ME 283 Manufacturing Process Control
Design of Experiments (DOE) Project
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EFFECTS OF HEAT TREATMENT FACTORS ON HARDNESS

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ABSTRACT

Material hardness is a measure of a material's resistance to localized plastic deformation caused by indentation or abrasion. It can be measured on macroscopic and microscopic levels and varies due to molecular bond structure. Typical means of increasing or decreasing material hardness are cold-working and heat treating respectively. This project explored the effects of heat treatment factors on 24 samples of cold-worked low-carbon steel. Three factors were considered, heat treatment temperature, duration of heat treatment, and quenching temperature. Each factor was varied between two set levels, referred to as high and low, to form a 2^3 factorial design. Heat treatment temperature was set at either 400 °C or 600 °C. Duration of heat treatment was either 10 or 20 minutes. Quenching temperature was either 0 °C or 22 °C (room temperature). Interactions between factors were also analyzed. Results indicated that heat treatment temperature and the combination of heat treatment temperature and duration were the most influential factors with heat treatment duration determined as the third most influential.

INTRODUCTION

Heat treatment is prevalent in the manufacturing realm as it is often used as a means to alter mechanical properties of a material in order to obtain a desired combination for the given application. This can be done by prompting phase transformations of the crystalline structure of metals which correlate to factors such as strength, hardness, ductility and toughness [1]. There are many different variations of heat treatment each with their own set of factors which influence the mechanical property output variable. Some of these factors include the temperature at which the material is treated, the length of time that the material undergoes temperature treatment, and how the material is cooled. These factors can further be broken down into more detail, such as the range of temperatures or fluctuations the material may observe during treatment, and how the material is cooled, be it fast or slow, the type of medium used, and even whether the medium is agitated.

Prior to the design of the group experimentation analyzed throughout this paper, some initial research was conducted to find similar studies. One such experiment observed the influence of heat treatment factors such as treatment temperature and time in order to determine the optimal combination of strength and toughness characteristics of HSLA-100 steel used for navy vessels. This study focused on the tradeoffs of the two output variables as typically an increase in material strength is accompanied by a decrease in toughness [4]. Another study with a focus on the cooling rate of heat treated specimens observed various combinations of preheating temperatures accompanied by a slew of quenching rates in order to observe a material toughness output [2].

From this background research the team decided to vary factors such as treatment temperature, treatment time, and quench temperature during the heat treatment process. These factors were selected as they are easily accessible and measurable. Though both research papers examined focused on material toughness as an output, the team decided to observe material hardness as the necessary measurement equipment was readily available. From this point, a factorial design study using 2 levels and 3 factors was constructed.

EXPERIMENTAL WORK

Factorial design studies are often used to determine the main effects and interaction effects of different factors throughout a process. This is ideal in the scope of manufacturing when determining the optimal input variations in order to achieve a desired output variable. By selecting a series of inputs, varying them slightly, and then measuring the difference in output it is possible to determine the level of significance a factor may have on the process. Furthermore, the factor can be compared against the other factors to see if one has an effect on another (typically called an interaction effect) and a regression model equation can be developed to predict outcomes for ranges and values not measured in the experiment itself. The alternative one factor at a time study lacks the ability to depict the interaction effects of various factors.

This experiment conducts a factorial design in order to observe the effects of various process input factors on the output of steel material hardness.

Factorial Design

The designed experiment consisted of analyzing three factors at two different levels. Three replicates of the experiment were selected in order to obtain a reasonable amount of degrees of freedom to account for error in experimentation. This meant that the 2^3 factorial design study would require a total of 24 specimens run at 8 different combinations of factor levels.

The 3 factors selected for the study were the heat treatment temperature, heat treatment duration, and the post heat-treatment quenching temperature. These factors were selected as they play a known role in the heat-treatment process, and are relatively easy to adjust as well as measure. Each factor has a low and high state as illustrated in the table below (figure 1), along with their units of measurement.

		Factors		
		A	B	C
		Heat Treatment Temperature (°C)	Heat Treatment Time (min)]	Quenching Temperature (°C)
Levels	Low	400	10	0
	High	600	20	23

Figure 1. Table documenting the 3 factors varied throughout the experiment along with the units of measurement.

The eight different combinations of factors are given in figure 2 with a -1 representing a low level and a 1 representing a high level.

