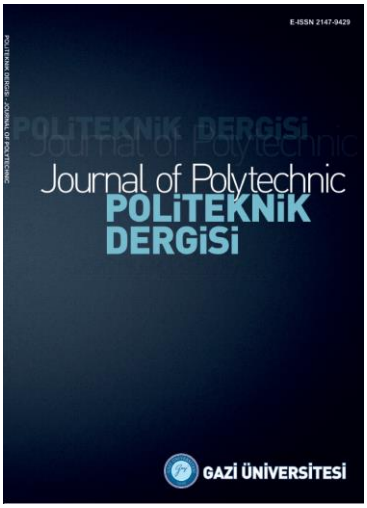


POLİTEKNİK DERGİSİ

JOURNAL of POLYTECHNIC

Gaz nitrasyon yapılan 4140 çeliğinin minimum kalıntı gerilim ve maksimum aşınma dayanımı için işlem parametrelerinin optimizasyonu

Optimization of processing parameters for minimum residual stress and maximum wear resistance during gas nitration of 4140 steel

***Yazar(lar) (Author(s))**: Sibel TÜZÜNER[1*], Ali Baran METİN[2], Ebru SARALOĞLU GÜLER[3], Tuğçe ŞAHİN[4]*

ORCID[1]: 0000-0002-6907-2198*

ORCID[2]: 0000-0001-9648-1774

ORCID[3]: 0000-0002-3732-1268

ORCID[4]: 0000-0002-6908-2310

Bu makaleye şu şekilde atıfta bulunabilirsiniz(To cite to this article): Tüzüner S., Metin A. B. and Saraloğlu Güler E., "Optimization of processing parameters for minimum residual stress and maximum wear resistance during gas nitration of 4140 steel", ***Journal of Polytechnic***, 25(2): 909-920, (2022).

Erişim linki (To link to this article): http://dergipark.org.tr/politeknik/archive

DOI: **10.2339/politeknik.993934**

Optimization of Processing Parameters for Minimum Residual Stress and Maximum Wear Resistance During Gas Nitration of 4140 Steel

Highlights

- *Nitration process using different processing parameters*
- *Measurement of wear resistance by pin on disc tribometer*
- *60% increase in wear resistance of 4140 steel*

Graphical Abstract

The optimized parameters of temperature, time and gas flow; rate resulted in minimum residual stress and maximum wear resistance during the nitration process (see Table 1 Sample 3).

Table 1. Nitration Parameters, Values and Measurement Results

Sample	Temperature [°C]	Time [h]	Flow Rate [m^3/h]	Mean Coefficient of Friction	Amount of Wear *(10^{-5}) [mm^3/n/m]	Hardness [HV0.5]	σ [MPa]
3	470	12	9	0.685	1.573	581.2	-267.6

Aim

The aim of this study is to increase the wear resistance together with minimum residual stress in the material by nitration of the threaded parts.

Design & Methodology

By determining the nitration parameters, the hardness and layer measurements of the samples were made and the microscope images were examined. Wear test was done using pin on disc tribometer and residual stress values were measured by XRD method.

Originality

In this study; different from the literature, the effect of three parameters on material properties was observed by studying three different values of these parameters.

Findings

As a result of the experiments and measurements, the sample that provides the "max. wear resistance and min. residual stress" values, which is the aim of the study, was determined as sample number 3. The threaded part was manufactured by using these test parameters.

Conclusion

As a result of all experiments, regression equations were created and it was determined that the most effective parameter is flow rate. After nitrocarburization, which is one of the surface treatments applied in HIDROMEK, the wear amount of the material was minimized by applying nitration with optimized parameters. Finally, the wear resistance is increased by 60%.

Declaration of Ethical Standards

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Optimization of Processing Parameters for Minimum Residual Stress and Maximum Wear Resistance During Gas Nitration of 4140 Steel

Araştırma Makalesi / Research Article

Sibel TÜZÜNER[1*], Ali Baran METİN[1], Ebru SARALOĞLU GÜLER[1], Tuğçe ŞAHİN

[1] Engineering Faculty, Department of Mechanical Engineering, Başkent University, Ankara, Turkey

(Geliş/Received : 10.09.2021 ; Kabul/Accepted : 12.02.2022 ; Erken Görünüm/Early View : 28.02.2022)

ABSTRACT

Several surface treatments has been applied to the 4140 specimens which are exposed to frictional conditions like gears in order to increase the wear resistance and hardness. Nitration is a common method to improve the hardness of the surfaces. However, the parameters must be chosen wisely. The parameters of temperature, time, flow rate during nitration of SAE 4140 steels were optimized in this study. The ranges for the parameters are selected as 470ºC - 520ºC - 570ºC, 7h - 9h - 12h and 6m^3/h - 7.5m^3/h - 9m^3/h for temperature, time and flow rate respectively. Residual stress, hardness and wear resistance were measured for the specific experiments together with microscopic observations. The nitration process of the threaded part was completed using the optimized parameters.

As a result of the study, it was observed that the maximum wear resistance and minimum residual stress values was obtained by the parameters of 470ºC, 12h, 9m^3/h.

Keywords: Gas Nitration, SAE 4140 steel, wear resistance, residual stress, microhardness.

Gaz Nitrasyon Yapılan 4140 Çeliğinin Minimum Kalıntı Gerilim ve Maksimum Aşınma Dayanımı İçin İşlem Parametrelerinin Optimizasyonu

ÖZ

Dişliler gibi sürtünme koşullarına maruz kalan 4140 çeliği numunesinin aşınma direncini ve sertliğini arttırmak için çeşitli yüzey işlemleri uygulanmıştır. Nitrasyon, yüzeylerin sertliğini iyileştirmek için yaygın bir yöntemdir. Ancak, parametreler dikkatli bir şekilde seçilmelidir. Bu çalışmada SAE 4140 çeliklerinin nitrasyonu sırasında sıcaklık, zaman, debi parametreleri optimize edilmiştir. Parametre aralıkları sıcaklık, zaman ve debi için sırasıyla 470ºC - 520ºC - 570ºC, 7sa – 9sa – 12sa and 6m^3/sa - 7.5m^3/sa - 9m^3/sa olarak seçilmiştir. Mikroskobik gözlemlerle birlikte spesifik deneyler için kalıntı gerilim, sertlik ve aşınma direnci ölçülmüştür. Optimize edilmiş parametreler kullanılarak dişli parçanın nitrasyon işlemi tamamlanmıştır.

Çalışma sonucunda maksimum aşınma direnci ve minimum kalıntı gerilim değerlerinin 470ºC, 12sa, 9m^3/sa parametreleri ile elde edildiği gözlemlenmiştir.

