

# Grain Growth in AISI 4140

A Major Qualifying Project submitted to the faculty of WORCESTER POLYTECHNIC INSTITUTE in partial fulfillment of the requirements for the Degree of Bachelor of Science in Mechanical Engineering

Submitted by:

Omari McPherson

Submitted to:

Project Advisors:

Richard D Sisson Jr Md Maniruzzaman

April 30, 2008

## Abstract

Grain growth during heat treatment has a negative effect on fatigue. As a result, grain growth kinetics have been determined for 4140 steel in order to predict the effective time for heat treating. A series of samples were heat treated at 850 °C and 900 °C and held for 60, 120 and 180 minutes. It was experimentally determined that grain growth kinetics were faster for samples with a higher austenizing temperature. Diffusivity growth constants were found to be 2.682E-06  $(mm^2/min)$  for the samples heated at 900C and 1.902E-06  $(mm^2/min)$  for those treated at 850°C. The maximum heating time *t* before the deterioration of the material properties, namely fatigue, was calculated to be 510min at 850°C and 442min at 900°C.

# Acknowledgements

I would like to thank Professor Richard Sisson Jr. for his advice and his help with this project. I would also like to thanks Md. Maniruzzaman and Boquan Li for their assistance in making sure I was on the right track and had all that I needed to successfully complete this project.

Table of Contents	
Abstract	i
Acknowledgements	ii
1 Introduction	1
2 Background	3
2.1 4140 Steel	3
2.2 Yield Stress and Heat Treatment	4
2.3 Heat Treatment of Steel	6
2.4 Method for revealing prior-austenite grain size	8
3 Experimental Plan	.11
4 Methodology	.12
4.1 Heat Treatment	. 12
4.1.1 Temperature-Time Data Acquisition and Furnace calibration	. 12
4.2 Sample Preparation	. 14
4.2.1 Cutting	. 14
4.2.2 Mounting	. 14
4.2.3 Grinding and Polishing	. 15
4.2.4 Etching	. 15
4.3 Image Acquisition and Optical Microscopy	. 16
5 Experimental Results	.17
5.1 Measurement and Analysis	. 18
5.1.1 Mean Grain Diameter	. 18
5.1.2 Average Grain Diameter	. 20
5.1.3 Grain Growth	. 20
6 Discussion	.23
7 Conclusion	.23
8 References	.24
9 Appendix	.26
9.1 Appendix A: Experimental Data	. 26
9.2 Appendix B: Micrographs	. 27

# List of Figures

Figure 1: Stress-Strain Diagram/Tensile Test [5]	5
Figure 2: Fe-Fe3C equilibrium phase diagram. [7]	7
Figure 3: Isothermal transformation diagram for 4140 Steel [7]	8
Figure 4: Heat Treatment Setup	13
Figure 5: Photo Micrograph of Sample 5	16
Figure 8: Temperature time curve for the first 15minutes after immersion	17
Figure 9: Temperature Time Curves. Held at 850 C for 1hr. Quenched in water.	17
Figure 6: Heyns Linear Intercept Method	19
Figure 7: Equivalent ASTM grain-size number	20
Figure 10: Grain Diameter vs. Time	21
Figure 11: Grain Diameter vs. Time	22

3
1
1
)
)
)

# **1** Introduction

Low alloy medium carbon steels are used in a variety of automotive and general engineering applications, particularly where strength and impact toughness are required.[1,2] Typical alloying elements include nickel, chromium, molybdenum, manganese, silicon or boron. The purpose of these elements are to optimize the mechanical properties, offering better toughness and material properties superior than plain carbon steels. For example, carbon increases hardness while combinations of chromium and molybdenum, increase elevated temperature strength. Although the chemical composition is important, the influence of microstructure on mechanical properties is more significant. [2] The only practical method of changing microstructure is heat treatment.

Grain growth as a result of heat treatment can affect properties such as strength and fatigue life. It is reported that grain size is one of the factors that cause fatigue failure. Taylor et al show that the threshold value for fatigue crack growth increases with increasing grain size and decreasing yield strength. [10] Furthermore, according to the Hall-Petch relationship, as the average grain size decreases, the yield strength of the steel increases. [6] Grain growth is related to austenizing temperature and hold time, as well as the original grain size of the steel before hardening. [5]

Therefore the goal of this project was to identify the effects of austenizing temperature and hold time on grain growth in a heat treated medium carbon low alloy steel (AISI 4140). To accomplish this goal a series of samples were heat treated at 850°C and 900°C for 60, 120 and 180 minutes. These samples were then cut, mounted, polished and etched in order for microstructure analysis to determine the prior austenitic grain size. The microstructure of each

1

sample was observed by high resolution optical microscopy. Using this methodology, its grain size was measured and grain growth kinetics were determined.

