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RESEARCH ARTICLE

Mechanical changes and analysis of heat-treated 4140 steel with Taguchi method and ANOVA

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Abstract

In this work, the heat treatment process is applied to the 4140 steel to see how the samples show mechanical changes under various conditions. The samples have a height and diameter of 20 mm and 10 mm. The samples are heated up to 800 and 850 °C and then cooled in oil or oil and water mixture. The samples were annealed at 150 and 300 degrees for 1 and 3 h, respectively. After these stages, the microscope images of the samples are taken by optical and SEM microscopes. In addition to these, hardness tests are conducted on the specimens and results are analyzed by the Taguchi method with ANOVA. Because of these tests, the properties of the samples were fixed and discussed. The process variations affected the hardness values of the samples. The maximum hardness is obtained on the sample the hardened in the mixture of the oil and water. The DOE analysis confirmed the experimental results and showed the minimum error and maximum consistency.

1. Introduction

Steel is an alloy made up of the elements iron and carbon, which typically range in concentration from 0.02 to 2.1 percent [1–3]. The categorization of steel is actively influenced by how much carbon is present in the steel alloy. The steels mechanical and physical characteristics of change depend on how much carbon is contained in them. Steels are divided into three classes based on the proportion of carbon in each category; i) low carbon steels ii) medium carbon steels and iii) high carbon steel.

AISI 4140 steel is AISI 4140 is a medium carbon Cr–Mo steel used for applications requiring strength and impact toughness. It possesses great fatigue strength and wear resistance [4–8] as well as the ability to withstand temperatures up to 480°C, making it ideal for high-stress applications. In this article, the heat treatments are applied on the 4140 steel, we will see how the sample shows mechanical changes under what conditions and what differences it creates when heated to the same temperature and different cooling methods are used. Unalloyed and alloyed machine-building steels with a carbon content that are particularly suited for hardening, 4140 steels exhibit excellent toughness at a specific tensile strength at the conclusion of the curing process. 4140 steel, which has good induction qualities, may be summed up as an incredibly strong and high-quality material [9–11]. Due to a combination of these qualities, 4140 grade-tempered steel is currently a very sought-after product on the market. 4140 steel, which has good induction qualities, may be summed as an incredibly strong and high-quality material. Due to a combination of these qualities, 4140 grade-tempered steel is currently a very sought-after product on the market. These steels can be used individually as unalloyed, manganese alloyed, chromium, and chromium-molybdenum alloys based on their chemical compositions.

Since steels are used more in industries than other metals and alloys, heat treatment of steels has special importance [12]. Depending on the phase diagrams of metals and alloys, the desired mechanical properties and microstructures can be obtained with different processes applied at temperatures below the melting temperature. Heat treatment is mostly used to improve the metallic properties of metals. It comes to the forefront in terms of shape, especially with hardness and strength. It is also applied to make some changes in the properties of a metal part. 4140 tempered steel crankshaft, spline shaft, axle shaft and sleeve etc. used in various parts. Additionally, machinery parts and apparatus, agricultural vehicles and machinery, defense industry production, oil and gas industry are various usage areas of 4140 tempered steel [13], [14]. AISI 4140 steels must have a certain hardness value. Verma and Singh investigated the mechanical properties of AISI 1040 steel [15]. The authors noted that the highest hardness is obtained at the lowest temperature and the hardness reduced with increasing tempering temperature and holding time. Çaligulu et al. declared that the tempering temperature of the steels affects the martensitic transformation and the temperature decreases the hardness [16]. The media of the water and ash are compared and the highest hardness is obtained by water cooling [17]. The influence of double quenching and tempering with conventional quenching and tempering on the microstructure and mechanical properties of AISI 4140 steel was compared and it found that the specimens the double quenched and tempered indicated much higher mechanical properties than the specimens conventional quenched and tempered owing to finer austenite grain size, finer martensitic packets and a lower level of impurity concentration [18]. The low-cycle fatigue properties of AISI 4140 steel were significantly improved by the annealing treatment [19].

The study aims to investigate the influences of the homogenization and tempering temperatures and tempering time and the cooling rate on the hardness and microstructure of

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AISI 4140 steel and compare the results with the Taguchi [20], [21] method with ANOVA [22,23].