Anahtar Kelimeler: Gaz Nitrasyon, SAE 4140 çeliği, aşınma direnci, kalıntı gerilim, mikrosertlik

1. INTRODUCTION

Nowadays, many heat treatment techniques are used to improve mechanical properties such as high wear and corrosion resistance and fatigue resistance expected from machine elements [1]. Nitriding has been widely used in applications since hardening the surfaces of many metallic machine parts, especially steel is necessary in order to increase their wear and fatigue resistance [2].

The nitriding process, first developed in the early 1900s. It has been continued to play an important role in many industrial applications. Along with the derivative nitrocarburizing process, nitriding is frequently used in the industries of aircraft, bearings, automotive, textile, and turbine generation systems. The secret of the nitriding process is that it does not require a phase change from ferrite to austenite, or a change from austenite to martensite. In other words, the steel remains in the ferrite phase (or cementite, depending on alloy composition) during the whole procedure. Furthermore, no subsequent transformation from austenite to martensite occurs because only free cooling takes place [3]. This means that the molecular structure of the ferrite (body-centered cubic, or bcc, lattice) does not change its configuration or grow into the face-centered cubic (fcc) lattice that is characteristic phase of austenite, as does in case of carburizing. Comparatively low temperatures are used in this process since no quenching is required and this will end up with no volume changes, less distortion and low amount of deformation than either carburizing or conventional hardening. Some growth occurs as a result

*Sorumlu Yazar (Corresponding Author)
e-posta: sibeltuzuner06@gmail.com

of nitriding, but volumetric changes are relatively small [3,4].

Nitriding can be done in a nitrogen-containing gas, solid or liquid medium. Industrial, automotive or aerospace gears are often gas nitrided. Recently, computer-controlled gas nitriding has been used [5]. Gas nitriding is a case-hardening process whereby nitrogen is introduced into the surface of a solid ferrous alloy by holding the metal at a suitable temperature (below Ac1, for ferritic steels) in contact with a nitrogenous gas, usually ammonia [4]. Gears to be nitrided are placed in an airtight container or oven and an atmosphere of ammonia (NH_3) is supplied continuously while the temperature is raised and held between 470 and 570 °C (878 and 1058 °F) to produce the best combination of surface hardness and case depth. At this temperature, NH_3 breaks down into atomic nitrogen and hydrogen [5,6].

According to the following reaction:

$2NH_3 \rightarrow 2N + 3H_2$ [5]

Here, the aim is to increase the solubility of nitrogen in α-Fe and to increase the nitrogen content of other elements such as Fe, Al, Cr, Mo, etc., that may present in the composition of the steel and by increasing nitrogen partial pressure. So the formation of hard nitride/carbonitride layer is provided due to the nitrogen affinity of the elements. Depending on the diffusion conditions, these nitrides/carbonitrides can form either at grain boundaries or present as massive precipitates [2,5,7].

2.MATERIAL AND METHOD

2.1 Materials

4140 steel is a highly preferred material in the industry with its high toughness and favorable hardening performance [11]. SAE 4140 steel, whose chemical analysis is given in *Table 2*, was used in the study as the substrate. OBLF Emission Spectrometer analyzer was used for chemical analysis detection. It can be used to determine the chemical composition of materials from their spectrums with the spectral analysis method [12]. *Table 2* also includes the min and max weight amounts of the elements given for the SAE 4140 in SAE standard [13].

2.2 Heat Treatment

10 samples of rolled SAE 4140 with the diameters of 30 mm and lengths of 500 mm were prepared. The samples were preheated up to 500 °C. Then, austenitizing was applied to the specimens for 2 hours at 840°C in atmosphere-controlled furnaces. After that the samples are exposed to water and they are immersed in oil bath at a temperature of 60°C. Then the samples were cleaned in a vacuum washing machine using solvent chemicals to remove oil. They were left to cool at room temperature. Then the samples were tempered at 630°C for 1 hour. After tempering, the samples were cleaned again in vacuum washing machine and nitration process was applied.

Table 2. Spectral Analysis Results

Alloying Elements	%C	%Si	%Mn	%P	%S	%Cr	%Mo
Min Value	0.380	0.150	0.750	-	-	0.800	0.150
Measured Value	0.420	0.223	0.766	0.004	0.038	0.963	0.184
Max Value	0.430	0.350	1.000	0.035	0.040	1.100	0.250

Gas nitriding is the best method for homogeneous nitriding of complex shaped parts [8]. Studies have been carried out about gas nitration with different parameters in literature. Gas nitriding was applied to AISI 4140 steel at a temperature range of 480ºC and 590ºC and a nitration thickness of 12 µm – 0.75 mm was achieved [9]. AISI 253MA stainless steel samples were nitrided at 450°C for 8, 16 and 24 hours, and the highest hardness value and the best wear resistance were obtained in the nitrided sample when the nitration time is 24 hours. [10]. Therefore, the optimization is a critical issue to control the thickness of the nitration layer and so the hardness and the wear resistance of the nitride steel. In this study, the ranges for the parameters that are 470ºC - 520ºC - 570ºC, 7h - 9h - 12h and $6m^3/h$ - $7.5m^3/h$ - $9m^3/h$ for temperature, time and flow rate were covered, respectively. As a result, the values of residual stress, hardness and wear resistance were measured to compare.

2.3 Experimental Design

Generally, nitration processes are carried out at temperatures between 470-570 °C [5,6]. In the study, temperature values were selected to be as close as to the average of the minimum and maximum values of generally used temperatures. In addition, nitration processes usually applied for 40 hours and up. In this study, the time was chosen low together with a high flow rate in order to minimize the costs and save time. All of the parameters and their values are listed in *Table 3*. The experiment design is given in *Table 4*.

2.4 Nitration

The nitration process of 10 experiments was conducted according to experimental by nitration furnace in *Figure 1*.

Figure 1. IPSEN Nitration Furnace

IPSEN brand single chamber type VDR(N)-1714-E vacuum nitration furnace was used in the study. The furnace measures 900 mm x 1200 mm x 900 mm (W x L x H) and has a loading capacity of up to 1500 kg. The operating temperature of the oven is between 150°C - 750°C. VDR nitriding furnace works with full automation system.

2.5 Hardness

After the nitration process, surface hardness, White layer Hardness and Thickness, Hard Tissue Hardness and Depth and Core Hardness values are measured by "Mitutoyo Vickers Micro Hardness Device". A load of 20 grams was applied when measuring the hardness from the core region, and 500 grams was applied when measuring the hardness from the material surface. In both cases, the load application time was 30 seconds. Then, microscope images of each sample were taken by "Optical Microscope.