### 2 Background

#### 2.1 4140 Steel

AISI 4140 is a medium carbon chromium - molybdenum steel used for a wide range of applications in most industry sectors because of its low cost, good forgeability and machinablility. [1] Typical applications are: Bearings, Bushes, Cylinders (Various), Gears, Conveyor Rolls, Hydraulic Shafts, Hollow Shafts, Hollow Parts (Various), Nuts and Rings.

This steel grade responds readily to heat treatment and is comparatively easy to machine in the heat treated condition. The chemical composition is listed in Table 1.

Chemical Composition	Min.%	Max.%
Carbon	0.38	0.43
Silicon	0.15	0.04
Manganese	0.75	1.00
Chromium	0.80	1.10
Molybdenum	0.15	0.25
Phosphorous	0	0.04
Sulphur	0	0.015

Table 1: Chemical Composition of AISI 4140 Steel. [9]

The chromium content provides good hardness penetration and the molybdenum imparts uniformity of hardness and high strength. [2] The material properties are show in table 3. This steel resists creep in temperatures up to 540 °C and maintains its properties even after long exposure at relatively high working temperatures. The wear resistance can be considerably increased by flame hardening or induction hardening particularly with nitriding. [9]

Dramanting		Conditions		
roperues	Properties			
<b>Density</b> (×1000 kg/m <sup>3</sup> )	7.7-8.03	25		
Poisson's Ratio	0.27-0.30	25		
Elastic Modulus (GPa)	190-210	25		
Tensile Strength (Mpa)	1020.4			
Yield Strength (Mpa)	a (Mpa) 655		normalized at 870°C	
Elongation (%)	17.7	25		
Reduction in Area (%)	46.8			
Hardness (HB)	197	25	annealed at 815°C	
Impact Strength (J) ( <i>Izod</i> )	54.5	25	annealed at 815°C	

Table 2: Mechanical Properties of AISI 4140 Steel. [13]

#### 2.2 Yield Stress and Heat Treatment

Yield strength is the stress at which a material starts to deform plastically. Prior to this a material will deform elastically, returning back to its original shape once the load is removed. As shown in the curve in figure 1, there is no definite point on the curve where elastic strain ends and plastic strain begins; the yield strength is chosen to be that strength when a definite amount of plastic strain has occurred. During the yielding stage, the material deforms without an increase in applied load, but during the strain hardening stage, the material undergoes changes in its atomic and crystalline structure, resulting in increased resistance of material to further deformation.



Figure 1: Stress-Strain Diagram/Tensile Test [5]

Yield strength is an important property in most engineering design. A component designed to support a force during use, must have a yield strength high enough to prevent plastic deformation. The value is influenced by factors such as raw material quality, chemical composition, heat treatment process, etc. However, major microstructural changes result from the different heat treating parameters.

The relationship between grain size and yield strength is given by equation 2-1, known as Hall-Petch equation. Where  $\sigma_y$  is yield strength,  $\sigma_o$  is the friction stress opposing dislocation motion, and K is the stress intensity factor and d is the mean grain size.

$$\sigma_y = \sigma_o + \frac{\kappa}{\sqrt{d}} \tag{2.1}$$

Mean grain size is determined by equation 2-2 which shows the relationship between grain growth and hold time.

$$d^2 - d_o^2 = kt \tag{2.2}$$

The results reported by many researchers indicate that the yield strength increases following the Hall-Petch equation, but if the grain size reduces to the nano-range grain boundaries start to slide. This means that by changing grain size one can affect dislocation movement and yield strength. [12]

In environments where crack nucleation is predominate; i.e in the low stress, high-cycle regime, grain size is directly proportional to fatigue life. [8] As grain diameter grows, planar slip increases which causing the grain boundaries to control the rate of cracking. The heat treatment parameters used to control grain size are considered to be mainly austenizing temperature, soaking time, control of quench rate and tempering temperature. These processes result in microstructural variations, especially the mixture of phases, and grain size.