2. Materials and methods

In this article, the heat treatments are applied on the 4140 steel, we will see how the sample shows mechanical changes under what conditions and what differences it creates when heated to the same temperature and different cooling methods are used. The composition of the samples is given in Table 1. The heat-treated samples are 30 mm long and 10 mm in diameter, as shown Fig. 1.

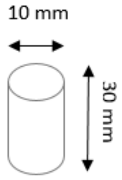


Figure 1. The schematic drawing of samples

As a heat treatment application, the samples are heated up to 800 and 850 °C and then cooled in oil and oil-water mixture (50 wt.% + 50 wt.%). Additionally, the samples were annealed at 150 and 300 degrees for 1 and 3 h at different temperatures and for different times. After these stages, the samples were sanded, polished and etched. For 50 seconds, the polished samples were etched in 96 ml HNO_3 – 4 ml HCl reagent. Then, the microstructures are studied with an optical microscope and SEM. The hardness test results were taken with ten reading averages and analyzed by the Taguchi method with ANOVA.

Table 1. Chemical composition of 4140 steel (wt%).

C	Si	Mn	P	S	Cr	Mo	Fe
0.45	0.35	0.75	0017	0.019	1.19	0.21	Bal

3. Results and discussion

In the Table 2, the experimental process and results are given. Heat-treated samples are in oil and the mixture of oil and water. The homogenous temperature, tempering temperature and time are changed and the hardness results are presented in Table 2. The samples displayed the different hardness values owing to various process parameters. The minimum and maximum hardness values of the heat-treated steels in oil are 153.57 HV and 568.77 HV, respectively, and 435.20 HV and 622.17 HV, respectively, for the heat-treated steels of the mixture of oil+water.

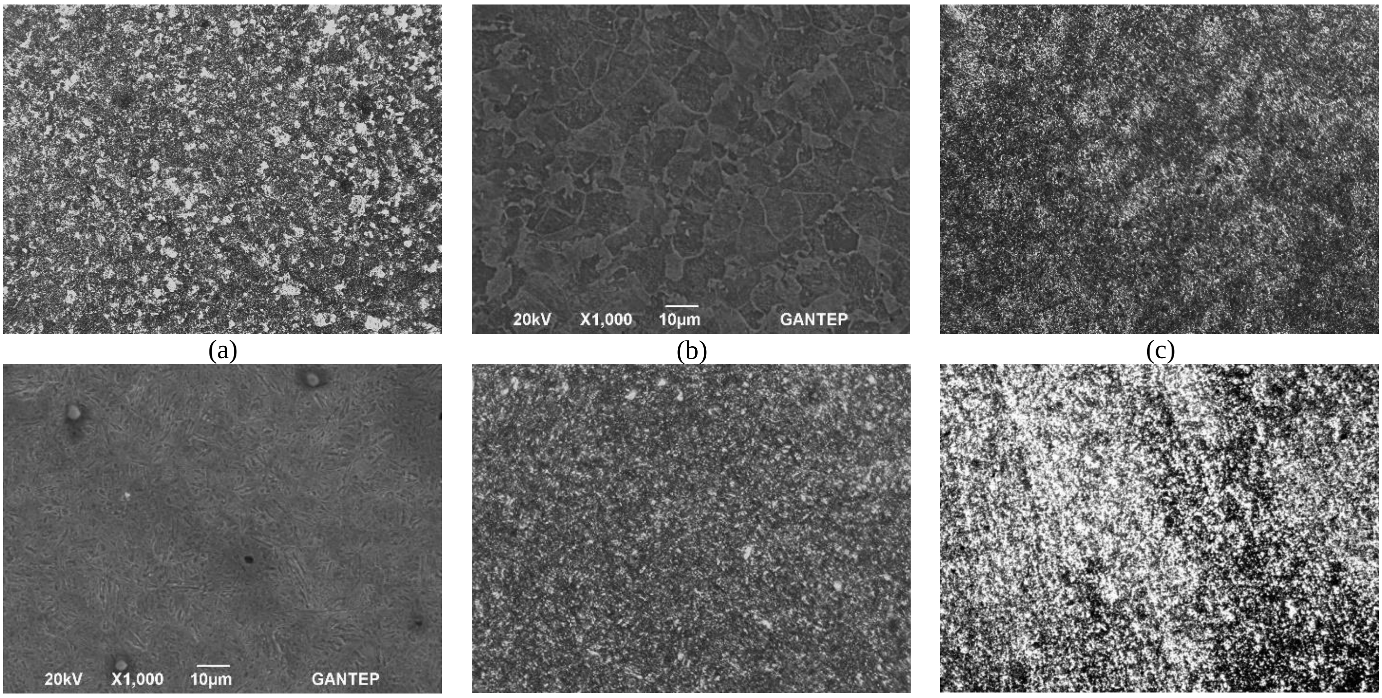
While the tempering temperature (150 °C) and tempering time (1 h) are constant for the oil hardened, the hardness increased when the homogenization temperature increased from 800 (°C) to 850 (°C). For the tempering temperature of 300 °C and tempering time of 3 h, the hardness decreased when the homogenization temperature increased from 800 (°C) to 850 (°C). In the mixture of oil and water, the hardness increased when the homogenization increased from 800 (°C) to 850 (°C) while the tempering temperature (150 °C) and tempering time (1 h) are constant. It is not seen an important change the hardness with the changing of the homogenization temperature from 800 (°C) to 850 (°C) for the tempering temperature of 300 °C and tempering time of 3 h. In oil quenching, the cooling rate in oil is slower than the cooling rate in water. As seen in this study, the reason for the obtained the higher hardness in the mixture of the oil and water compared to oil is the higher the cooling rate of the mixture of the oil and water [24–26].