Table 3. Nitration Parameters and Their Values (Heating Rate = 14 C/min)

Nitration Parameters	Value 1	Value 2	Value 3
Temperature [°C]	470	520	570
Time [h]	7	9	12
Flow Rate [m^3/h]	6	7.5	9

Table 4. Total Experiment Design

Number of Experiments	Temperature [°C]	Time [h]	Flow Rate [m^3/h]
1	470	7	6
2	470	9	7.5
3	470	12	9
4	520	7	7.5
5	520	9	9
6	520	12	6
7	570	7	9
8	570	9	6
9	570	12	7.5
10	570	12	6

2.6 Wear Resistance

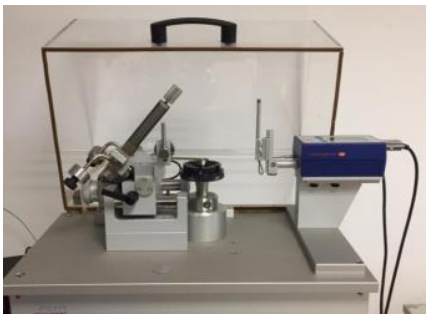


Figure 2. Tribometer Device

The wear resistance measurements were conducted for the maximum and minimum values of the previously determined parameters shown in *Table 5* and also fractional factorial design is used in order to keep the number of experiments low.

The number of experiments was reduced to "2^2 = 4" by Fractional Factorial Design listed in *Table 6*. Wear measurements of 4 samples given in *Table 6* were conducted by the pin-on-disc tribometer (*Figure 2).* Whose details are given in the *Table 8.* The wear test samples have a diameter of 28 mm and a height of 5 mm.

Table 5. Parameter Values for Wear Test

Nitration Parameters	Value 1	Value 2
Temperature [°C]	470	570
Time [h]	7	12
Flow Rate [m^3/h]	6	9

Table 6. Experimental Design for Wear Tests

Number of Experiments	Temperature [°C]	Time [h]	Flow Rate [m^3/h]
1	470	7	6
2	470	12	9
3	570	7	9
4	570	12	6

Table 7. Experimental Design for Wear Tests

Tribometer Parameter					
Tribometer Module 7 / Version 4.4 K		**Sample**		**Static Partner**	
Radius	7.50-10.00 [mm]	**Substrate**	SAE 4140	**Substrate**	100Cr6
Linear Speed	50.00 [mm/s]	**Cleaning**	Ultrasonic	**Cleaning**	Aseton
Normal Load	10.00 [N]	-	-	**Dimension**	6.00 [mm]
Stop Condition	150.06 [m]	-	-	**Geometry**	Ball
Effective Stop	Meters	-	-	-	-
Acquisition Rate	15.0 [hz]	-	-	-	-

2.7 Residual Stress

The residual stress values of two samples (3 and 10) with maximum wear resistance were measured by X-ray diffraction method with the XRD device given in *Figure 3*.

Figure 3. Residual Stress Measurement with XRD Method

Stresstech G2R X-Stress 3000 XRD device was used for residual stress measurement. Electropolishing device used for layer removal is LectroPOL-5. Depth measurements were taken with a Mitutoyo digital depth gauge. The range that can be measured for residual stress is determined as "200 - 250 micron" for these samples. In this value range determined by removing the layer with the electro-polishing method, measurements were taken from sample 10 at a depth of 212.5 microns and from sample 3 at a depth of 219.0 microns. This depth difference can be tolerated, as these depths are performed at specified intervals and in micron units.

In order to interpret the effect of different values of the parameters on the result, "Regression Analysis" was performed in the Minitab program. The equations created as a result of the analysis were examined through the main effect graphs.

3.RESULTS AND DISCUSSION

3.1 Hardness Measurements

Nitration processes were carried out according to *Table 4*. After the nitration process, the hardness (white layer, stiff tissue, core) values, white layer thickness and stiff tissue depth values were measured and summarized in *Table 8.*

Nitride depths were measured approximately 0.47 mm after the experiments. The maximum hardness value was obtained at 470 ˚C, and the maximum hardness depth was obtained at 570 ˚C. Similarly, nitride layers up to 0.5 mm was obtained after gas nitriding and the maximum hardness was obtained at 538 ˚C whereas the maximum

Table 8. Micro Hardness Values

Number of Experiments	**Temperature [°C]**	**Time [h]**	**Flow Rate [m³/h]**	**Surface Hardness [HV0.5]**	**White Layer Hardness [HV0.02]**	**White Layer Thickness [µm]**	**Stiff Tissue Depth [mm]**	**Core Hardness [HV0.5]**
1	470	7	6	607	1195.4	8.78	0.35	250
2	470	9	7.5	550	961.7	12.36	0.24	259
3	470	12	9	589	888	14.03	0.32	288
4	520	7	7.5	581.2	1069	15.85	0.27	290
5	520	9	9	545	1102.5	14.40	0.30	250
6	520	12	6	567.5	1137.5	12.50	0.29	276
7	570	7	9	469	781	22.65	0.47	250
8	570	9	6	491	943	18.40	0.23	335.5
9	570	12	7.5	546	1091	16.44	0.37	265
10	570	12	6	521	875.1	22.40	0.36	317

nitride depth was obtained at 650 ˚C. The results were compatible with the claim of hardness value decreased with increasing temperature and the depth [14].

In the study, when the 8th and 10th experiments, in which the temperature and flow values are constant, are examined as a result of the hardness measurements, the surface hardness and the hard tissue depth decrease as the time decreases. Similar to a study that has a result of hardness values and hardness depths decrease in short processing times and low temperatures, since diffusion becomes difficult [2]

3.2 Microstructure

After the hardness measurements, the microscope images of the samples were taken that show the layers formed after nitration in *Figures 4 - 13*.

The formation of nitrided zones begins from a series of nucleated growth sites on the steel surface. These nucleation growth areas are referred to as the "white layer" [15]. The white layer, the outermost nitride layer, name with nital (3-5% HNO_3 + alcohol) appear white after etching because of the area. White layer hard, brittle and is resistant to wear. In this layer, γ' (Fe_4N) and ε (Fe_2N and Fe_3N) phases or a mixture thereof. γ' (Fe_4N) has a face centered cubic (Fe_4N) structure. while ε (Fe_2-3N) has a hexagonal structure. Also, the white layer mechanical properties, the presence of these phases depending on the amount and the thickness of the layer largely depends. In the white layer, the γ' (Fe4N) phase formation, due to its soft and ductile nature, low wear resistance, impact resistant. While it is preferred in applications, ε (Fe2-3N) phase formation, high wear resistance it is preferred in the parts that are desired to be [16]. The region just below the white layer is called the "diffusion zone". This region consists of stable nitrides formed by the reaction of nitrogen with nitride-forming elements. The region below the diffusion zone is the core of the steel, usually composed of tempered martensite [15]. The core region is below the diffusion region and tempered martensite is observed in this region. Microscope images of the samples were made with device “OLYMPUS GX41” and photos were taken with magnification “x50 & x100”.