Run		A	B	C	AB	AC	BC	ABC
1	-1	-1	-1	-1	1	1	1	-1
2	A	1	-1	-1	-1	-1	1	1
3	B	-1	1	-1	-1	1	-1	1
4	AB	1	1	-1	1	-1	-1	-1
5	C	-1	-1	1	1	-1	-1	1
6	AC	1	-1	1	-1	1	-1	-1
7	BC	-1	1	1	-1	-1	1	-1
8	ABC	1	1	1	1	1	1	1

Figure 2. Table documenting the structure of a 2^3 factorial design.

The selected output variable of the experimentation was material hardness as measured on the unitless HRC Rockwell scale. The Rockwell Hardness scale is often used to measure the resistance to indentation of the sample by applying a force loading to the sample and measuring the depth of penetration of the indenter.

Fabrication Process

Running a 2^3 factorial design study with 3 replicates meant the project required a total of 24 samples, with 8 unique specimen combinations (refer to figure 2). Prior to starting the fabrication process various materials were researched by the team. A low carbon steel bar with part number 8910K268 was ordered from McMaster-Carr as it was previously cold-worked and met the specifications of being heat treatable to a range of B70 to C60 on the Rockwell Hardness Scale. The steel bar measured a total of 0.25” x 0.5” x 2’ and was cut down into the 24 samples (see figure 3), each measuring 0.25” x 0.5” x 0.75”.

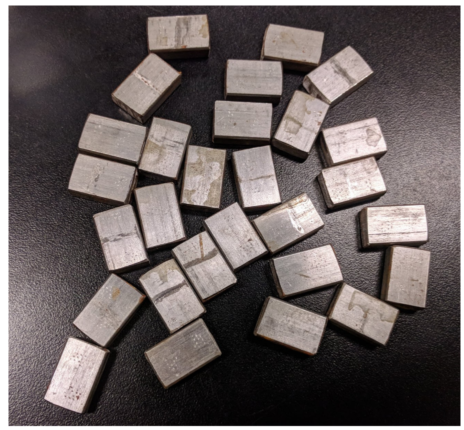

Figure 3. Steel specimens prior to heat treatment process.

From here the samples were split up into 4 runs of 6 samples. The first run was placed into a Thermo-Scientific furnace at a temperature of 400 °C for a total of 10 minutes. Once the 10 minutes had passed the team removed all 6 samples and placed them on bricks for a total of 5 minutes. During those 5 minutes the 6 samples were broken up into 2 subgroups of 3 samples one of which was placed in the ice bath quench (0 °C) while the other was placed in the room temperature quench (23 °C). Once the 5 minutes of preparation were complete the samples were placed in their respective quench container and left to sit for a total of 8 minutes. After quenching the samples were marked by a sharpie with their respective factor levels (see figure 14) indicating the treatment they received (for example LLL for low treatment temperature, low treatment time, and low quenching temperature) and then measured for hardness value.

This process was then repeated with another group of 6 samples at 400 °C for 20 minutes. Once all the samples for the low level of heat treatment temperature had completed the heat treatment process, the furnace temperature was increased to 600 °C and the process was repeated two more times, once for the 10 minute treatment, and another for the 20 minute treatment.

Measurement Technique

Once the heat treatment experimental process was completed the samples were organized by their respective 3 character nomenclature. This marking was the added after each sample was removed from the quench bath and was developed to denote the specific sample's heat treatment factor level combination. The nomenclature consists of a 3 letter combination either marked as an 'H' or 'L' indicating the high or low level of treatment. The first character corresponds to the temperature that the specimen was treated at, while the second and third characters correspond to the length of heat treatment time and the temperature of the quench respectively.

A series 500 Wilson Rockwell Hardness Tester equipped with a HRC 120 degree spheroconical tip (figure 5) applied a 150 kgf to obtain each of the dimensionless hardness values collected. Most hardness measurements are obtained using the "B" and "C" scales which overlap, were the "B" scale is commonly used for aluminum, brass, and softer steels, whereas the "C" scale is used for harder steels. Each sample was measured a total of 4 times at 4 separate points on the sample in a rectangular pattern as shown in figure 4. The 4 measurements were then averaged and this value was noted as the hardness value for the designated sample.

Figure 4. A sample post measurement process, showing the indentations from the Rockwell Hardness Tester. The 4 measurement points are organized in a rectangular fashion.

