 Min 2000X under the SEM (Center)

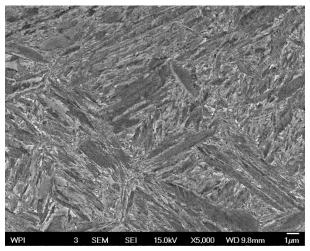


Figure 17: Austempered 1 Min 5000X under the SEM (Center)

5.1.4.2 2 Minute Austempering Holding Time

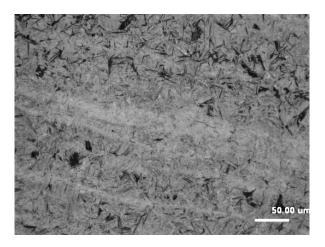


Figure 18: Austempered 2 Min 200X under the microscope (Center)

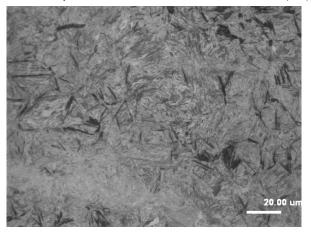


Figure 19: Austempered 2 Min 500X under the microscope (Center)

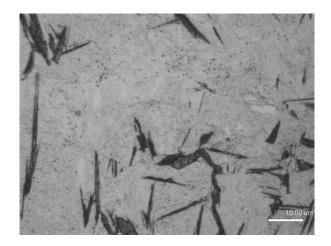


Figure 20 : Austempered 2 Min 1000X under the microscope (Center)

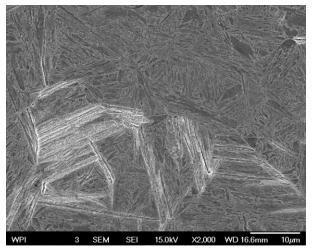


Figure 21: Austempered 2 Min 2000X under the SEM (Center)

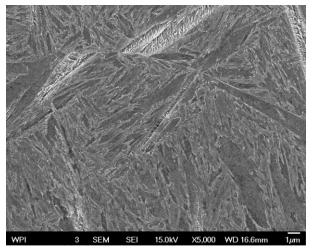


Figure 22: Austempered 2 Min 5000X under the SEM (Center)

5.1.4.2 5 Minute Austempering Holding Time

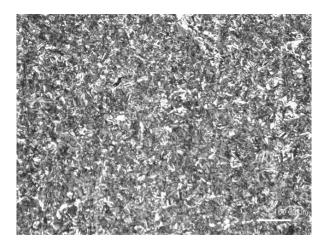


Figure 23: Austempered 5 Min 100X under the microscope (Center)

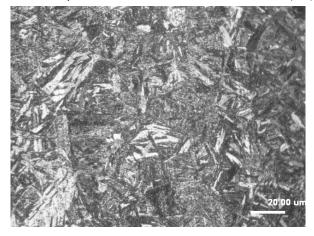


Figure 24: Austempered 5 Min 500X under the microscope (Center)



Figure 25: Austempered 5 Min 1000X under the microscope (Center)

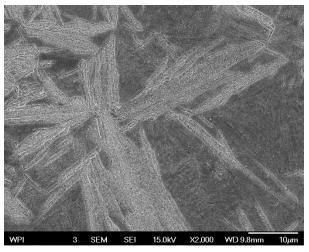


Figure 26: Austempered 5 Min 2000X under the SEM (Center)

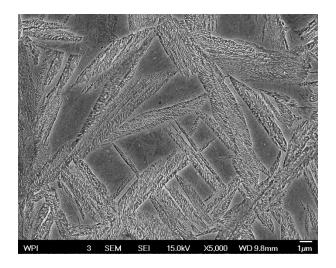


Figure 27: Austempered 5 Min 5000X under the SEM (Center) 5.1.4.3 30 Minute Austempering Holding Time



Figure 28: Austempered 30 min 500X under the microscope (Intermediate)

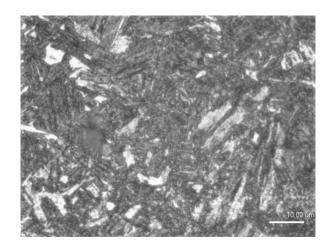


Figure 29: Austempered 30 min 1000X under the microscope (Intermediate)

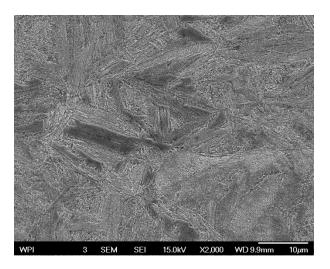


Figure 30: Austempered 30 Min 2000X under the SEM (Center)