1.Experiment

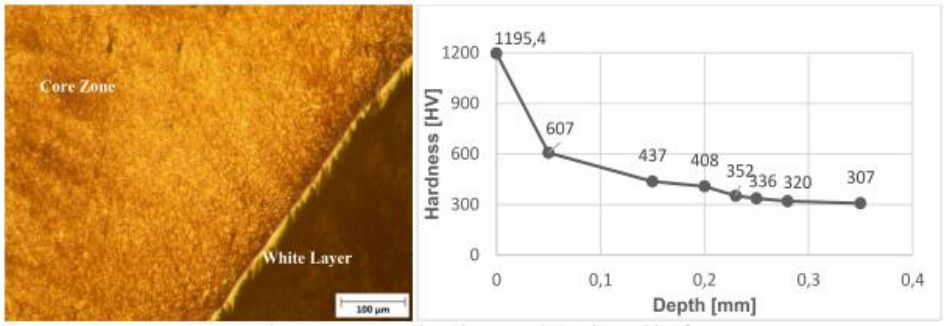


Figure 4. Microscope Image and Hardness-Depth Graph Resulting from Exp-1

Micro Hardness Values;

- **White Layer Hardness:** 1195.4/0.02/30/HV
- **White Layer Thickness:** 8.78 µm
- **Surface Hardness:** 607/0.5/30/HV
- **Stiff Tissue Hardness:** 307/0.5/30/HV
- **Stiff Tissue Depth:** 0.35 mm
- **Core Hardness:** 250/0.5/30/HV

2.Experiment

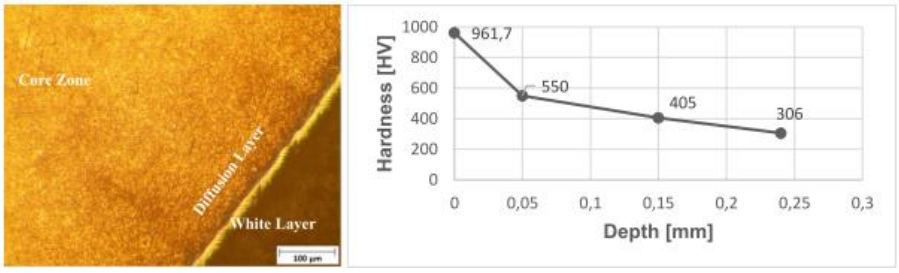


Figure 5. Microscope Image and Hardness-Depth Graph Resulting from Exp-2

Micro Hardness Values;

- **White Layer Thickness:** 12.36 µm
- **White Layer Hardness:** 961.7/0.02/30/HV
- **Surface Hardness:** 550/0.5/30/HV
- **Stiff Tissue Hardness:** 306/0.5/30/HV
- **Stiff Tissue Depth:** 0.24 mm
- **Core Hardness:** 259/0.5/30/HV

3. Experiment

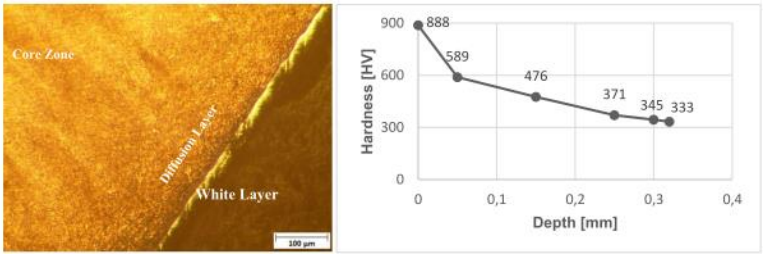


Figure 6. Microscope Image and Hardness-Depth Graph Resulting from Exp-3

Micro Hardness Values;

- **White Layer Thickness:** 14.03 µm
- **White Layer Hardness:** 888/0.02/30/HV
- **Surface Hardness:** 589/0.5/30/HV
- **Stiff Tissue Hardness:** 333/0.5/30/HV
- **Stiff Tissue Depth:** 0.32 mm
- **Core Hardness:** 288/0.5/30/HV

4. Experiment

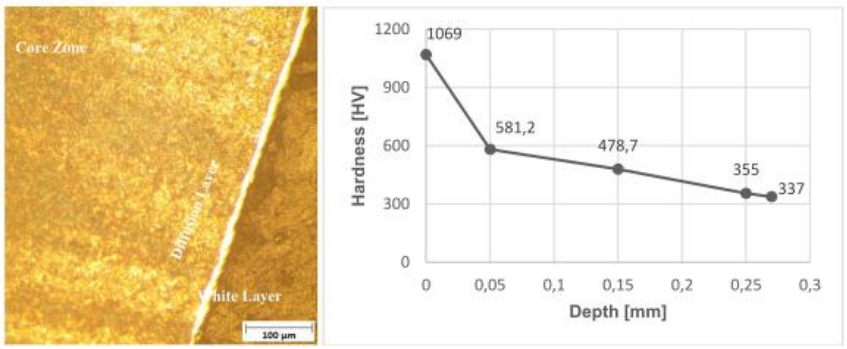


Figure 7. Microscope Image and Hardness-Depth Graph Resulting from Exp-4

Micro Hardness Values;

- **White Layer Thickness:** 15.85 µm
- **White Layer Hardness:** 1069/0.02/30/HV
- **Surface Hardness:** 581.2/0.5/30/HV
- **Stiff Tissue Hardness:** 337/0.5/30/HV
- **Stiff Tissue Depth:** 0.27 mm
- **Core Hardness:** 290/0.5/30/HV

5. Experiment

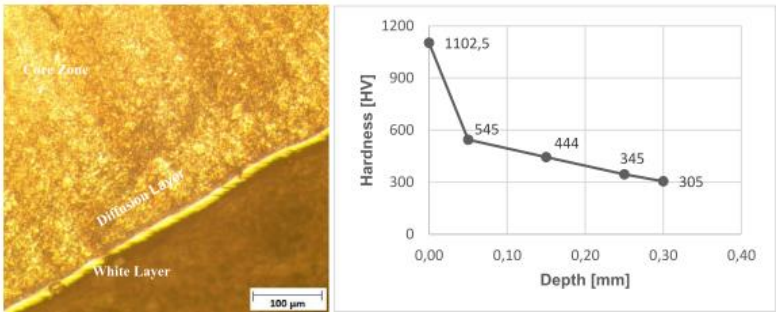


Figure 8. **Microscope Image and Hardness-Depth Graph Resulting from Exp-5**

Micro Hardness Values;