Figure 5. A team member carefully measures the hardness of a sample using the Series 500 Rockwell Hardness Tester on an HRC scale.

DATA ANALYSIS

Analyzing the data began with observing how each factor affected the output hardness. After these effects were determined, a more in depth analysis took place by calculating the statistical significance of each factor using analysis of variance (ANOVA). The levels of significance, also called p-values, obtained from the ANOVA were then compared with a chosen level of significance of $\alpha = 0.05$ (a 95% confidence interval). Values smaller than the chosen level of significance were deemed statistically significant and influential. Analysis for the experiment was conducted simultaneously using both Minitab and Excel in order to cross reference results and ensure correctness.

Factor Effects

Analyzing the data, the factors with the most effect on the output hardness were A (furnace temperature) and the interaction effect AB with main effects of -3.68 and -1.12. As interaction effect AB was observed to have a large effect on the output hardness, factor B was deemed important as well despite its main effect being similar in value to factor C. Main effects are shown in figure 6 and in the Appendix.

The fact that the main effects are negative just means that particular factor is inversely related to the output. In this case, an increase in furnace temperature (factor A) decreases the output hardness of the steel. Increasing heat treatment time also decreases steel hardness.

	A	B	C	AB	AC	BC	ABC
One Way Effect	-1.84	-0.30	-0.21	-0.56	-0.01	-0.03	0.10
Two Way Effect	-3.68	-0.60	-0.42	-1.12	-0.01	-0.06	0.20
Grand Average	21.31	21.31	21.31	21.31	21.31	21.31	21.31
Average Low	23.15	21.61	21.52	21.87	21.31	21.34	21.21
Average High	19.47	21.01	21.10	20.75	21.30	21.28	21.41

Figure 6. Calculated Effects (main effect being two way effect).

Figure 7 provides a visual of the relation between the main effect and hardness for factor A.

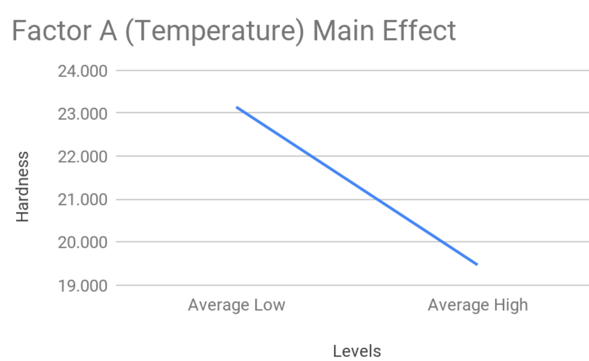

Figure 7. Main Effect of Temperature on Output Hardness

The plot in figure 8 shows the interaction effects between each pair of factors. If the two lines have the same slope, there is little to no interaction effect. If the two lines have noticeably dissimilar slopes, the two factors interact; one factor change affects the other. In this case, the AB plot shows signs of a significant interaction effect while the AC and BC plots do not. This Minitab analysis agrees with the excel calculated effects shown in figure 6.

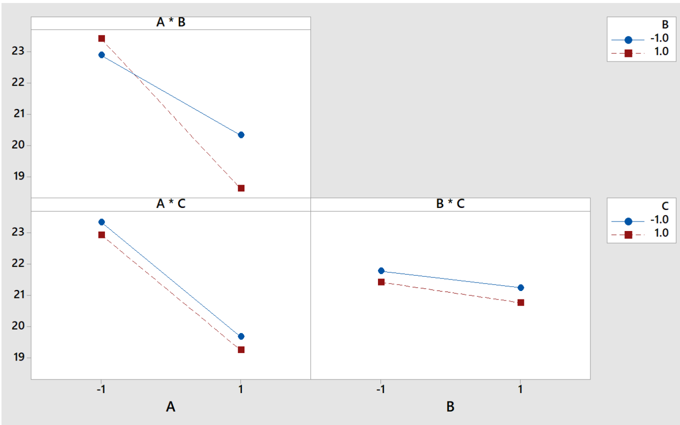

Figure 8. Main Effect of Temperature on Output Hardness

Analysis of Variance

While looking at the factor effects gave a general idea of which factors influence the output hardness, a more in depth analysis was performed using ANOVA (figure 9). Each combination of factor changes was totaled and normalized by dividing by the total number of samples to find the sum of squares. This was then divided by the degree of freedom for each factor to find the mean squares. An f value was then determined by dividing the mean square of each factor by the mean square error. The p-value (level of significance) of each factor was determined using this f value adnd the excel function f.dist.rt(). Comparing this value to a preset level of significance $\alpha = 0.05$, factors A, B, C, and AB were deemed statistically significant. This outcome agrees with the calculated main effect and interaction effect values (figure 6).

