

Surface Treatment Effects of Nitration Parameters on 2344 Tool Steel

Fırat Çetinkaya¹, Bilgehan Tunca², Bahadır Karaca³, Anıl Demirci⁴

^{1,2,3,4}Department of R&D Center, Zahit Alüminyum San. ve Tic. A.Ş.

ABSTRACT

In this study; The effects of different processes applied to heat-treated tool steels on the hardness and wear resistance of the steel were examined. In line with this study, the effects of steels subjected to different times and processes on diffusion depth, white layer thickness and hardness were investigated. The molds were tested with the extrusion process in the determined parameters and their wear resistance was determined. After examining the results, the most suitable parameters for mold steels were determined.

Keywords: 2344 Tool Steel, White Layer, Homogenization, Extrusion, Microstructure, Diffusion, Gas Nitriding

1. INTRODUCTION

The use of steels constitutes the most important effects of the beginning of modern life. Combining the material properties given to us by nature with each other's properties and using them as a new material, instead of using them alone, has become one of the most effective methods developed by humankind. Buildings have been built in different shapes and sizes throughout history, but after the use of steel as a building material, people began to build higher and stronger structures. Using the metallurgical properties of steel has paved the way for us to build skyscrapers and structures that will cross the seas. Steels are an alloy with high tensile strength and low cost produced by combining the properties of more than one metal. These alloys, whose basic element is iron, are classified by different carbon contents. 2344 Heat treated tool steels are a type of steel that resists high forces loaded on it. These steels, which are generally used in extrusion processes, enable the desired metal to be shaped by resisting the force applied by the presses. The steels to be used in the extrusion process must be a suitable metal for nitriding. 2344 tool steel is a suitable steel for nitriding due to the carbon content it contains and the properties of the alloy metals. In this study, the effect of the nitriding process on steel to which different parameters were applied and its contribution to the durability in the extrusion process were examined. The formation of the nitrided case results from the "compound layer" and "diffusion zone" from the surface to the core [1]. The field under the diffusion zone is the core of the steel including tempered martensite. The compound layer contains epsilon phase (ϵ -Fe₂₋₃N), gamma phase (γ -Fe₄N) or a mixed-phase (ϵ + γ) [3].

2. EXPERIMENTAL STUDY

2.1. Nitriding Process

The nitriding process is a thermochemical surface treatment based on the development of the surface with nitride phases in consequence of the diffusion of nitrogen ions into the material surface [2]. Nitriding is a type of surface hardening process using heat. With the effect of temperature, the space between the metal

atoms expands and the diffusion effect of ammonia gas passes down the steel surface, hardening the surface. Another purpose of the nitriding process is to prevent surface wear that may occur due to thermal fatigue and friction of the surface of another material. As a result of the nitriding process, an increase in the fatigue, corrosion and wear resistance of steels is observed. Improvement of the fatigue life using nitriding requires steels which contain nitride-forming alloying elements. The steel composition should also display good fatigue properties of the unnitrided core [4].

In this study, nitriding processes were applied to 2344 tool steels with different parameters. The surface of the sample steels was cleaned by sandblasting and nitriding was carried out. The prepared samples were nitrided at different times and different gas flows. After the nitriding process, the samples were cooled with air and made ready for examination. The samples have the same chemical composition but different parameters. The nitrided steels were cut to the same dimensions and after surface treatments, their metallographic structure was examined and their hardness values were measured.