- **White Layer Thickness:** 14.4 µm
- **White Layer Hardness:** 1102.5/0.02/30/HV
- **Surface Hardness:** 545/0.5/30/HV
- **Stiff Tissue Hardness:** 305/0.5/30/HV
- **Stiff Tissue Depth:** 0.30 mm
- **Core Hardness:** 250/0.5/30/HV

6. Experiment

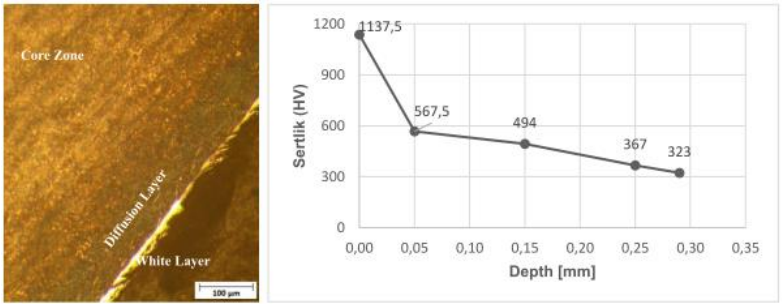


Figure 9. Microscope Image and Hardness-Depth Graph Resulting from Exp-6

Micro Hardness Values;

- **White Layer Thickness:** 12.5 µm
- **White Layer Hardness:** 1137.5/0.02/30/HV
- **Surface Hardness:** 567.5/0.5/30/HV
- **Stiff Tissue Hardness:** 323/0.5/30/HV
- **Stiff Tissue Depth:** 0.29 mm
- **Core Hardness:** 276/0.5/30/HV

7. Experiment

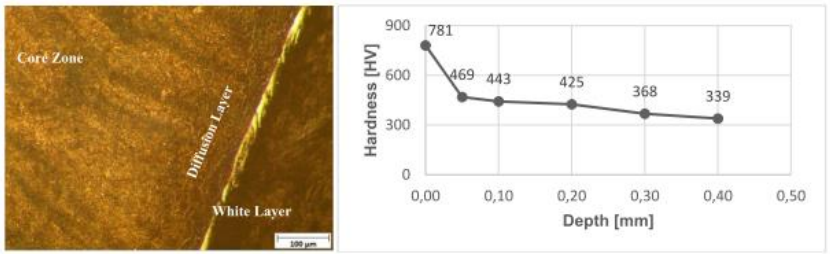


Figure 10. Microscope Image and Hardness-Depth Graph Resulting from Exp-7

Micro Hardness Values;

- **White Layer Thickness:** 22.65 µm
- **White Layer Hardness:** 781/0.02/30/HV
- **Surface Hardness:** 469/0.5/30/HV
- **Stiff Tissue Hardness:** 300/0.5/30/HV
- **Stiff Tissue Depth:** 0.47 mm
- **Core Hardness:** 250/0.5/30/HV

8. Experiment

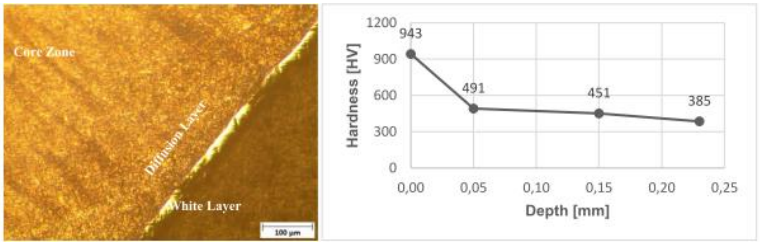


Figure 11. Microscope Image and Hardness-Depth Graph Resulting from Exp-8

Micro Hardness Values;

- **White Layer Thickness:** 18.4 µm
- **White Layer Hardness:** 943/0.02/30/HV
- **Surface Hardness:** 491/0.5/30/HV
- **Stiff Tissue Hardness:** 385/0.5/30/HV
- **Stiff Tissue Depth:** 0.23 mm
- **Core Hardness:** 335.5/0.5/30/HV

9. Experiment

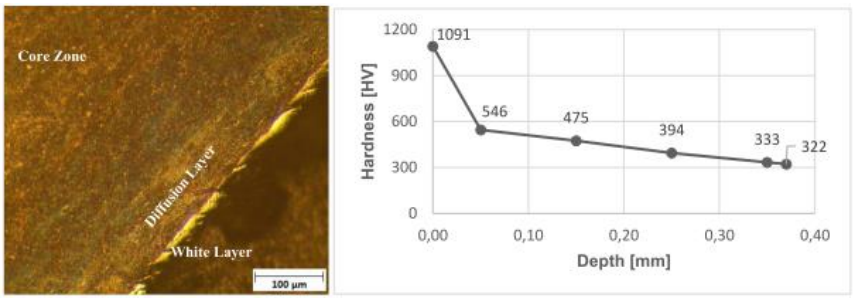
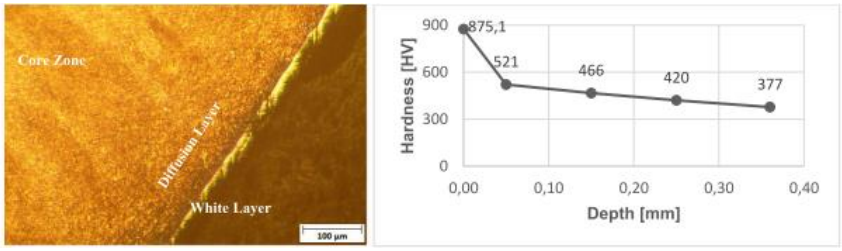


Figure 12. Microscope Image and Hardness-Depth Graph Resulting from Exp-9

Micro Hardness Values;

- **White Layer Thickness:** 16.44 µm
- **White Layer Hardness:** 1091/0.02/30/HV
- **Surface Hardness:** 546/0.5/30/HV
- **Stiff Tissue Hardness:** 322/0.5/30/HV
- **Stiff Tissue Depth:** 0.37 mm
- **Core Hardness:** 265/0.5/30/HV

10. Experiment



icro Hardness Values;

- **White Layer Thickness:** 22.4 µm
- **White Layer Hardness:** 875.1/0.02/30/HV
- **Surface Hardness:** 521/0.5/30/HV
- **Stiff Tissue Hardness:** 377/0.5/30/HV
- **Stiff Tissue Depth:** 0.36 mm
- **Core Hardness:** 317/0.5/30/HV

