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JOMINY END QUENCHING OF 4140 STEEL: THE EFFECT OF TIME AND TEMPURATURE ON AUSTENITIC GRAIN GROWTH

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Jominy End Quenching of 4140 Steel:

The Effect of Time and Temperature

on Austenitic Grain Growth

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Abstract

This project investigated the effects of austenitizing temperatures and time on the hardness of 4140 steel. The most common test for studying these effects is the Jominy End Quench test. In this test a 140mm long and 25.4 mm in diameter sample is heated into the austenite range for selected temperatures and times and water spray quenched at one end, producing a varying cooling rate across the sample. Currently the Jominy End Quench test focuses more on the hardenability of the steel. An improved model for correlating the Jominy End Quench curves and the resulting grain size curves proposed introduce a better defined heating and cooling cycle. The improved model obtained will optimistically result in less time spent in research and development. Through the process of preparing, etching, and observing the samples under an optical microscope, photomicrographs are obtained. The experimental results show a difference between the prior austenitic grain size among the different heating times and temperatures used. The conclusion compares the effects of temperature and time in the grain growth of the prior austenitic grain size.

Introduction

Determining a relationship between the heating, cooling process will help the heat treating industry by reducing the time spent in the research and development stage. As a corollary this means less money spent. This relationship is an expansion of the Jominy End Quench test and has not yet been defined which is the scope of this paper. The Jominy End Quench Test determines the capacity of steel to harden under defined heating and cooling parameters. The addition of the grain size relationship coupled with the results of the test would produce better defined heating and cooling rates. This allows the engineer to correctly identify the correct steel alloy and heat treatment depending on its intended application, such as rock breaker pistons, or aircraft undercarriages. Extensive research has already been done in hardenability of steel, but has not yet made any significant correlations between hardness and grain size. It is imperative to find a relationship because grain size influences the outcome of many mechanical properties of steel such as hardness, ductility and yield strength. A desired austenitic grain size can be obtained experimentally by using a specific temperature and time.

1 Background

1.1 Heating

Through the experimentation of different temperatures and times it is possible to determine the optimal temperature and time for a specific grain size. These two factors play a major role in the austenitic grain growth of 4140 steel. The first place to look to determine which temperatures to use can be found by using the phase diagram. The phase diagram shows at what temperature the material reaches its austenizing temperature. This is the temperature at which the carbon redistributes and the structure becomes crystalline. On the following diagram this section is represented by the γ on the left hand side of the diagram. To find the correct temperature the weight percentage of carbon needs to be known. In this case it is 0.4% which is determined by the last two digits of the steel. A vertical line is then drawn up to the lower austenite boundary and then a perpendicular line drawn to the temperature on the side. In the case of 4140 steel it is 843°C. The following figure is the phase diagram for steel.