#### 2.3 Heat Treatment of Steel

The heat treating of steels results in a variety of microstructures. The important equilibrium phases are shown in the Fe-Fe<sub>3</sub>C equilibrium phase diagram (Figure 2). [7] Typical heat treatment processes usually start with an austenization treatment where the steel is heated into the austenite region. Here the ferrite phase transforms and all the carbides are dissolved in the austenite. Slow cooling to below the eutectoid temperature results in the formation of ferrite and cementite, as shown in Figure 3. Depending on the carbon content of the steel and the cooling rate, this might appear as proeutectoid ferrite and pearlite. Slower cooling rates produce coarser microstructures and very slow cooling rates, or holding at a temperature just below the eutectoid temperature, can result in a microstructure consisting of spherical cementite grains in a field of ferrite. [7] Heat treatments designed to strengthen steel, however, are based on the formation of bainite, martensite and other phases, which can be formed by rapid cooling from the austenite field and holding at a relatively low temperature. [7]



Figure 2: Fe-Fe3C equilibrium phase diagram. [7]

The grain boundaries of the austenite phase produced during the treatment mentioned above are the prime location for nucleation of the new phases. For the slow cooling of a medium carbon steel from the austenite phase field, a proeutectoid ferrite would nucleate and grow from the prior austenite boundaries. When the temperature reaches eutectoid temperature the remaining austenite would transform into the eutectoid phase.



Figure 3: Isothermal transformation diagram for 4140 Steel [7]

### 2.4 Method for revealing prior-austenite grain size

The determination of prior-austenite grain size has been the subject of metallurgical research efforts for many years. Metallurgical laboratories are often required to perform prior-austenite grain size determinations on martensitic steel components that have been heat treated. [4] In heat-treated steels, the carbon is distributed though the specimens leaving a martensitic microstructure. The grain size cannot be measured in martensite especially in medium and high carbon steels, thus one must measure the size of the parent austenite grains, formed during heat

treatment. The difficulty here lies in the etching procedure required to reveal the grain boundaries.

There are several standard methods for determination of prior-austenite grain size: McQuaid-Ehn carburizing, oxidation, copper diffusion, and thermal etching. The carburization method by McQuaid and Ehn consists of delineating the austenite grain boundaries by the precipitation of a proeutectoid cementite network. The sample is austenitized, and then pack carburized at 925 C° for 8 hours, and slowly cooled to room temperature. [11] Oxidation etching consists of heating a polished face of hypoeutectoid in a furnace with an oxidizing atmosphere. The grain boundaries are revealed by preferential oxidation or by grain-boundary decarburization. [4] Thermal etching or grain boundary grooving in vacuum is an established method for delineating austenite grain boundaries in a wide range of steels. The method consists of heating the polished specimen to the desired temperature into the austenite range under a vacuum of 10-3mm of mercury. Grooves form at the austenite grain boundaries as a result of preferential evaporation and surface tension effects and can be seen after cooling to room temperature. [3]

Although these methods may occasionally be mandated by material or procedural specifications, they are typically not preferred because they have a tendency of altering the as-received microstructure. These processes can also be labor-intensive and costly. [4] Nevertheless, the key element to revealing the prior austenite grain size is the etching technique. Special etchants must be used and often times they are not satisfactory in revealing a high percentage of grain boundaries.

Chemical etching solutions are widely used. Etching reagents based on saturated aqueous picric acid plus a wetting agent are reported to give the best results in quenched and tempered

9

steels. [4] The most successful etchant for revealing prior-austenite grain size in martensitic and bainitic steels is Bechet and Beaujard's etch [3].

# **3** Experimental Plan

The initial material was a bar of 4140 steel approximately 12 inches long. The rod was sectioned into 16 discs approximately <sup>1</sup>/<sub>4</sub>" thick using a MarkV CS600 abrasive saw in conjunction with a SiC blade. Extra discs were cut for use as control samples and samples instrumented with thermocouples.

After looking the different standard methods for determination of prior-austenite grain size: McQuaid-Ehn carburizing, oxidation, copper diffusion, and thermal etching, it was determined that they would be too costly and time consuming. The most appropriate strategy for this project would be to heat the sample into the austenite phase region and cool at a controlled rate. Since 4140 is a hypo-eutectoid steel with medium carbon content, the austenite grain boundaries are outlined by ferrite. After polishing and etching with the right etchant, microscopic examination may reveal the ferrite network around prior austenite grains.