3.3 Wear Test Results

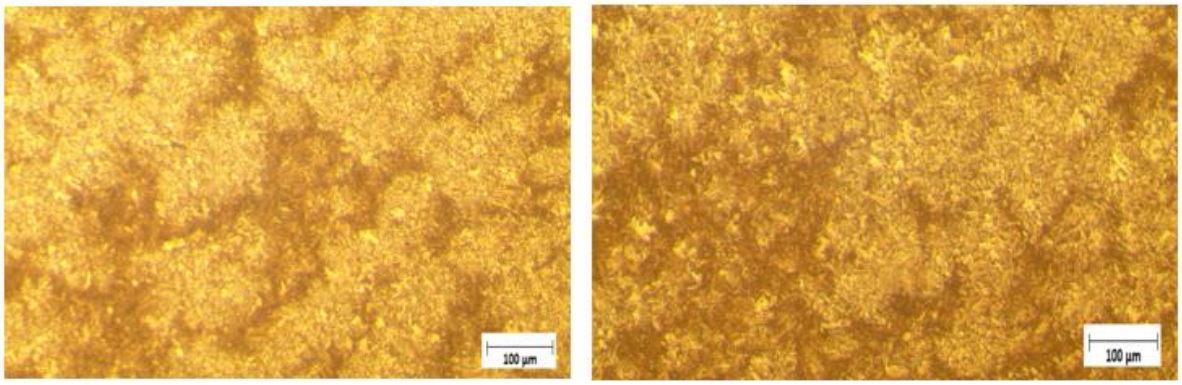


Figure 14. Microscope images taken from different parts of the 1st sample as a result of wear

As a result of the wear test performed with the parameters specified in Table 7, the amount of wear for the test sample "1" was measured as 43.470 x 10^{-5} mm^3/n/m.

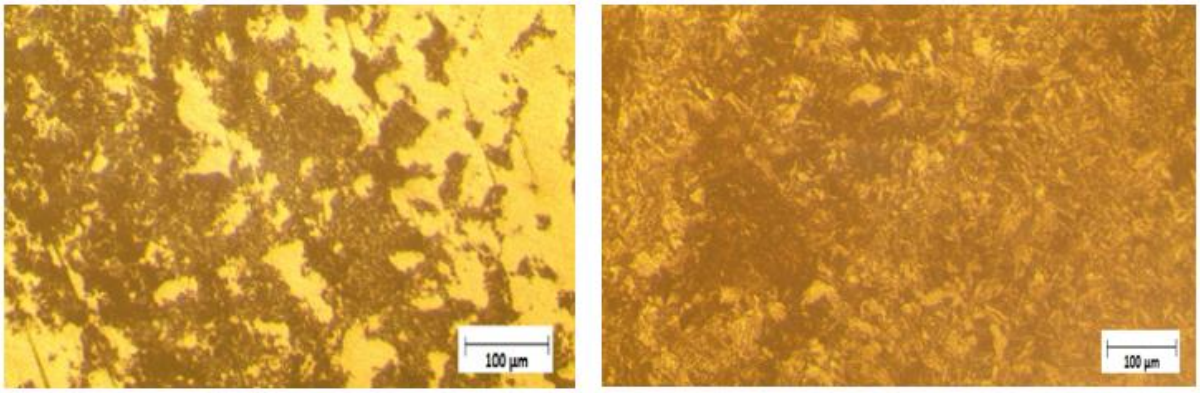


Figure 15. Microscope images taken from different parts of the 3rd sample as a result of wear

As a result of the wear test performed with the parameters specified in Table 7, the amount of wear for the test sample "3" was measured as 1.573 x 10^{-5} mm^3/n/m.

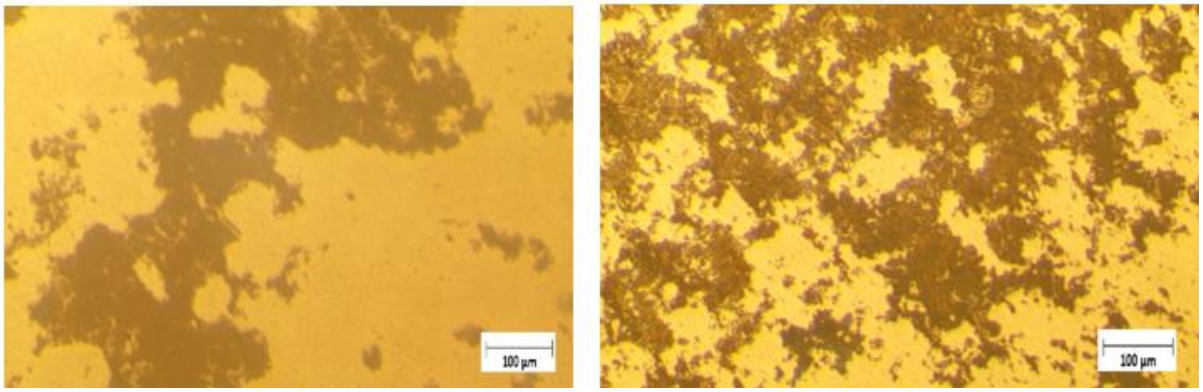


Figure 16. Microscope images taken from different parts of the 7th sample as a result of wear

As a result of the wear test performed with the parameters specified in Table 7, the amount of wear for the test sample "7" was measured as 4.280 x 10^{-5} mm^3/n/m.

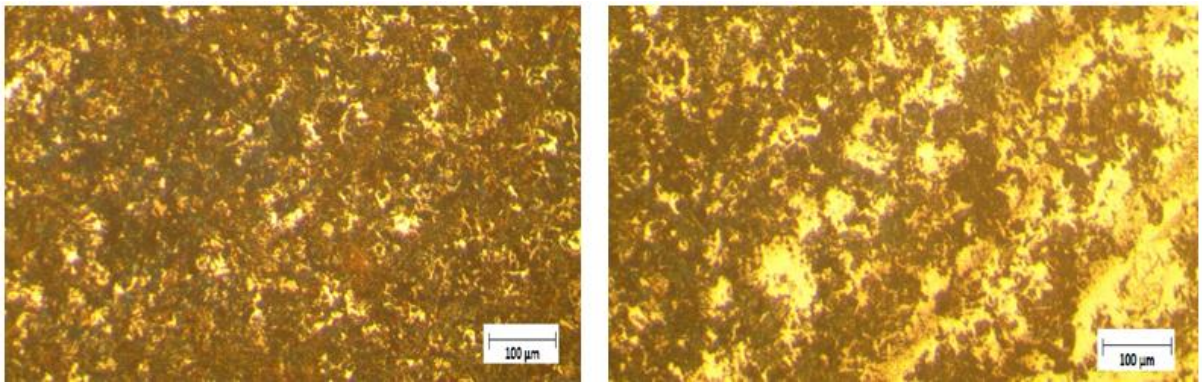


Figure 17. Microscope images taken from different parts of the 10th sample as a result of wear

As a result of the wear test performed with the parameters specified in *Table 7*, the amount of wear for the test sample "10" was measured as 2.341 x 10^{-5} mm^3/n/m.