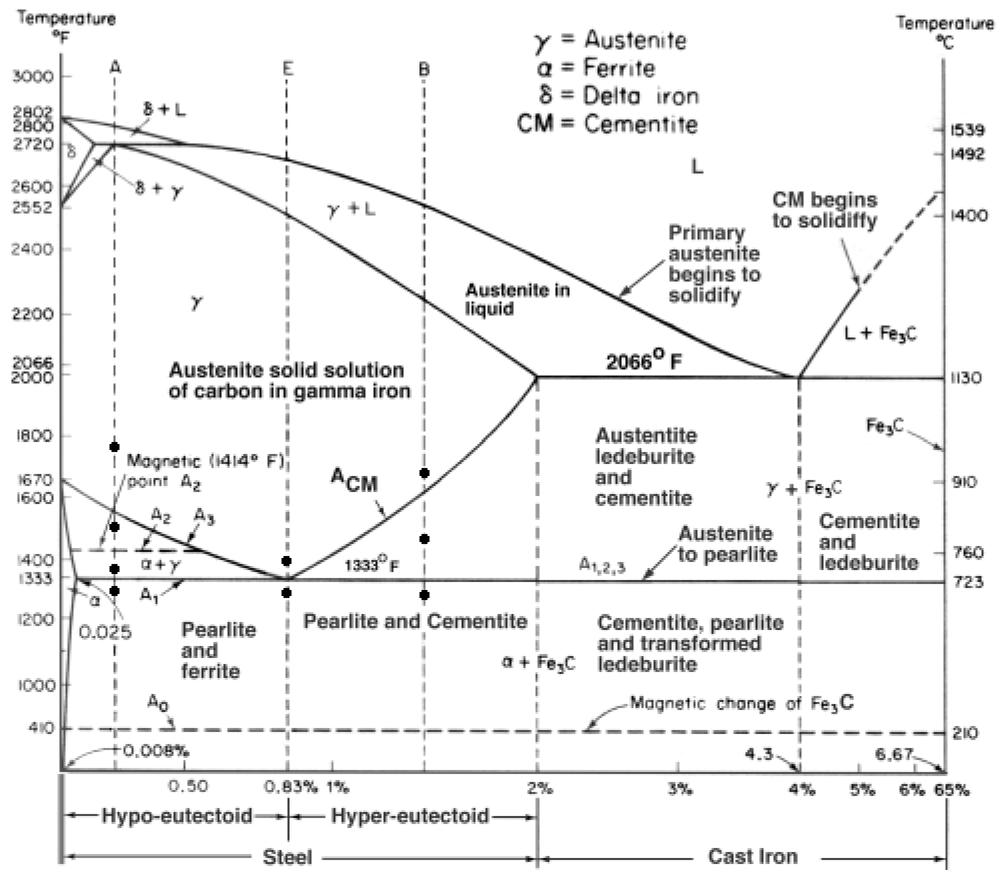

Figure 1: phase diagram of steel

The closer you are to the austenizing temperature the slower the grain growth of the structure. Given this fact, more time spent at the austenizing temperature the larger grain size. When heating a jominy bar in the furnace it is required to add a half an hour to the desired time spent in the furnace. The addition of the half hour is called normalizing. This process provides the sample time to reach the desired temperature and become uniform on a structural basis.

1.2 Quenching

Quenching is a process where a heat treated part is cooled down rapidly. There are many ways to quench a part as well as multiple quenching mediums such as air, oil, water, brine or spray. This process is just one step in the manufacturing process and depending on the desired result, one of the many methods of quenching can be used. As a result of the cooling process, the structure of the metal is altered. Through the use of a Time-Temperature-Transformation diagram the desired result can be used to determine the proper cooling process. From the following diagram you can see the cooling time is the determining factor in the resulting microstructure. Given a desired microstructure, the cooling rate can then be found. Based on that information the correct quenching process can then be determined.