Anova						
Factors	**Contrast**	**SS**	**D.O.F**	**MS**	**F**	**P**
A	-44.15	81.22	1	81.22	553.167	0.0000000
B	-7.15	2.13	1	2.13	14.508	0.0015434
C	-5.00	1.04	1	1.04	7.095	0.0169913
AB	-13.4	7.48	1	7.48	50.957	0.0000024
AC	-0.15	0.00	1	0.00	0.006	0.9373016
BC	-0.75	0.02	1	0.02	0.160	0.6947792
ABC	2.40	0.24	1	0.24	1.635	0.2193001
Error		2.35	16	0.15		
Total		94.48	23			

Figure 9. ANOVA Table.

Regression Modeling

A linear regression model was then created using the data obtained from the main effects calculations and ANOVA results. Since factors A (furnace temperature) and B (heat treatment time) along with the interaction effect AB were determined statistically significant, they were included in the regression model. Factor C (quenching temperature) was not included at this time despite being deemed statistically significant. This was decided because the p-value for factor C was an order of magnitude larger than the p-value for factor B, and factor B was mainly included due to the large effect the interaction AB has on the output. With this in mind, the regression equation (Eq 1) was generated where A and B are the coded inputs with values ranging from -1 (low level) to 1 (high level). It is then plotted in figure 10.

$$y = 21.31 - 1.83A - 0.30B - 0.56AB \qquad (1)$$

As seen in the 3D response surface plot in figure 10, the hardness value of the sample decreases as the treatment time and temperature are increased. This model predicts that the lowest output hardness would be obtained by setting the furnace temperature (factor A) and heat treatment time to their respective high settings (600 °C and 20 minutes respectively for this experimental setup).

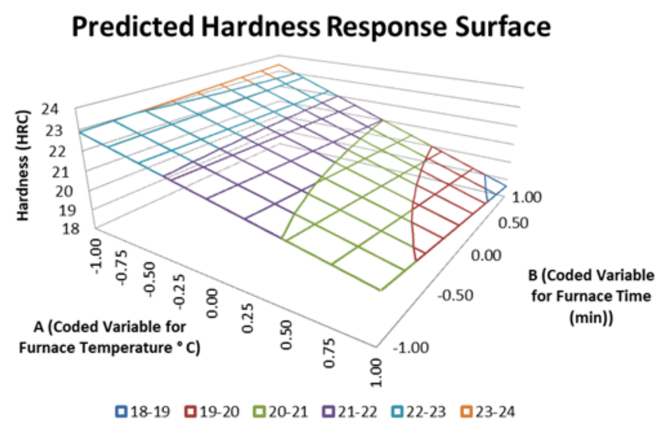

Figure 10. Regression Model generated response surface.

This conclusion agrees with the basic underlying principles of annealing a previously cold-worked sample such as the steel material used throughout this experiment. Having previously been cold-worked the crystal structure of the material would have undergone phases of recovery, recrystallization, and potentially grain growth to resemble the previous state prior to cold-working modifications. This process occurs as the temperature of the material is increased, as seen in figure 11 on the following page.

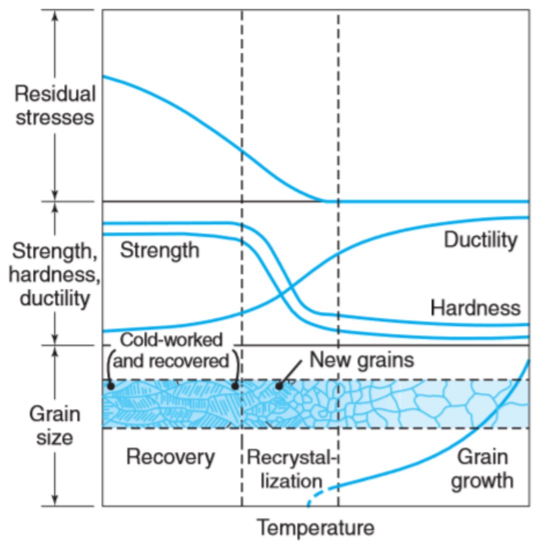

Figure 11. Annealing process diagram from *Manufacturing Engineering Technology* pg. 49 [3]

Knowing that the results of the experiment agree with literature and the principles of materials, helps verify that the process was completed correctly, and that the factors selected maintain a significant impact on material properties such as hardness.