In Fig. 2, a martensitic structure formed due to the cooling effect in the oil and mixture of the oil and water. The microstructures are taken and presented to the minimum and maximum hardness results. In the microstructures, the grain coarsening is observed in the microstructures with increasing temperature (Fig. 2a-b). The martensitic structure and plates in Fig. 2d form to a ferritic structure with the increasing of the tempering temperature and tempering time (Fig. 2a-b). For the mixture of oil and water hardened steels, the intense perlite structure is relocated with a ferritic structure, as seen in Fig. 2e-f. Before and after the tempering process, the temperature and time are carefully selected for temper embrittlement, which is generally seen in the temperature range of 400–600 °C [27].

Time-temperature-transformation (TTT) and continuous-cooling-transformation (CTT) diagrams are shown in Fig. 3. TTT diagrams are used to examine the multifaceted events that occur during the austenite transformation and primarily determine the properties of the transformation product. CTT diagrams show transformation events depending on temperature and time. With the help of the CCT diagram, the structures that will form in the steel are seen at the end of the preferred cooling rates. It can be very clearly seen that the structure will completely transform into martensite with very rapid cooling from the austenite phase temperature. The Ms temperature of 4140 steel is approximately 300–350 °C according to the continuous cooling transformation diagram.

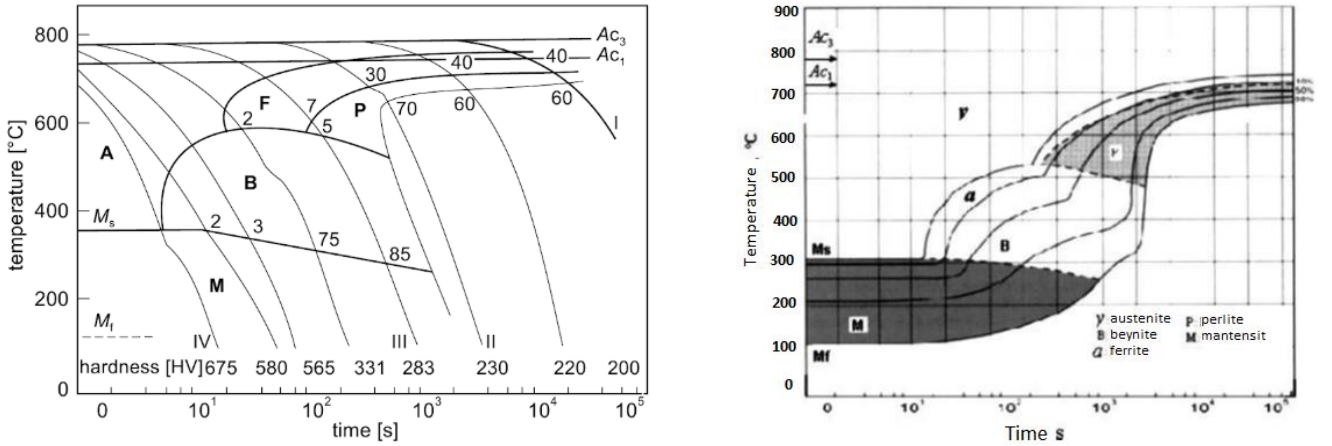
Table 2. Experimental Process and Results