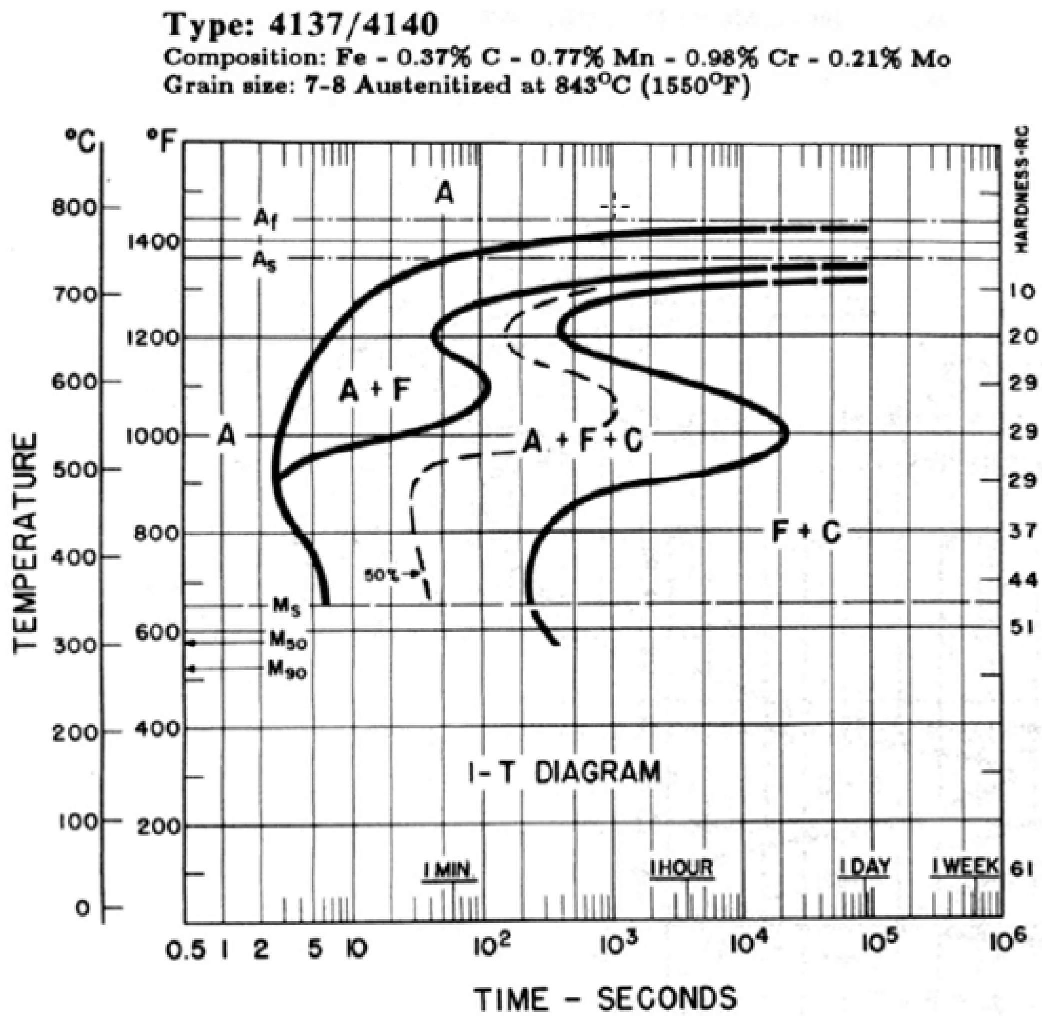

Figure 2: TTT diagram for 4140 steel

In this project, quenching is used to cool the part and just as important to stop the grain growth process. The bigger a grain grows the higher the hardness value. In this project the focus is on the austenitic grain growth. When the bar is quenched, it immediately changes in the microstructure at the end of the bar that gets sprayed with the water. The part of the bar furthest from the quenched end still allows for microstructure changes, because it does not cool as quickly. The end of the bar that cools first remains austenite while the other end changes to pearlite and bainite. In this project I am only interested in the austenite region.

2 Experimental Plan

4140 steel was used and heated to 850°C, 900°C and 950°C. At 900°C and 950°C the heat times are as follows a half hour, two hours, and four hours. At 850°C two bars the heat times are a half hour, and four hours. Through the heating and quenching process the desired result is to see a change in hardness data but the expectation is to see higher Rockwell C numbers towards the quenched end of each jominy bar and lower values towards the other end. There should also be a difference of rate of chance of the hardness values in the bars heated to 850°C and the bars heated to 950°C. The austenitic grain size should be smaller for the 850°C bars and larger in the 950°C bars. In addition to seeing microstructure changes on each bar, I expect to see a difference from the bars heated to 850°C and the bars heated to 950°C.

3 Procedure

3.1 Heating

The heat treating process has three stages. The first stage is heating the metal slowly to a uniform temperature. This ensures the sample is at the desired temperature throughout the part. The second stage is soaking. This is when the sample is held at a given temperature for a given time. The third and final stage is cooling the metal to room temperature (13). The third stage will be explained in further detail in the following section. For the first stage each sample was held for a half hour for to confirm a uniform temperature. There were 8 jominy bars of 4140 steel used in this experiment. The three temperatures used were 850°C, 900°C and 950°C. At both 900°C and 950°C samples were heated in the furnace for a half hour, two hours and four hours, for 850°C the times are, a half hour and four hours. The electric furnace used took two and a half hours to get up to 950°C and two hours to heat up to 850°C. The samples were hung in the middle of the furnace through the use of a thermocouple wire. The wire was wrapped around the screw that came out of the top of the bar. The other end of the wire was then secured to a metal bar two feet above the furnace. Heat resistant padding was used to cover the opening at the top of the furnace to reduce heat loss. In order to reduce the time spent using the furnace all samples for each temperature were heated at the same time. The length of each heat cycle allowed this to be done with ample time to remove it from the furnace and begin the quenching process. Heat resistant gloves were worn to remove each sample from the furnace. A set of wire cutters were used to cut the wire and a long pair of thongs were used to hold the sample via the screw. The sample was then placed in the quenching apparatus.