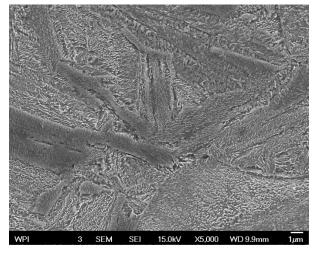


Figure 31: Austempered 30 Min 5000X under the SEM (Center)

5.1.4.4 90 Minute Austempering Holding Time

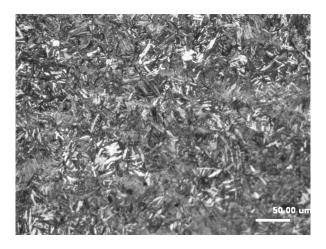


Figure 32: Austempered 90 min 200X under the microscope (Near surface)

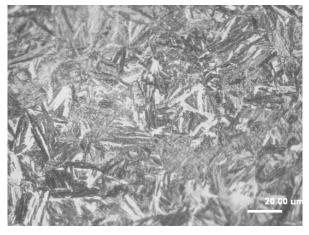


Figure 34: Austempered 90 min 500X under the microscope (Near surface)

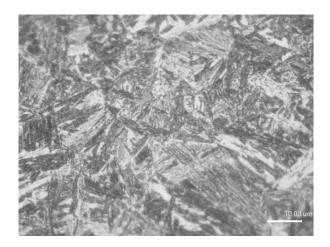


Figure 35: Austempered 90 min 1000X under the microscope (Near surface)

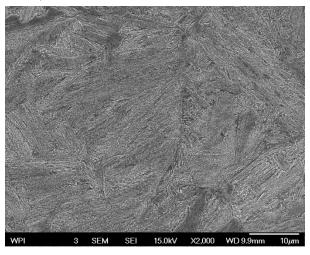


Figure 36: Austempered 90 Min 2000X under the SEM (Center)

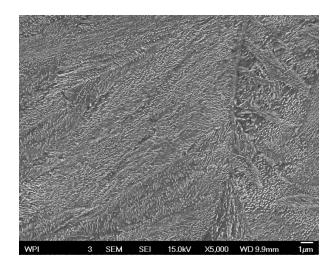


Figure 37: Austempered 90 Min 5000X under the SEM (Center)

5.2 Hardness

distance	1min	2min	5min	30min	90min	5min-2	5min-3
from							
surface(µm)							
400	832	818	705	515	549	648	663
800	815	815	710	522	550	684	693
1200	800	768	675	532	562	772	726
1600	829	814	668	530	541	753	736
2000	826	784	648	528	551	764	682
2400	806	766	630	533	537	782	634
2800	807	764	620	519	540	721	619
3200	821	798	632	533	555	748	630
3600	815	764	602	534	551	736	583
4000	831	798	606	526	543	758	656
4400	836	764	623	532	547	711	617
4800	821	790	639	543	539	748	697
5200	815	772	691	521	534	703	759
5600	831	783	683	538	548	745	798
6000	840	802	668	532	551	728	755
6400	827	818	736	539	541	686	683
6800	818	800		547	537	672	716
7200	830	821		535			
7600	835	826		533			
8000		809		539			
8400		815		540			

Table 2 Vickers Microhardness Results

Table 3 Rockwell Hardness Results

Time	Test 1	Test 2	Test 3	Test 4	Test 5	Test 6	Average
(Minutes)							
1	63.000	62.000	63.000	63.000	63.000	62.500	62.750
2	62.000	61.200	60.200	61.000	61.000	59.800	60.867
5	55.000	54.000	55.200	54.500	56.000	55.000	54.950
30	50.800	50.000	49.500	50.000	48.500	48.000	49.467
90	47.000	48.200	49.500	47.400	49.000	49.000	48.350