Residual Analysis

Following experimental analysis, residuals are examined in order to determine the appropriateness of the regression model. A random order with no indication of predictable pattern represents a good fit for the data. The residuals for the regression model in equation 1 are shown in figure 12. The horizontal line represents zero residuals. When fitted points disperse evenly above and below zero residual lines, it indicates the linear regression model is appropriate for this case. In the case of this model, the residuals are random with no trend, although there are some residuals spaced out more than others. This may be due to having left out factor C in the regression model despite it having a similar effect to factor B.

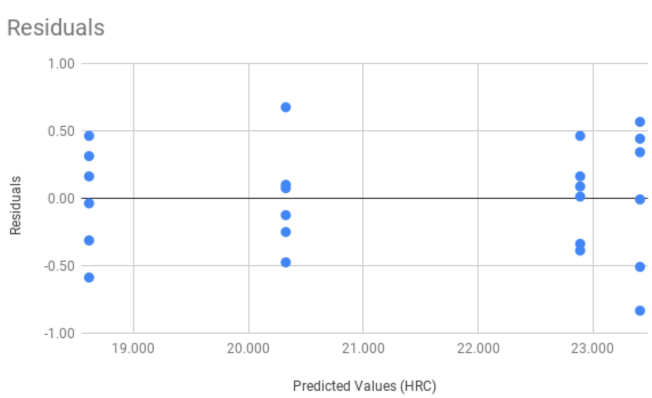

Figure 12. Residual Plot

A normal probability plot was also generated using the residuals in order to observe the linearity of the data points. A good fit of the model would cause the points to fall along a relatively smooth line. Figure 13 shows this plot. Though most of the points seem to be close or on the line, there does seem to be some deviation near the -0.2 residual value. This could be due to the same reason the residual plot in figure 12 shows spacing variations: leaving out factor C from the regression model.

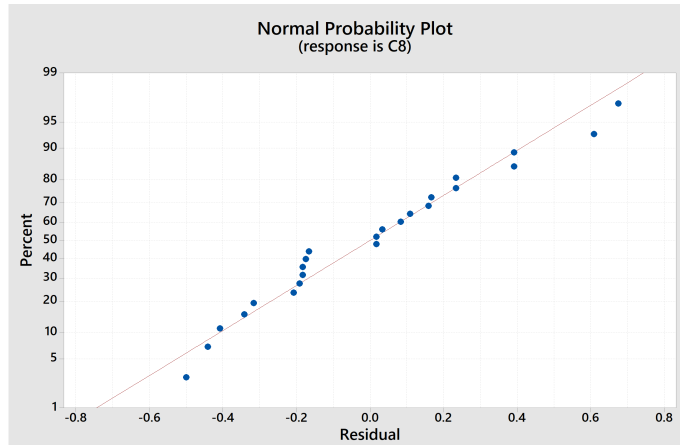

Figure 13. Normal Probability Residual Plot

Sources of Error

Throughout the experiment, there were sources of error that could have affected the material hardness. Sources of error include variability in furnace temperature and repeatability in hardness measurement. Thermo scientific furnaces have one digital reading from one thermocouple within the furnace, which means that the temperature may vary throughout different locations inside the furnace. Another source of error was that any time the furnace was opened to load samples the temperature dropped significantly and would have to heat back up to the desired range. With regards to the hardness measuring process using the Rockwell Hardness Tester, due to the destructive nature of measurement (i.e. creating an indentation in the sample) the measurements could not be conducted in the same exact location. Some measurement values could have varied because of temperature differences throughout the sample during treatment and different rates of cooling throughout the sample itself leading to different microstructures. This is why the average of 4 measurements was used throughout the analysis portion of the experiment.