3.2 Quenching

The quenching process in this test is the same for each sample regardless of temperature or time. The sample is taken out of the oven and placed into an apparatus that has it rest a half an inch away from a water pipe. As seen in the following figure.

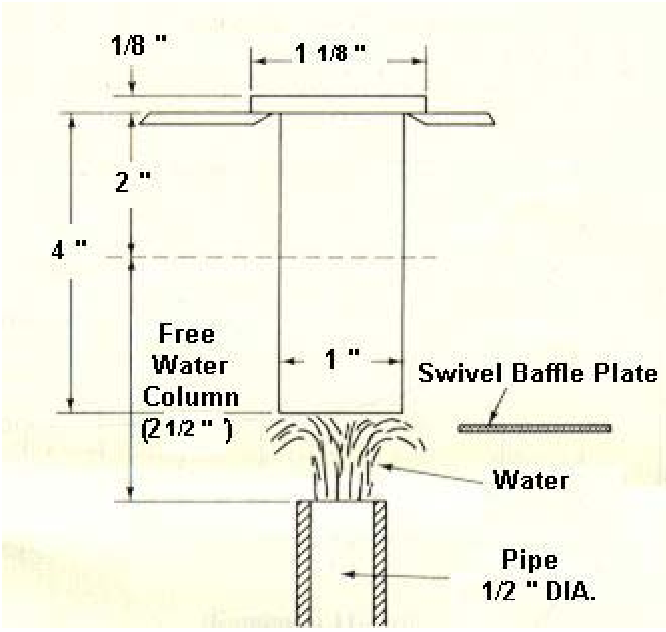

Figure 3: Jominy end quench fixture

This step has to be completed within 15 seconds of removal from the furnace. It is important to not allow the ambient air temperature to cool the part prematurely. This would severely alter the results of the test. The apparatus used did not allow the bars to rest any distance away from the water spout. To solve the problem two round metal discs were used to prop up the jominy bar. This gave the clearance from the water spout needed to properly quench the bar. Once the sample is securely set, the water is turned on so that it continually sprays the bottom of the bar. The sample takes three to five minutes to change from bright red to black. The water is left on for 10 minutes in total to make certain it is at room temperature. The water is collected in a tub below the water spout and has to be siphoned to be emptied. There is a video on YouTube® that shows this test being performed; the link is located in the reference section. This was helpful in the beginning stages to see firsthand what the process entailed (14). When the bars were cooled to room temperature, they were labeled using a permanent marker. A labeling system helps to keep track of the samples for the later testing.

3.3 Machining

Upon cooling the bars are taken to be machined. This required the learning how to use the CNC machine. It is a complex machine and proper training is required in order to use it. A training and safety tutorial was required by the machine shop and WPI to be to ensure proper usage. Subsequently a test had to be taken. Once these steps were completed the machining process could begin. The washer and screw assembly was removed from the bar and it was placed into the CNC machine. The next step was to remove 0.0015 inches of material off of the surface. In order for this to be done a computer program was written telling the machine what to do. This was accomplished with the assistance of the lab director. A surface mill was used in order to accomplish this task. A low RPM of the mill was used to assure the bar was not reheated during the milling process. This part of the machining process was repeated for each sample. The next step in the process was to flip each bar over 180° and 0.0015 inches of material was removed. The flat surfaces that resulted allowed an area for the hardness data to be obtained on both sides of each bar.