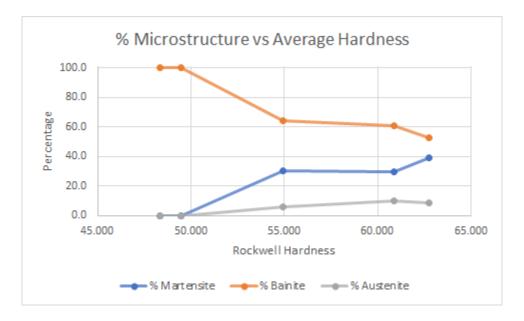


Figure 38 : Microstructure vs Average Hardness

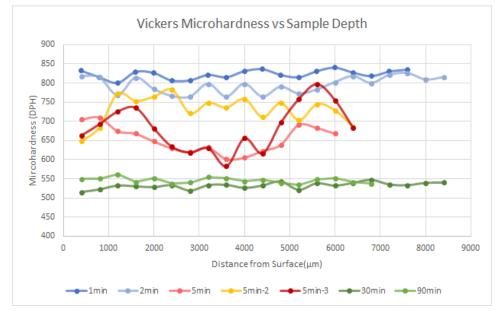


Figure 39 Vickers Microhardness vs Sample Depth

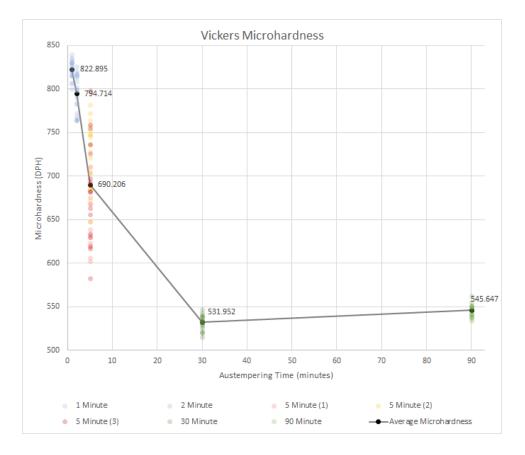


Figure 40 Vickers Microhardness

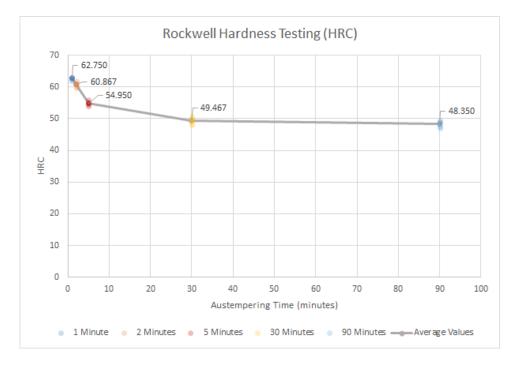


Figure 41 Rockwell Hardness Testing (HRC)

5.3 Tensile Test

Material	Nominal Sample Diameter	Nominal Gage Length	Austenitizing Temp (°F)	Austenitizing Time @ Temp (min)	Austempering Temp (°F)	Austempering Time @ Temp (min)	Sample Label	Yield Stress (ksi)	Ultimate stress (ksi)	Elongation (%)
5160	0.25"	1"	1550-1575	25	600	1	600-1min-1	-	158.8	0.64
							600-1min-2	-	177.8	0.80
							600-1min-3	-	154.4	0.63
						2	600-2min-1	-	167.9	0.74
							600-2min-2	-	137.4	0.54
							600-2min-3	-	177.4	0.79
						5	600-5min-1	-	164.1	0.74
							600-5min-2	-	152.5	0.66
							600-5min-3	172.4	201.4	1.06
						30	600-30min-1	199.2	251.4	10.03
							600-30min-2	199.6	249.6	10.33
							600-30min-3	199.4	251.4	10.04
						90	600-90min-1	198.9	245.7	10.85
							600-90min-2	198.0	245.9	10.28
							600-90min-3	196.0	243.6	10.38
5160	0.25"	1"	Sample without heat treatment					90.8	124.8	13.67

Table 4 Tensile Test Results

(The sample without heat treatment will be represented as having spent 0 minutes being heat treated)

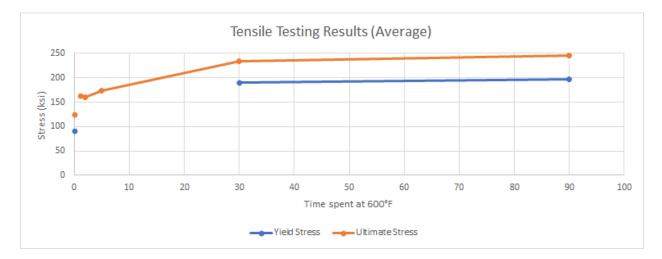


Figure 42 Tensile Testing Results (Average)