Hardening Media	Homogenization Temperature (°C)	Tempering Temperature (°C)	Tempering Time (h)	Hardness (HV)	Code
Oil	800	150	1	479.20	A
	800	300	3	235.20	B
	850	150	1	568.77	C
	850	300	3	153.57	D
Oil wt.50% + Water wt.50%	800	150	1	603.90	E
	800	300	3	436.67	F
	850	150	1	622.17	G
	850	300	3	435.20	H



(a) (b) (c) (d) (e) (f)

Figure 2. a) Optical and b) SEM images of sample D, c) Optical, and d) SEM images of sample B, Optical images of e) sample G and f) sample H



(a) (b)

Figure 3. a) TTT and b) CTT diagrams of 4140 steel [28]

The authors declared that while the transformation from a martensitic structure to bainitic or ferritic structure is observed in steels depending on the tempering temperature, the hardness values also decrease depending on the tempering temperature [16]. Similar results are also reported in the related studies [29], [30]. The changes in the homogenization temperature, tempering temperature and tempering time affect the mechanical properties of the samples. Thus, a detail analysis of the results must be done. In this work, we studied the results with the experiment of design (DOE) with ANOVA for this aim.

4. Experiment of design (DOE)

The Taguchi method was used as the experimental design and analysis method. The main steps of this method are (1) determining the factors and interactions, (2) determining the levels of each factor, (3) choosing the appropriate orthogonal matrix, (4) transferring the factors and interactions to the columns of the orthogonal matrices, (5) performing the experiments, (6) analysis of data and determining of optimal levels, and (7) performing validation experiments [31]. The parameters and levels given in Table 2 are used to determine some parameters and their levels effective in adsorption with the Taguchi technique. For this purpose, Taguchi experimental design with 2 parameters and three levels, L4 (2^3) orthogonal array, is extracted using the Minitab program [32,33]. The characteristic of the system is its performance with Larger-the-Better using the Equal 1.

$$\frac{S}{N} = -10\ \log_{10}\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y^2}\right) \quad \text{Eq. 1}$$

Where:
y = Response value
y'= Mean of the response value
s = Standard deviation
n = Number of trails in given experiment

Table 3. The statistical results for hardened steel in the oil

Level	Homogenization Temperature (°C)	Tempering Temperature (°C)	Tempering Time (h)	Code	S/N ratio
1	50.52	54.35	48.67	A	53.6103
2	49.41	45.58	51.26	B	47.4287
Delta	1.11	8.78	2.60	C	55.0987
Rank	3	1	2	D	43.7261