Table 9. ***Parameters and Results for Wear Test***

Sample	Temperature [°C]	Time [h]	Flow Rate [m^3/h]	Mean Coefficient of Friction	Amount of Wear *(10^{-5}) [mm^3/n/m]	Hardness [HV0.5]
1	470	7	6	0.625	43.470	607
3	470	12	9	0.685	1.573	581.2
7	570	7	9	0.550	4.280	469
10	570	12	6	0.721	2.341	521

Wear value is inversely proportional to wear resistance as seen in *Table 9*, the best wear resistance was observed in samples 3 and 10.

Due to the low hardness, hard wear are observed on the surface [17]. Similar to Topcu's study, the results of wear tests of 3rd,7th and 10th experiments it can be concluded that the hardness is inversely proportional to the amount of wear. Except the 1st experiment. The 1st sample was worn more than the other samples. That the reason can be attributed to the high hardness leads to brittleness, thus causing fracture and loss of material.

3.4 Residual Stress Test Results

The results of residual stresses are listed in *Table 10.* A compressive stress of 267.6 MPa at a depth of 219 microns from the surface in the 3rd test sample and a compressive stress of 289.3 MPa at a depth of 212.5 microns in the 10th test sample were found for SAE 4140 steel. Similarly, it was mentioned that the compressive stress increases as the temperature value increased [18]. It was also found that the tensile residual stress measured on the surface of the (12 HRC) AISI 4140 steel sample as 211 MP at a depth of 0.03 mm from the surface [19].

"**White Layer Hardness** = 1968 - 1.18 Temperature - 3.4 Time- 42.2 Flow"

In order to interpret the equation created as a result of the regression analysis more clearly, the hardness values and the main effect graph in the Anova module of the Minitab program were created (*Figure 18*).

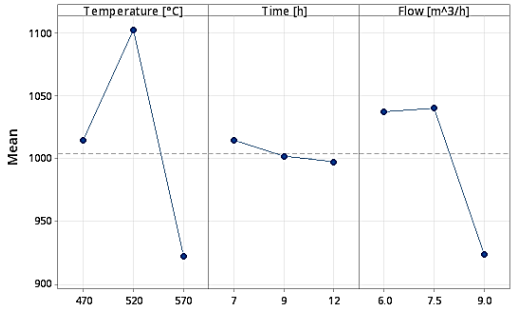


Figure 18. Main Effects Plot for White Layer Hardness

The hardness and depth of hardness values are decreased at short processing times and at low temperatures since the diffusion becomes difficult. At high temperatures (over 500 °C), a decrease in hardness is observed due to the formation of coarse precipitates [20]. As a result of the Minitab studies given in *Figure 18*, the hardness values generally increased at temperature up to 520 °C, and decreased after 520 ° C.

Table 10. Measured Results of Residual Stress

Sample	Temperature [°C]	Time [h]	Flow Rate [m^3/h]	σ [MPa]
3	470	12	9	-267.6
10	570	12	6	-289.3

3.5 Minitab Analysis

"Regression Analysis" was performed in Minitab program to interpret the effect of different values of parameters on the result. The regression equation for the hardness value is givens. While performing this analysis, the "White layer hardness" parameter as the "Response" value, and the "Temperature, Time and Flow" parameter values, which are the nitration parameters of the study, as the "Predictor" value and given in *Table 3*. The created equation is;

The same analysis was made for the white layer thickness values, and the "White Layer Thickness" parameter as the "Response" value, and the nitration parameters of the study, "Temperature, Time and Flow" parameters, as the "Predictor" value. The created equation is;

"**White Layer Thickness (µm)** = -36.1 + 0.0873 Temperature – 0.013 Time + 0.838 Flow"

In order to interpret the equation created as a result of the regression analysis more clearly, the white layer thickness values and the main effect graph in the Anova module of the Minitab program were created (*Figure 19*).

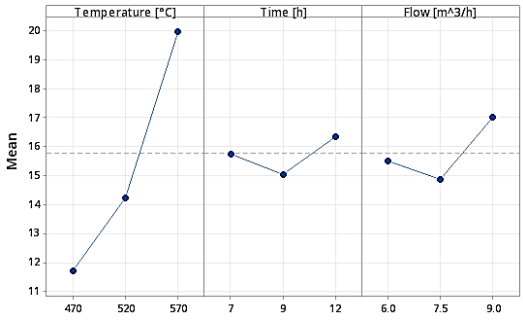


Figure 19. Main Effects Plot for White Layer Thickness

The analysis for the White Layer Hardness and white layer thickness were made for the wear values, and the "Response" value was added to the "Wear Value" parameter, and the "Predictor" value was the nitration parameters of the study, "Temperature, Time and Flow" parameters are entered. The created equation is;

"**Wear Value x (10^{-5})** = 204.4 - 0.1921 Temperature- 4.384 Time - 6.660 Flow"

Among these parameters, it has been observed that the parameter that affects the wear value the most is the flow parameter.
In order to interpret the equation created as a result of the regression analysis more clearly, the wear values and the main effect graph in the Anova module of the Minitab program were created (*Figure 20*).