To identify the effects of austentizing temperature and hold time on grain growth in a heat treated medium carbon low alloy steel, a number of samples were heat treated at different austentizing temperatures. Heat treatments were performed at the temperatures of 850 and 950, degrees Celsius for durations of 60, 90, 120 minutes. This required a total of 6 samples.

Heating Matrix				
Quench: Wate	er			
Heat Rate: Im	mersion			
Temp.				
Hold Time	850 °C	900 °C		
60min	Sample 1	Sample 2		
120min	Sample 3	Sample 4		
180min	Sample 5	Sample 6		

# 4 Methodology

The goal of this project is to identify the effects of austenitizing temperature and hold time on grain growth in a heat treated medium carbon low alloy steel (AISI 4140). This required the delineation of the prior-austenitic grain size, which can be problematic and time consuming depending on the method used. To accomplish the goal data was obtained by heat treating 6 samples at different austentizing temperatures. These treated samples were then cut, mounted, polished and etched in order for microstructure analysis to determine the prior austenitic grain size. The microstructure of each sample was observed by high resolution optical microscopy. Using this methodology, different microstructures were identified and grain growth was measured. In this chapter, the heat treatment process, sample preparation, image acquisition and sample measurement and analysis techniques are presented.

#### 4.1 Heat Treatment

This section outlines the method used to heat treat each sample.

#### 4.1.1 Temperature-Time Data Acquisition and Furnace calibration

The time-temperature data was collected by using a sample of 4140 steel, with a diameter of 4cm and thickness of 1.5cm instrumented with a thermocouple. A hole was drilled through the geometric center with 1/16" drill bit. The thermocouple used was an Omega brand K-type thermocouple. The hole was just slightly larger than the thermocouple to create a snug fit. Powdered graphite was place in the hole to eliminate air gaps between the thermocouple and the sample. The thermocouple was attached to an Omega HH506RA multilogger thermometer. To acquire the temperature of the sample and time, the HH506RA was used in conjunction wit a Compaq Armada laptop computer, running Windows XP. Omega software for the HH506RA was installed allowing for time and temperature data to be saved to an excel spreadsheet. The program also allowed the data capture rate to be changed.



Figure 4: Heat Treatment Setup

The furnace used for all heat treatments was a Thermolyne 1400 Furnace, model number FB1415M. The thermocouple was tested and there was a significant difference between the furnace display temperature and the thermocouple reading. The offset was about 30  $C^0$  so for each target temperature the furnace was adjusted to compensate.

Appropriate temperatures were set based on the furnace calibration. Once the temperature was achieved, the thermocouple sample was placed in the furnace. The time temperature curve for that test is shown in Figure 6. It took 20 minutes for the thermocouple sample to reach the desired temperature and from that empirical test, the heat treatment duration timer could be started without needing a thermocouple. When the desired time duration was achieved, the

furnace door was opened and the sample was removed and immediately quenched in room temperature water.

To observe grain boundaries after heat treatment, samples would need to be sectioned to reveal an inner surface, devoid of any anomalies or oxidation that the surface of each sample might contain. Standard practices for mounting and polishing were used to provide a clean polished surface to view under optical microscope.

#### 4.2 Sample Preparation

#### 4.2.1 Cutting

Heat treated samples were each sectioned on a Mark V Series 600 abrasive saw, model number 11-1180. A control sample that had not been heat treated was also sectioned on this saw. The blade used was a SiC for medium steels. It had a 4 inch diameter and was 0.012 inches thick. The blade specified is made specifically for hard alloys. A constant supply of coolant was maintained in the saw for the duration of the cutting operation. The samples were later mounted.

#### 4.2.2 Mounting

After each sample was sectioned to reveal an internal face, one half of each was mounted in preparation for polishing and etching. The samples were mounted on a Buehler Simplimet II mounting machine with a 1-1/4in ram using Beuhler Phenolic powder (green - catalog number 20-3300-400). The samples were kept at a pressure of 4.2 ksi or 29 MPa and heated to 120 degrees Fahrenheit. The samples where then allowed to cool to 60 degrees Fahrenheit, and taken out of the mounting machine. Once mounted, the backs of the sample mounts were inscribed with a sample number.