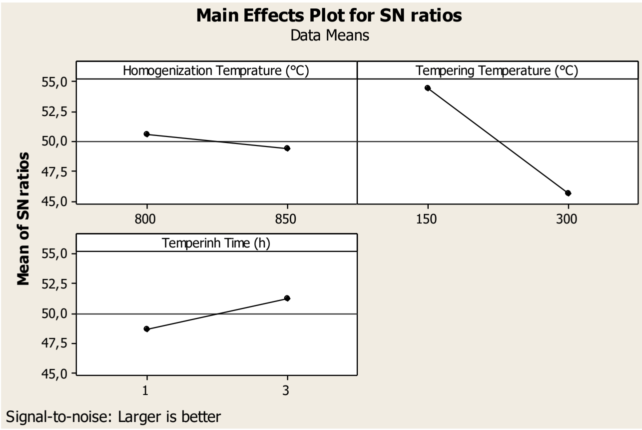


Figure 4. Mean Effects Plot for S/N ratios for hardened steel in the oil

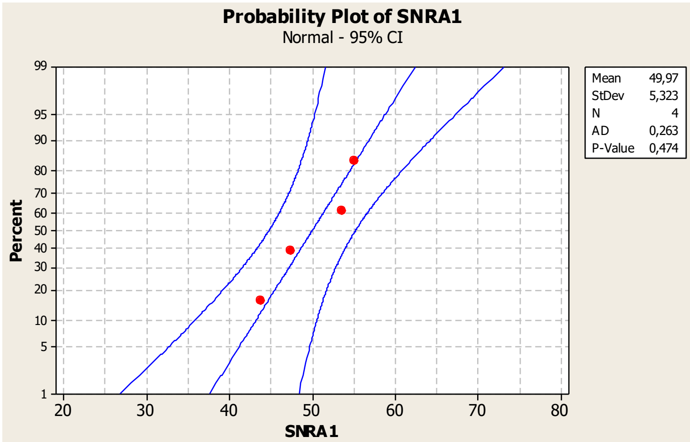


Figure 6. The probability plot for hardened steel in the oil

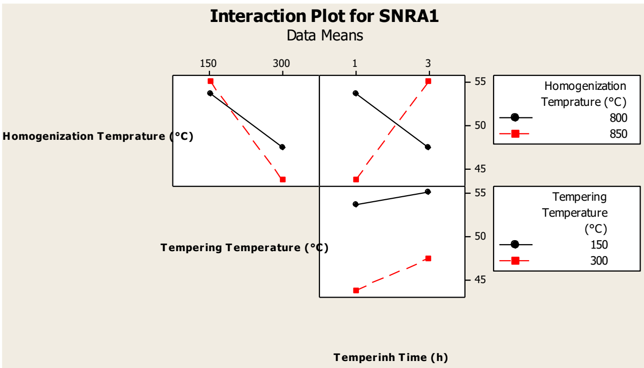


Figure 5. The interaction plot for hardened steel in the oil

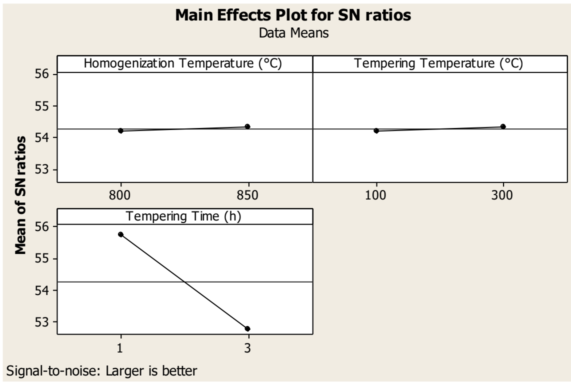


Figure 7. Mean Effects Plot for S/N ratios for hardened steel in the mixture of the oil and water

The statistical results are tabulated in Table 3. The signal (S) and noise (N) are the desirable and undesirable values. This ratio is widely used to measure the performance importance of a system. The Taguchi approach has two main advantages, i) to select the optimum level of design parameters and ii) to compare two experimental data. The highest S/N ratio value was obtained for sample C. Delta is the difference between the highest and lowest average response. Within the level 1 and 2, the highest and lowest different values are obtained at the homogenization and tempering temperatures. Rank was used to determine which factor had the largest effect. It can be seen that the tempering temperature is the most important parameter for this study. The smaller the rank for a factor has the higher the influence of the parameter on the process performance. The mean effects plot and interaction plot for S/N ratios are given in Fig. 4 and 5, respectively. It is clear from the Figure that the process chances have the different effects on the mechanical properties of the samples. The homogenization temperature affects less the hardness of samples than the tempering temperature and tempering time. In Figure 5, there is an inverse relationship between the homogenization temperature and tempering time, and a parallel relationship between the tempering temperature and tempering time. Any chance of these parameters would affect the other parameters and consequently the results.

In Fig. 6, the probability plot is display in the points are aligned along the line with a minimum deviation. We can see that the p-value is 0.474 and the standard deviation is 5.323, and therefore, the model is significant. To these results, the null hypothesis states that the data follow a normal distribution. Because the p-value is 0.474, which is greater than the significance level of 0.05.

The regression equation is obtained and presented for this works. The regression equation is

Hardness (HV) = 702 + 0.0794 Homogenization Temperature (°C) – 2.20 Tempering Temperature (°C) + 42.8 Tempering Time (h)

The R and R^2 values of the results are obtained as 0.96 and 0.93, respectively. It can be said that the strength of the specimens under a given condition can be predicted to about 93% by the Taguchi method.

The statistical results for hardened steel in the mixture of the oil and water are tabulated in Table 4. The highest S/N ratio value is obtained for sample G. Within the level 1 and 2, the highest and lowest different values are obtained at the tempering time and homogenization temperature. It can be seen that the tempering time is the most important parameter for this study.

The mean effects plot and interaction plot for S/N ratios are given in Fig. 7 and 8, respectively. The tempering time affects the hardness of samples more than the homogenization temperature and tempering temperature. In Figure 7, there is an inverse relationship between the homogenization temperature and tempering temperature, and a parallel relationship between the tempering temperature and tempering time, and between homogenization temperature and tempering time. Any chance of the parameters would affect the other parameters and consequently the results.