3.4 Hardness

A hardness indenter measures a materials resistance to deformation using a diamond and weight. There are several types of diamonds that can be used, but there are two that are more commonly used. One is a Vickers diamond and the other is a Knoop diamond. The Knoop diamond is the most commonly used. It is more elongated than the Vickers diamond and therefore is able to impact the material closer to the surface of a part. The Rockwell scales are most commonly used in lab testing facilities for measuring hardness. The different scales correspond to different loads used depending on what the hardness of the material is thought to be. The Rockwell C scale is the most common one since it pertains to the hardest materials. In the case of this project a hardness value of Rockwell C 50 is expected near the surface. A Knoop indenter was set into the hardness indenter. Prior to testing, calibration of the machine was performed to ensure the accuracy of the data to be obtained. This is the first step in obtaining hardness values as set forth in ASTM E140. In order to properly calibrate the machine a calibration block must be used. This is a block that has a specific hardness value. The indention on the block has to be within a few hardness points of that number to be valid. When obtaining data, each indention has placed approximately two and a half times the size of the diamond away from the previous indention. Twenty indentions were taken on each side of all the bars. The data was then put into a spreadsheet as to easily compare the differences.

3.5 Etching

There are two steps that must be done before etching can take place. The samples have to be prepared, this entails grinding and polishing. These two steps play a significant role in the outcome of the etching and photos. The sanding process involves four steps. It involves sanding the part on 60 grit paper, 120 grit paper, 320 grit paper and finally 600 grit paper. The sand paper is placed on metal platform that rotates at a high RMP. The polishing process is the next step in the preparation process. A felt pad is placed on a similar metal platform that rotates at a high RPM. A mixture of alumina powder and water is poured onto the pad. The next step in the process is making the etchant. Every etchant has a recipe. The recipe can contain acids, toxic or corrosive ingredients. When creating an etch it is important to wear safety gloves and goggles. In this project a modified 4% picric acid was used.

3.6 Metallographic Examination

The microstructure of each sample was observed using an optical microscope. Each sample was viewed at 50X to better understand what was going on in the microstructure. The samples were viewed from the quenched end to the non quenched end. The optical microscope was equipped with a camera with which photomicrographs were taken. Photomicrographs were taken so an evaluation of the grain size could be done. The evaluation process for grain size is done using ASTM E112. This specification covers the different methods acceptable for the determination of grain size. It explicitly covers each method in a step by step fashion. The two more common methods are the Comparison method, and the Heyn Intercept method. The comparison method entails taking a photomicrograph and simply eyeballing it and comparing it to standard grain size photos in ASTM E112. It is a basic way of evaluating the grain size, but in most instances it is sufficient. This project focuses on the determination of the austenitic grain size. Austenitic grains are often difficult to see, which makes using the comparison method much too difficult. The Heyn Intercept method makes determining the grain size easier. It is more complicated than the comparison method but often gets more accurate results. The way to perform this test is by overlaying three lines across a picture. One of the lines is horizontal, one vertical and on diagonal. While running a pen along each of the lines, each grain boundary is counted. Those numbers are then plugged into a formula in ASTM E112. That formula gives you the average grain size number.

4 Results

4.1 First Heat Treating Cycles

The first two heating trials ran garnered results that did not reach the minimum Rockwell C numbers. The expected hardness number was Rockwell C 50, the hardness numbers of HRC 25 obtained suggested that the bars were not getting heated to the correct temperature. The furnace read the desired temperature so it was assumed the part was also at that temperature. It did not register that the bars were not the bright red color they were supposed to be when it was removed from the furnace. Nor did it convey that the lack of steam upon cooling that something had gone wrong. It took until the fourth step in the process to realize something had gone wrong. Notwithstanding it happened on a second separate occasion until it was realized something was obviously wrong.

The bar was stood up on a ceramic piece that closed the bottom of the heating cylinder. A meeting with the professor ensued and resulted in the suggestion that the jominy bar be hung towards the middle of the furnace somehow.

4.2 Second Heating Cycle

The furnace was getting hotter in the middle of the cylinder because, the heating element is located there. It took some time to find something thin enough and durable enough to withstand the heat. It made sense to use thermocouple wire. This allowed the bars to reach the desired temperature. The corrections were made and the following tables show the HRC results. This data is also arranged from quickest cooled end to the slowest.