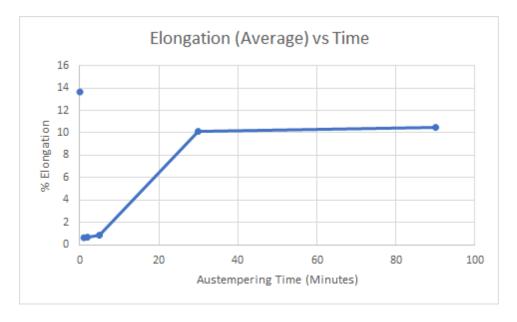


Figure 43 Elongation (Average) vs Time

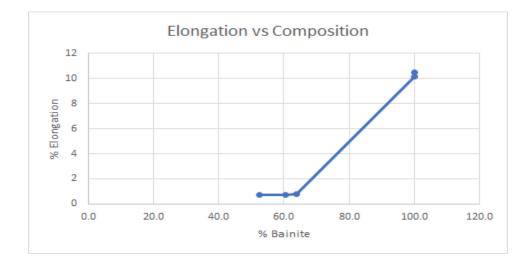


Figure 45 Elongation vs Phase Composition

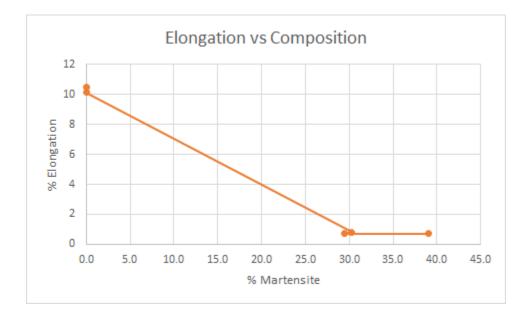


Figure 46 Elongation vs Phase Composition

5.4 X-Ray Diffraction and Rietveld Refinement

Table 5 Rietveld Refinement Results

Time (minutes)	% Martensite	% Bainite	% Austenite	
1	39.0	52.5	8.5	
2	29.5	60.6	9.8	
5	30.3	63.9	5.8	
30	0.0	100.0	0.0	
60	0.0	100.0	0.0	

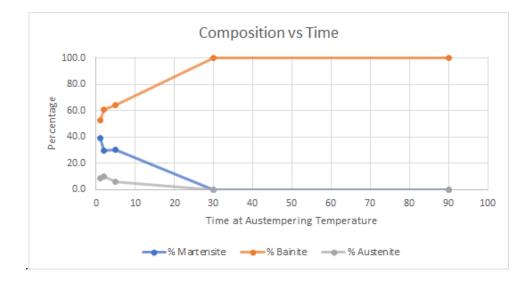


Figure 47 Composition vs Time

6.0 Discussion:

In general terms, the longer the steel was submerged at temperature for the austempering process, the more ductile and less hard it became, as presented in Figure 40, Figure 41, and Figure 43. The most obvious exception to this is the sample that wasn't heat treated, which has both the greatest elongation at 13.67% of its total length and the lowest yield and ultimate tensile strengths at 90.8 and 124.8 ksi, respectively. As it hasn't undergone any heat treating, it is represented in the graphs as an unconnected data point. These features can be explained by through microstructure analysis, which as shown in Figure 8, clearly displays the lamellar structure of pearlite.

Even with a very short period of time in the austempering salts, the amount of bainite in the sample after treatment is above 50%, as shown in Table 5. Between 1 and 2 minutes, a sharp increase in the presence of bainite and a comparative decrease in the presence of martensite occurs, visible in Figure 47. This is the fastest change of phase composition in the samples, as the rate of change becomes more gradual after 5 minutes.

An interesting change in elongation percentage occurs between the 5- and 30-minute samples. Figure 45 depicts the percentage of bainite versus the elongation of the sample. Figure 46 depicts a similar change. At some point between 5 and 30 minutes, the increase in elongation percentage changes from gradual to sharp, corresponding to some percentage of the overall composition remaining bainite. It is also somewhere in this time period that the retained austenite is eliminated Up to 5 minutes, the tensile testing bars do not undergo visible plastic deformation, with the fractures up to this point lacking the distinct dimples and curving that characterize a ductile fracture. Of the 5-minute samples, only one had a distinguishable yield strength, with more distinct plastic deformation showing past 30 minutes. Past 30 minutes, the samples are entirely composed of bainite. As a result, ductility, yield strength, and ultimate tensile strength values stay roughly the same.