In Fig. 9, the probability plot for hardened steel in the mixture of the oil and water is displayed. The points are aligned along the line with a minimum deviation. We can see that the p-value is 0.079 and the standard deviation is 1.712, and therefore, the model is very significant. To these results, the null hypothesis states that the data follow a normal distribution. Because the p-value is 0.079, which is greater than the significance level of 0.05. Similarly, the tribological properties of AISI 4140 were optimized by the Taguchi method with ANOVA [34].

The regression equation for hardened steel in the mixture of the oil and water is obtained and presented for this study. The regression equation is

The hardness (HV) = 553 + 0.168 Homogenization Temperature (°C) + 0.0494 Tempering Temperature (°C) – 88.5 Tempering Time (h)

The R and R^2 values of the results are obtained as 0.99 and 0.99, respectively. It can be said that the hardness of the specimens under a given condition can be predicted to about 99% by the Taguchi method.

Table 4. The statistical results for hardened steel in the mixture of the oil and water

Level	Homogenization Temperature (°C)	Tempering Temperature (°C)	Tempering Time (h)	Code	S/N ratio
1	54.21	54.20	55.75	E	55.6193
2	54.33	54.34	52.79	F	52.8031
Delta	0.11	0.14	2.96	G	55.8782
Rank	3	2	1	H	52.7738

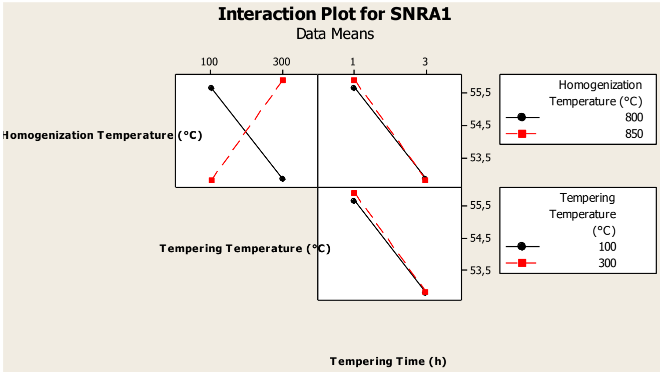


Figure 8. The interaction plot for hardened steel in the mixture of the oil and water

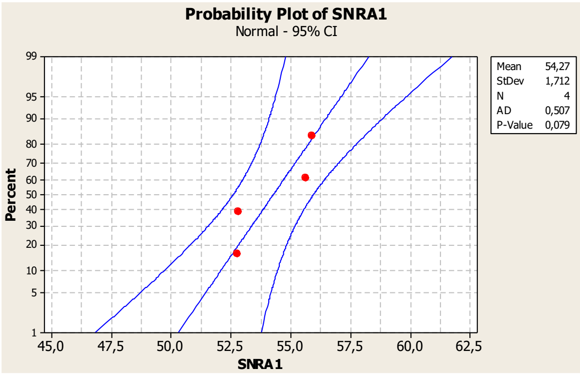


Figure 9. The probability plot for hardened steel in the mixture of the oil and water

5. Conclusion

In this study, the heat treatment process is applied to the AISI 4140 steel. The effects of the homogenization temperature, tempering temperature and tempering time on the hardness of the samples are studied. The following results can be drawn from this study.

- With increasing tempering temperature and time, the hardness values of the samples decreased. This situation is thought to be related to the phase transformation that occurs in the microstructure of AISI 4140 steel with the tempering process. The martensite structure, which has a hard structure at low tempering temperature, decreased with increasing tempering temperature and time, the grains became spherical and coursing.
- The heat treatment process is responsible for the microstructural changes leading to the declared hardness chance.
- Hardness properties of AISI 4140 are affected by the hardening media. The hardened steel in the mixture of the oil and water showed the higher hardness value than the hardened steel in the oil.
- In the DOE, the different results are obtained for each process. In the hardened steel in the mixture of the oil and water, the minimum standard deviation and p-value, and the maximum correlation and prediction ratio are observed.
- The regression equation has a high accuracy for the hardened steel in the mixture of the oil and water.

Author contributions

Halil Ibrahim Kurt: supervizor, discussion. Utku Berk Akyuz: experimental, writing. Engin Ergul: writing, control, editing.

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