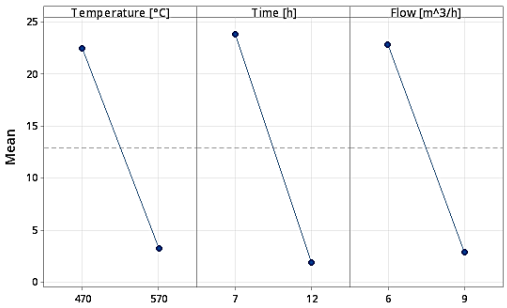


Figure 20. Main Effects Plot for Wear Value

In general, it is expected that the wear value will increase as the friction coefficient increases [18]. However, in this study, the non-planarity of the test samples and the presence of traces on the sample surface due to the cutting process during the sample preparation process caused the correct ratio between the friction coefficient and the wear value to not be fully achieved. Therefore, maximum wear could not be obtained in the sample with the maximum average friction coefficient in the study.
In the white layer hardness, analysis and the least affecting parameter is the temperature parameter.
As a result of the wear measurements, the samples with the highest and lowest wear resistance were the sample of the 3rd Experiment, and 1st Experiment, respectively. As can be seen from the test parameters and the regression equation, the flow parameter has a significant effect on the strength.
It was observed that the two samples with the highest wear resistance were the 3rd Experiment and 10th Experiment samples, respectively. When the test parameters of these two samples were examined, it was determined that the flow rate parameter had more effect on the strength value than the temperature.
Residual stresses of test samples 3 and 10 were measured by X-ray diffraction method in order to correlate the residual stresses between the two samples with the best wear resistance so to reach the aim of the project that is maximum wear resistance and minimum residual stress. As a result of the measurements listed in *Table 10*, compressive stresses were 276.6 MPa in experiment 3, and 289.3 MPa in experiment 10, respectively.
In conclusion, experiment 3 provides both the maximum wear resistance and the minimum residual stress value.

4.CONCLUSIONS

- In this study; the effect of nitration parameters on material properties was observed by using 3 different parameters and different values of these parameters.
- After all the experiments and measurements were completed, the data was evaluated by the Minitab program and regression equations were generated. It was observed that the flow rate was the most effective parameter.
- As a result of the study, the highest surface and white layer hardness was obtained in the 1st Experiment.
- The sample with the highest wear resistance was obtained in the 3rd Experiment, and the lowest one was obtained in the 1st Experiment.
- The sample with the lowest residual stress value was obtained in the 3rd Experiment.
- Based on these results, the sample that provides the "maximum wear resistance and minimum residual stress" values, which is the aim of the study, was determined as number 3.
- The application of nitration by the optimum parameters specified in this study increased the wear resistance of the material by 60%.

ACKNOWLEDGMENT

This study was carried out at Hidromek Component Factory. So, we would like to thank HIDROMEK for their support in the realization of this study, and

TUBITAK BIDEB for their support within the scope of TUBITAK 2209-B Industry-Oriented Undergraduate Research Projects Support Program.

DECLARATION OF ETHICAL STANDARDS

The author(s) of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

AUTHORS' CONTRIBUTIONS

Sibel TÜZÜNER: Performed the experiments and wrote the manuscript.
Ali Baran METİN: Performed the experiments and wrote the manuscript.
Ebru SARALOĞLU GÜLER: Helped with article layout.

Tuğçe ŞAHİN: Helped with article layout.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

REFERENCES

[1] Kaplan Y., Yildirim A., Aksoz S., "The Effect of Oxidation Process After Nitrocarburization on Tribological Properties of AISI 4140 Steel", ***Journal of Polytechnic***, 23(4): 1357-1362, (2020)

[2] Polat S., Atapek S.H., Topac H., "Microstructural Characterization on AISI 4140 and DIN 1.2344 Steels Surface Hardened by Gas Nitration", ***6th International Advanced Technologies Symposium (IATS'11)***, Elazig Turkey, 498-502, (2011).

[3] Mcquaid H.W., Ketcham W.J., "Practical Nitriding and Ferritic Nitrocarburizing", Source Book on Nitriding, ***ASM International***, Ohio USA, (2003).

[4] ASM International Handbook Committee, "Gas Nitriding of Steels", Metals Handbook, ***ASM International***, Vol.4 Heat Treating, 880-882, (1999).

[5] Rakhit A.K., "Nitriding Gears", Heat Treatment of Gears, ***ASM International***, USA, (2000).

[6] BS EN 10085, "Nitriding Steel Technical Delivery Conditions", (2001).

[7] Peng D. Q., Kim T. H., Chung J. H., Park J. K., "Development of nitride-layer of AISI 304 austenitic stainless steel during high-temperature ammonia gas-nitriding", ***Applied Surface Science***, 256: 7522-7529, (2010)

[8] Hernandez, M. H. S. M., Staia, M. H. & Puchi-Cabrera, E. S., "Evaluation of microstructure and mechanical properties of nitrided steels", ***Surface and Coating Technologies***, 202, 1935-1943, (2008)

[9] Yaman O., "Assesment of the Nitriding Process Parameters to Improve the Wear Resistance of Selected Steels", ***M.Sc.***, Bogazici University Mechanical Engineering, (2009).

[10] Sree, K. R., Reddy, G. K., Aishwarya, K., Devi, E. N., & Subbiah, R., "New Insights of Wear Behavior Analysis on Low Temperature Treated AISI 253MA Stainless Steel Material by Gas Nitriding Process", ***E3S Web of Conferences ICMED (Vol. 184, p. 01003)***, (2020).

[11] Pinar M.A., Firat K., "Optimization of Surface Roughness Performance in the Turning of AISI 4140 Steel with Multi Directional Cutting Tool", ***Journal of Polytechnic,*** 19(4): 491-498, (2016)

[12] Wang S., Wang Y., Liu C., & Mazumder J., "In-Situ Monitoring on Micro-Hardness of Laser Molten Zone on AISI4140 Steel by Spectral Analysis", ***Scientific Reports***, 10(1): 1-12, (2020)

[13] SAE J 1268, "Standard Alloy H Steels", (2010).

[14] Porter D.A., Easterling K.E., "Phase Transformations in Metals and Alloys 4th Edition", ***CRC Press***, USA, (2021).

[15] Korkmaz. K, "Effects of The White Layer Formed by Gas Nitriding on The Wear Behavior of Nitriding Steel", ***M.Sc.***, Gebze Technical University Department of Materials Science and Engineering, (2020).

[16] Holm, T., Sproge, L., "Nitriding and Nitrocarburizing", Furnace Atmospheres 3, ***AGA***, SWE, (1996).

[17] Topcu S., "Determination of Wear Behavior of Plasma Nitrided Steels Under Different Conditions", ***M.Sc.***, Uludag University Institute of Science Department of Mechanical Engineering, (2010).

[18] Gecu R., "Investigation of Residual Stress and Hardness Changes in Gas Nitrided 31CrMoV9 Steel by Indentation Method", ***M.Sc.***, Istanbul Technical University Metallurgical and Materials Engineering Department Materials Engineering Program, (2014).

[19] Subasi M., Karatas C., "Investigation of the Effect of Different Hardnesses Obtained in AISI 4140 Steel on Residual Stress", ***Journal of Polytechnic***, 14(4): 289-295, (2011)

[20] Asan N.O., "Improving the Surface Properties with Nitrogen Diffusion to the Steel Surface", ***M.Sc.,*** Yıldız Technical University Metallurgical and Materials Engineering, (2004)