850°C 0.5 hr side 1	850°C 0.5 hr side 2	850°C 4 hr side 1	850°C 4 hr side 2
61.8	56.5	64.9	57.1
52.6	56.9	58.1	57.4
53.7	55.5	58.2	54.7
53.8	56.7	57.5	52.8
54.5	54.7	54.6	51.3
52.6	47.5	51.8	48.5
50.1	49.6	48	41.2
45.4	42.5	48.3	38.3
40	39.4	39.1	36.9
36.6	40	36	34.6
38	34.6	34	37
36.7	37.5	36.8	34.9
41.6	32.6	32.2	39.5
38	31.9	30.7	34.5
32.8	32.9	30.6	34.8
32.9	27.4	30.7	34.5
33	31.8	30	33.5
36.4	30	29.8	
31.9	32.1		

Table 1: Hardness values for 850°C

900°C 0.5 hr side 1	900°C 0.5 hr side 2	900°C 2 hr side 1	900°C 2 hr side 2	900°C 4 hr side 1	900°C 4 hr side 2
60.2	62.5	59.8	65.4	59	52.3
60.1	60.7	61.5	58.5	57.8	49.7
58.5	58.2	57.9	55.7	59.5	47.6
62.2	58.3	53.9	54.5	63.1	47.6
57.4	58.2	57.8	51.5	54.6	49.5
58.4	57.6	54.5	53.5	51.7	47.9
54.5	53.8	47.7	48.4	49	45.1
52.1	50.6	44.7	43.9	46	36.7
48.8	50.6	42.4	39.1	42.6	39.7
49.7	46	41.6	43.2	45.9	38.5
40.7	37.4	44.2	38.8	40.5	37.7
42.8	37.2	38.9	36.2	43	37.4
44.1	33.8	42.7	38.7	39.8	37.5
37.7	38.7	38.9	33.7	38.2	36.9
39.1	33	39.3	31.8	38.9	31.5
34.5	31.2	41.7	35.3	39.9	33.8
35.2	35.5	37	35.8	35.2	34.3
36.6	35.2	39.6	36.1	34	38.7
34.9	35.5	41.5	34.1	30.5	38.3
37.8	30.6	39	37.3	29	36.6
33.5	29.8	35.6	37.2	32.8	37.9
32.8	28.5	34.9	34.9	31.3	33

Table 2: Hardness values for 900°C

950°C 0.5 hr side 1	950°C 0.5 hr side 2	950°C 2 hours side 1	950°C 2 hours side 2	950°C 4 hr side 1	950°C 4 hr side 2
58.5	65.9	56.5	55.8	58.4	54.8
58.6	62.3	55.5	55.2	54.2	53.6
58.5	55.7	55.6	54.2	52.8	54.6
57.7	56.2	54.1	54.5	50.1	50.8
57.3	55.6	49.4	52.2	49.4	49.1
55.6	52.9	44.9	48.7	46.4	51.3
54.7	51.3	40.6	44.8	48.2	45.3
53.2	47.1	37.9	42.5	43.6	47
52.2	46.5	35.6	37.2	44.3	45.2
50.2	42.6	33.6	33.8	43	43.7
48.5	44	33.4	33.7	41.4	38.1
44.7	45.2	34.2	31.9	36.6	37.1
41	40.6	30.1	31.4	35.2	33.3
40.8	42.1	32.9	30	33.8	32.8
36.3	34.2	31.4	30.2	35.2	29.9
33.8	34.6	30.4	30.3	30.2	29.2
33.3	31.5	32	30.3	29.6	28.4
32.1	34.1	29.9	27.4	28.8	28.6
31.8	34.2	28.2	28.8	26.5	27.8
31.2	33.1	29.6	28.3	27.4	25.3
31	31.1			27.9	26
26.7	28.9			26.9	26.8

Table 3: Hardness values for 950°C

4.3 Etching and Grain Size

4% picric acid etchant was used to provide a representation of the microstructure of each sample. They were viewed using an optical microscope and photomicrographs were taken. Those photos were then used to determine the grain size. The photos did not provide adequate representations to show the grain size. A modified 4% picric acid etchant was used based on a suggestion from and outside source. This etchant provided a better representation and sufficient photomicrographs were taken. The suggested etchant was similar to the one used in the previous trial with one minor addition. A wetting agent was added to the etchant and brought out the microstructure sufficiently. The following photo shows an example of the grain size at 50X magnification.