#### 4.2.3 Grinding and Polishing

The mounted samples went through a series of grinding and polishing phases. Initially, each sample was ground on 180 grit silicon carbide paper to get consistent scratches in one direction and maintain a flat surface. Samples were ground for the duration required to achieve the desired flatness and surface finish on Buehler grinding paper. Grits used were 180, 240, 320, 400, 600, 1200, and finally 2400 Buehler Microcut silicon carbide grinding paper. Samples were then washed and rinsed with acetone to remove any loose particles.

Cleaned samples were then polished using 3 micron alumina polishing solution until the surface could no longer be improved. Lastly a Buehler polisher was used with Buehler Meastermet one micron colloidal silica polishing suspension, to achieve a final mirror polish on each sample. Once it was determined that the sample surface was well ground and polished, the samples were ready for etching. The polishing cycle was often repeated when samples needed to be re-etched in order to remove each previous etch from the sample surface.

#### 4.2.4 Etching

To see the microstructure, etching is needed, with the most commonly used being 2% Nital. It was determined from several sources that the most successful etchant for revealing prior austenite grain size is Bechet and Beaujard's etch. [3] The solution consisted of 2g picric acid, 100mL water and a wetting agent. 3 common wetting agents were investigated: Sodium tridecylbenzene sulfonate, sodium dodecylbenzene sulfonate and Teepol. Dodecylbenzene was used because it was reported to be successful and was easiest to obtain. [4] The sample was immersed vertically in the solution at room temperature and etched in an ultrasonic cleaner for 7 minutes. 1% HCl was added to the solution because it improved the results. Light surface smut was removed with warm water and an acetone rinse. Several other etchants were explored,

15

including 2% Nital, Picral (2-4pct picric acid in ethanol) and Vilellas (1 pct picric acid and 5 pct concentrated hydrochloric acid in ethanol), each producing unsatisfactory results.

### 4.3 Image Acquisition and Optical Microscopy

After the samples had been prepared, images were taken using an optical microscope. A Nikon Epiphot metallograph with attached Nikon Digital Sight DS-U1 image acquisition system was used to take optical photos of the microstructure of every sample. Pictures were taken using the 50X and 100X lenses. A computer attached to the Nikon Epiphot was used to capture images using Nikon ACT-2U image acquisition software. The software allowed for contrast and brightness adjustment along with a user created scale to be placed on the picture for later analysis. Figure 5 displays a typical micrograph.



Figure 5: Photo Micrograph of Sample 5

# **5** Experimental Results

The following graphs show heat rate of immersion of sample 3 and the overall temperature



time curve of sample 1.

Figure 6: Temperature time curve for the first 15minutes after immersion



Figure 7: Temperature Time Curves. Held at 850 C for 1hr. Quenched in water.

#### 5.1 Measurement and Analysis

The next step was to measure grain size. ASTM E112, outlines procedures for Standard Test Methods for Determining Average Grain Size. These are standard procedures that include microstructure measurements and estimation by comparison to templates and overlays.

#### 5.1.1 Mean Grain Diameter

Four images of each sample's microstructure were printed and analyzed. Each image was taken at a different location on the sample so that the mean grain diameter could be evaluated for the entire specimen. On each image 5 lines were drawn, at a length of 10cm and the number of grains intersected was counted according to the Heyn Method, as shown in Figure 6. The calculation for the linear intercept is seen in equation 4-1

$$\overline{L}_{L} = \frac{1}{\overline{N}_{L}} = \frac{L_{T}}{PM}$$
(4.1)

where  $N_L$  is the number of intercepts per total length of the test lines  $L_T$ , P is the total number of grain boundary intersections and M is the magnification.



Figure 8: Heyns Linear Intercept Method

Table 4:	Sample	Intercept	Calculations
----------	--------	-----------	--------------

Intercepts	6
Line Length	97.5
(mm)	
Grains/mm	.03
Magnification	50x
Constant	.3125
Grain Diameter	.0201
(mm)	

Individual grains were also analyzed. First a line was drawn across the longest dimension of the grain and another line perpendicular to the first. These two values were measured, and then averaged, giving an approximate average diameter relative to the scale. All of the visible grains

in a particular sample were averaged to obtain a mean diameter for the sample. Using the scale in Figure 7, an equivalent value for ASTM grain size could be found.