CONCLUSIONS

Factors A, B, C, and interaction effect AB had statistically significant effects on the output hardness of the steel. Of these, factor A and the interaction effect AB had the largest effects: -3.68 and -1.12 respectively. The p-values corresponding to these two effects are 7.7×10^{-14} for factor A and

2.3×10^{-6} for the AB interaction effect. From a physical standpoint, this makes sense since the higher the furnace temperature, the faster grain recovery and regrowth occur in low carbon steel. A longer heat treatment duration at that elevated temperature results in more widespread grain restructuring. Heat treatment time alone is not as significant since certain temperatures are needed in order for grain restructuring to even occur.

The regression model adequately depicts how heat treatment temperature and time affect the output hardness, confirmation of which is obtained from analyzing the residuals. The residuals, when plotted against the predicted values, are seemingly randomly distributed. One concerning aspect is the variation of spacing between residuals at different predicted values. Looking at the normal probability plot of the residuals also shows that there are some unaccounted for sources leading to non-normality. The concerning aspects in the residuals plots may be due to not including factor C in the regression model among other sources of error.

ACKNOWLEDGMENTS

The team would like to thank Dr. Chung and Neil Peters for their support and permission to access the E105 laboratory equipment used throughout this study. An additional thank-you is extended to Dr. Lee as the instructor of the ME283 course for teaching the analysis and experimental concepts demonstrated throughout this paper.

NOMENCLATURE

Symbol	Description	Units
A	Treatment Temperature	°C
B	Treatment Time	minutes
C	Quench Temperature	°C

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APPENDIX

			Hardness Mea
	1	2	3
LLL 1	23.20	23.20	23.50
LLL 2	23.00	21.60	23.50
LLL 3	22.60	22.30	22.80
LLH 1	23.00	22.50	23.90
LLH 2	21.00	22.80	23.00
LLH 3	23.00	23.40	23.50
LHL 1	23.60	23.50	24.50
LHL 2	24.00	24.40	23.60
LHL 3	22.20	23.60	24.50
LHH 1	23.50	24.00	24.00
LHH 2	20.90	23.50	22.30
LHH 3	21.50	23.10	24.50
HLL 1	19.90	19.50	21.20
HLL 2	20.80	21.30	21.50
HLL 3	20.20	19.30	20.20
HLH 1	18.90	21.10	20.00
HLH 2	19.80	19.10	21.00
HLH 3	19.20	19.60	20.70
HHL 1	18.70	19.10	19.20
HHL 2	18.10	18.50	19.40
HHL 3	18.10	18.90	18.40
HHH 1	17.30	18.70	18.30
HHH 2	19.40	18.50	19.60
HHH 3	18.70	17.60	17.50

Figure 14. Collected Raw Data

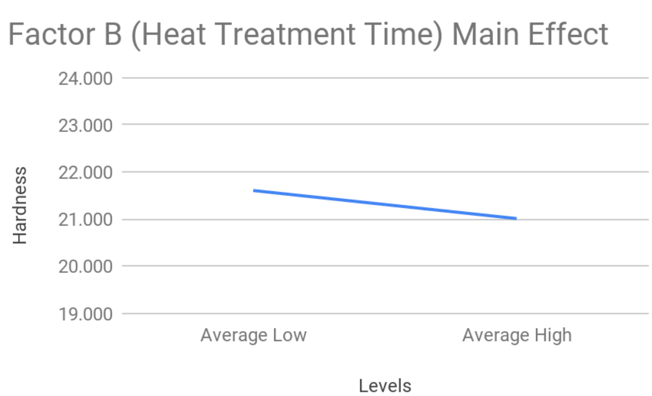

Figure 15. Main effect for Factor B

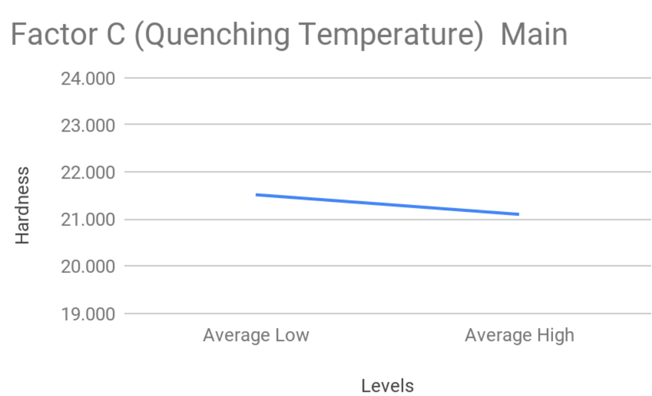

Figure 16. Main effect for Factor C