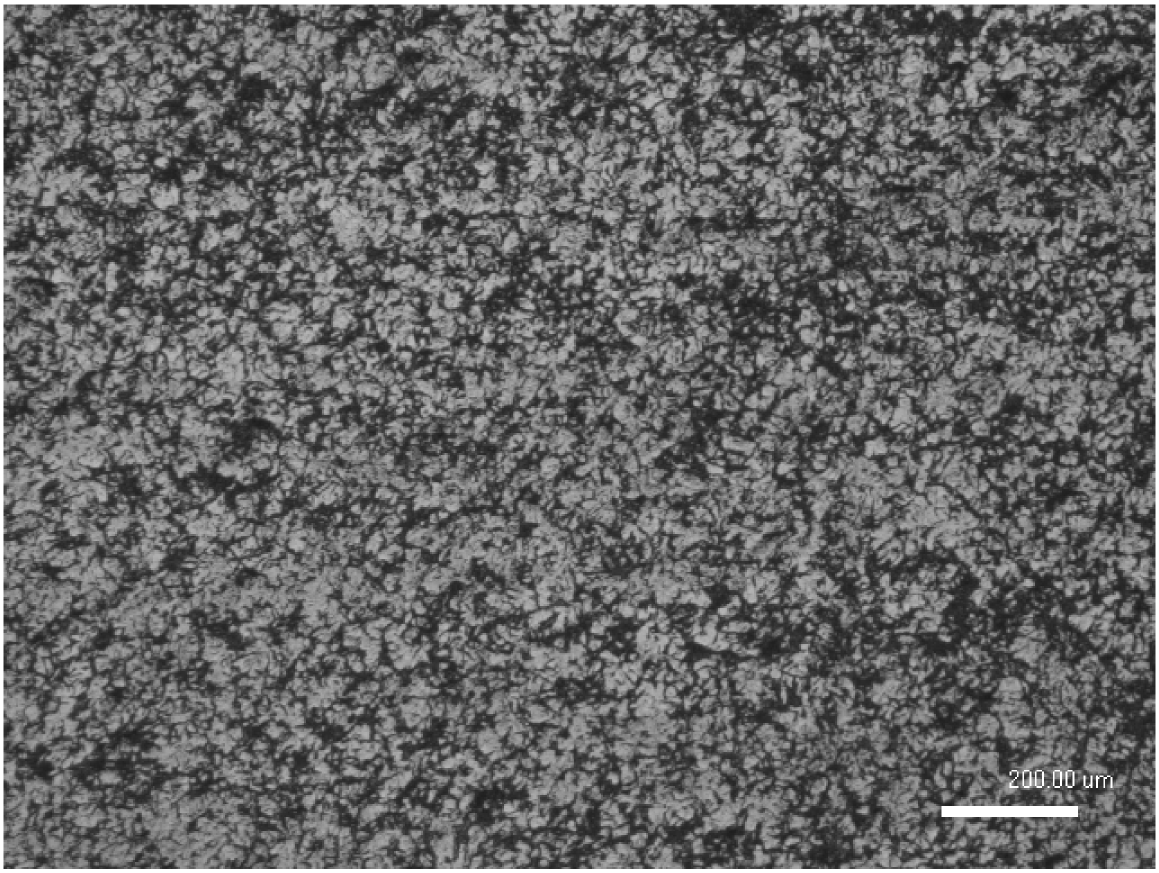

Figure 3: 850°C 0.5 Hr grain size micrograph

The grain size photos were used to determine the grain size of each sample using the Heyn Intercept method.

5. Conclusions

The hardness data suggests that there is a difference in the microstructure though out the part. The data obtained through the hardness testing did not show any real significant difference between the different heating temperatures. The difference between the times at a specific temperature was slightly different. The hardness values followed the hypothesized curve was encouraging. All of the photomicrographs were taken at 50X. The modified 4% picric acid etch used provided ample contrast to obtain photomicrographs.

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