Figure 9: Equivalent ASTM grain-size number

### 5.1.2 Average Grain Diameter

Table 5: Average Grain Diameter 4140

Sample	Grain Size	Equivalent
	(mm)	ASTM Number
1	0.0150	8.8
2	0.0172	8.5
3	0.0170	8.5
4	0.0203	8.0
5	0.0213	7.8
6	0.0249	7.4

The data in table 6 corresponds with what is expected of heat treated steel. The samples showed grain growth as time and temperature increased. The experimental data from all samples can be found in appendix A.

### 5.1.3 Grain Growth

 Table 6: Experimental Data

Sample	Austentizing	Hold Time	Grain Size	
	Temperature (C)	(min)	(mm)	
1	850	60	0.0150	
2	900	60	0.0172	
3	850	120	0.0170	
4	900	120	0.0203	
5	850	180	0.0213	
6	900	180	0.0249	

$$d^2 - d_0^2 = kt (5.1)$$

Based on the grain growth equation (5-1), diameter squared was plot vs. time. After interpolating and obtaining a linear trend line in MS Excel, two diffusivity growth constants were found.  $k_{900} = 2.682E-06 \text{ (mm}^2/\text{min})$  for the samples heated at 900C and  $k_{850}= 1.902E-06 \text{ (mm}^2/\text{min})$  for those treated at 850C.



Figure 10: Grain Diameter vs. Time

The grain growth kinetics were also simulated using a Beck type model. [14]

$$d^2 - d_o^2 = k_o \cdot \exp\left[\left(-\frac{Q}{RT}\right)t\right]$$
(5.2)

Where *d* is the mean grain diameter after hold at temperature *T*,  $d_0$  is the initial grain diameter,  $k_0$  is the pre-exponential rate constant, *R* is the gas constant, *Q* is the overall activation energy for grain growth, and *t* is the holding time. The experimental data was plot (Figure 11) and the

coefficient  $k_0$  and  $\exp\left(-\frac{Q}{RT}\right)$  for each hold temperature T was derived from an exponential curve.

According to Karabelchtchikova et. al. [14] maximum grain size limit before the negative effects of fatigue require rework is 55 $\mu$ m. Using equation (5-2) calculations were made to determine the maximum heating time *t* before the deterioration of the material properties, namely fatigue. The maximum heating time was calculated to be 510min at 850°C and 442min at 900°C.



Figure 11

# 6 Discussion

Initially the oxidation method was considered for revealing prior austenite grain size. This proved unsuccessful because the oxide layer was not clear. Several etchants were used to reveal the grain boundaries however only Beaujard and Bechets etch gave favorable results. However there was still a lack of clearly visible prior austenite grain boundaries in the many of the test samples. Once micrographs were taken this posed problems in measuring grain size. In addition, the linear intercept method is intended for single phase microstructure so manual calculations were used to accommodate.

# 7 Conclusion

Grain growth during heat treatment is one of the factors that cause fatigue failure. Therefore it is important to know the relationship between grain growth and different heat treating variables. This project looked at austentizing temperature and hold times and proved that there is indeed a linear relationship between grain diameter squared and hold time. It was experimentally determined that grain growth kinetics were faster for samples with a higher austenizing temperature. It would be beneficial to further investigate fatigue and fracture crack in relation to grain size. With that relationship one could optimize heat treatment variables to reduce grain size thus improving fatigue strength.