The increase in bainite also corresponds to a decrease in hardness, visible in both the Vickers microhardness and Rockwell hardness tests. Through taking an average of the different depths of the Vickers microhardness test for each sample, one can see a distinct decrease in hardness as submersion time increases. A point of note here is the 90-minute average in Figure 40, which is greater than the 30-minute average. When tracking the Vickers microhardness measurements throughout the sample (Figure 39), one can see that while the values of the 90minute sample are generally greater than the 30-minute sample, they difference between the two generally decreases the deeper into the sample the measurements get. The increase is slight and still far below the value for 5 minutes. This increase is not present in the Rockwell hardness tests (Figure 41).

Microstructure analysis through both optical and scanning electron microscopes show progressive changes as more time is spent austempering. Supported by the data from XRD, one can see the increase in bainitic structures, with the images from the 1-minute sample showing a much less bainite-dense structure. Both the 30- and 90minute samples display similar structures, which aligns with both being 100% bainite, where the 5-minute sample with 30.3% martensite is distinctly different.

7.0 Conclusion

It can be concluded that as austempering time increases, the microstructure of AISI 5160 retains more bainite, reaching 100% composition in about 30 minutes. The steel is 100% austenite above 1550°F, and depending on the cooling method and time, transforms into different phases, as shown in the TTT diagram for AISI 5160 shown in Figures 1, 3, 4, and 5. The unstable austenite transforms to bainite during the salt bath austempering, and upon cooling the remaining austenite transforms to martensite. As a result, the microstructure that results from incomplete austempering contains bainite and untempered martensite with some retained austenite. Experimental observations show the percentage of bainite in the composition increases and the percent martensite decreases with increased austempering time at 600°F.

As shown in Figure 43, elongation increases with an increase in the austempering holding time. For a 5 minute sample, the elongation in the tensile testing device was under 2%, while elongation was just over 10% for the 30 minute sample. Plastic deformation is likewise not pronounced until the sample is 100% bainite, with recordable yield stresses occurring consistently after the 30-minute point. Appendix 8.1 shows the fractures that resulted from tensile testing. Visual inspection of the tensile testing bars reveals ductile behavior starting at the 30-minute sample, further supporting this finding. As bainite content increases, the yield strength (when applicable) and ultimate tensile strengths increase, visible in Figure 42.

In both the Vickers microhardness and Rockwell hardness tests, the hardness of the sample decreases as the austempering holding time increases. Figure 40 and Figure 41 show this relationship. This also reflects a decrease in hardness with an increase in bainite content, reflecting the softer nature of bainite when compared to martensite.

8.0 Appendix:

8.1 Tensile Testing Images



Tensile Testing Bars (1 minute)



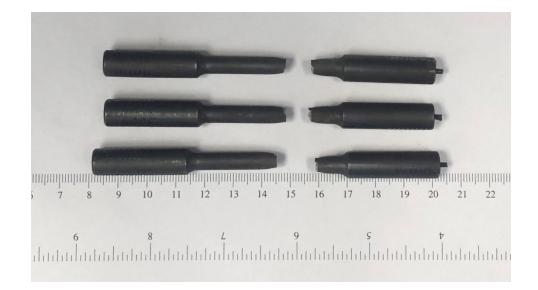
Tensile Testing Bars (2 minutes)



Tensile Testing Bars (5 minutes)

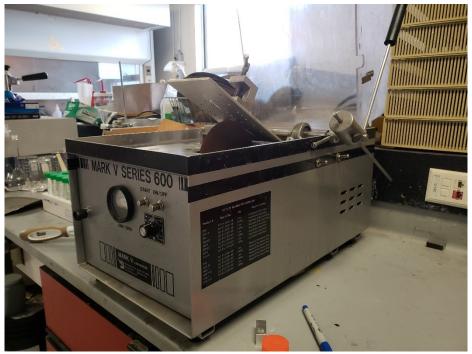


Tensile Testing Bars (30 minutes)



Tensile Testing Bars (90 minutes)

8.2 Machinery



Mark V Series 600 Saw



Ace Technologies Grinding Machine



Polishing Machine used for the experiment



SimpliMet 3000 Mounting Machine



Thermo Scientific Furnace used for Quenching and Annealing



Nikon Microscope used for optical tests

8.3 Microscopy Images

A file containing all optical and scanning electron microscopy images will be uploaded with this report.

References:

- Han, Xue & Barber, Gary & Zhang, Zhenpu & Wang, Bingxu & Zhu, Jian & Shi, Jing & Sun, Xichen. (2018). Austenite-Bainite Transformation Kinetics in Austempered AISI 5160 Steel. European Scientific Journal. 14. 10.19044/esj.2018.v14n12p1.
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