# 8 References

- (1) Philips, T. V. and McCaffrey, T. J., Ultrahigh-strength steels. *Metals Handbook*, Vol. 1, 10th edn. ASM, 1990, pp. 430–448.
- (2) "A Guide to Mechanical Properties." (2005). <http://www.mid-atlanticcasting.com/caststeel-guide\_FEB05.pdf>.
- C. Garcı'a de Andre's, M.J. Bartolome', C. Capdevila, D. San Martı'n,
   F.G. Caballero, V. Lo'pez, "Metallographic Techniques for the Determination of The." <u>Material Characterization</u> 46 (2001).
- Schreiman, Richard A., Bolton, Wendy 1. "Estimation of Prior-Austenite Grain Size in Heat Treated Martensitic Carbon and Low Alloy Steels"
   Buehler Ltd. Research and Development Laboratory, Lake Bluff, IL 60044
   Gulf Coast Laboratory Services, Houston, TX 77429
- (5) "Yield Strength and Heat Treatment" <http://tppinfo.com/defect\_analysis/yield\_strength.html>
- Meier, Mike "Hall Petch Relationship." Department of Chemical Engineering and Materials Science
   University of California, Davis September 13, 2004
- Meier, Mike Heat Treatment of Steel, Department of Chemical Engineering and Materials Science
   University of California, Davis September 13, 2004
- (8) Effect of metallurgical variables on fatigue <www.key-to-steel.com/articles/art137.htm>
- (9) ASM Metals Handbook, Properties and Selection: Nonferrous Alloys and Pure Metals, 9<sup>th</sup> edition, ASM International, Metals Park, Ohio, vol. 2, (1986).
- (10) Akhmad A. Korda a, Y. Miyashita a, Y. Mutoh a, T. Sadasue b "Fatigue crack growth behavior in ferritic–pearlitic steels with networked and distributed pearlite structures"
- (11) Sinha, Anil Kumar, Physical Metallurgy Handbook. McGraw-Hill, 2003.
- (12) V. V. Bukhanovskii, t V. A. Borisenko, t and V. K. Kharchenko
   "Effect of Heat Treatment and Welding on the Fatigue Resistance of Molybdenum Alloys of the Mo- Zr-B and Mo- AI- B Systems" <u>Metal Science and Heat Treatment</u> 36 (1997)
- (13) Properties of Alloy Steel AISI 4140 <http://www.efunda.com/materials/alloys/alloy\_steels/show\_alloy.cfm?ID=AISI\_4140& prop=all&Page\_Title=AISI%204140>

 (14) Karabelchtchikova, Sisson, Hsiang, "Multi-Objective Optimisation of Gas Carburising Process in Batch Furnaces with Endothermic Carburising Atmosphere" Accepted for publication in Surface Engineering, 2008

# 9 Appendix



# 9.1 Appendix A: Experimental Data

<u>Temperature</u>	<u>Time</u>	<u>Average Grain</u>				
<u>(c)</u>	<u>(min)</u>	<u>Diameter (mm)</u>	<u>D1</u>	<u>D2</u>	<u>D3</u>	<u>D4</u>
850	60	0.0150	0.0132	0.0140	0.0170	0.0156
900	60	0.0172	0.0162	0.0155	0.0190	0.0182
850	120	0.0170	0.0175	0.0170	0.0175	0.0161
900	120	0.0203	0.0205	0.0195	0.0200	0.0210
850	180	0.0213	0.0215	0.0218	0.0203	0.0215
900	180	0.0249	0.0260	0.0249	0.0241	0.0245
		<u>Diameter</u>				
		Squared(mm <sup>2</sup> )	$D1^2$	$D2^2$	$\underline{D3}^2$	$\underline{D4^2}$
850	60	0.00022	0.00017	0.00020	0.00029	0.00024
900	60	0.00030	0.00026	0.00024	0.00036	0.00033
850	120	0.00029	0.00031	0.00029	0.00031	0.00026
900	120	0.00041	0.00042	0.00038	0.00040	0.00044
850	180	0.00045	0.00046	0.00048	0.00041	0.00046
900	180	0.00062	0.00068	0.00062	0.00058	0.00060

# 9.2 Appendix B: Micrographs



Micrograph 1-1



Micrograph 2-2



Micrograph 3-3



Micrograph 4-4



Micrograph 5-1



Micrograph 6-2



Micrograph 7-3



Micrograph 8-4



Micrograph 9-1



Micrograph 10-2



Micrograph 11-3



Micrograph 12-4



Micrograph 13-1

Microstructure of 4140, austenized at 900°C for 2hours then water quenched. Etched using Bechet-Beaujard reagent



Micrograph 14-2

Microstructure of 4140, austenized at 900°C for 2hours then water quenched. Etched using Bechet-Beaujard reagent



Micrograph 15-3

Microstructure of 4140, austenized at 900°C for 2hours then water quenched. Etched using Bechet-Beaujard reagent



Micrograph 16-4

Microstructure of 4140, austenized at 900°C for 2hours then water quenched. Etched using Bechet-Beaujard reagent



Micrograph 17-1



Micrograph 18-2



Micrograph 19-3



Micrograph 20-4



Micrograph 21-1



Micrograph 22-2



Micrograph 23-3