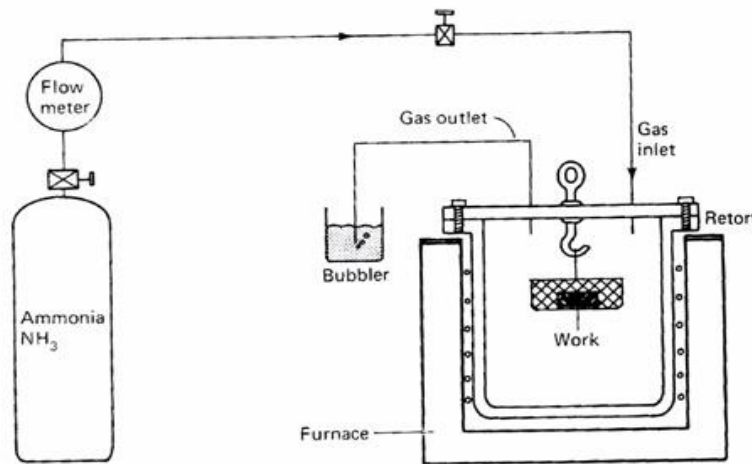


Figure 1. Schematic representation of the nitration process

Application of the gas nitriding process ensures that ammonia gas (NH_3) is broken down by high temperature and the effect of the process, nitrogen gas is released and spread to the surface. The diffusion depth of steel increases in direct proportion to the processing time and temperature. During the nitriding process, iron and nitride precipitates, which are the basic alloying elements of steel, combine to form iron nitride (white layer) on the surface of the steel. Although the thickness of this layer varies depending on the flow rate of the applied gas, it provides corrosion and wear resistance to the steel. In addition, corrosion is also a possible mode of degradation due to combined action of temperature and aggressive lubricants[10][11]. The nitrogen is found as an intermediary atom in the iron cubic or finely dispersed alloy nitride in the diffusion zone. The hardness value decreases due to the decreasing amount of nitrogen in the inner parts of the steel[6]. While the white layer contributes to the development of the corrosive properties, the diffusion layer improves the tribological properties and fatigue strength of the steels[5][1].

The growth rate of the nitrided layer can be increased by increasing the nitriding temperature, which is the basis for development of the solution nitriding or high temperature gas nitriding process for stainless steels[7][8][9].

2.1.1. Nitriding Process Parameters

Differences in parameters were taken into account in the nitration process. The sample steels have the same physical properties and chemical composition. In the nitriding process, the properties of the steel's resistance to diffusion depth and surface wear were investigated at different gas flow rates and times. The parameters in the nitration process were tested in 3 different periods and with 2 different gas flow rates in each period. These parameters are shown in Table 1.

Table 1. Nitriding process parameters

Sample No	Time (Minutes)	Gas Flow (mbar)
Sample 1	360	1800
Sample 2	360	1600
Sample 3	390	1800
Sample 4	390	1600
Sample 5	420	1800
Sample 6	420	1600

2.1.2. Chemical Composition

Different numbers of samples were taken from a single steel in order to learn the parameter differences of the sample steel. Steels with the same chemical composition were subjected to the same cleaning time and surface treatment. The chemical composition of the steel from which the samples were taken is given below. Table 2 shows the chemical composition standard of 2344 tool steel. Table 3 shows the chemical composition analysis of the 2344 tool steels we used in this study.

Table 2. Standard chemical composition of 2344 steel

Steel Grade	C%	Si%	Mn%	P%≤	S%≤	Cr%	Mo%	V%
DIN 1 .2344	0.35- 0.42	0.8-1.2	0.25- 0.50	0.03	0.02	4.80- 5.50	1.20- 1.50	0.85- 1.15

Table 3. Chemical composition of 2344 steel sample

C%	Si%	Mn%	P%	S%	Cr%	Mo%	Ni%	Cu%
0,35	0,886	0,318	0,022	0,007	4,843	1,318	0,216	0,142
Al%	As%	Co%	Sn%	Ti%	V%	W%	N%	Fe%
0,029	0,015	0,052	0,001	0,007	0,926	0,072	0,035	90,86

2.2. Metallographic Process

The samples brought to the laboratory for metallographic examination were first cut homogeneously with a wet saw to the same size and weight. Then, the sized samples were molded with black bakelite powder with the same parameters so that they could be exposed to the same pressure during the sanding process. After the bakelite process, the surfaces of the samples were smoothed with 180, 240, 400, 600, 800 and 1200 grit SiC sandpaper, respectively.

After the sanding process, the surface was washed with soapy water and dried with ethanol to remove small grain burrs formed during the process. Since an optical microscope was used during the metallographic examination, the surface was polished using 3 different sized cloths with 6 micron, 3 micron and 1 micron diamond suspension. 3% nital etching solution was prepared for the etching process. The etching solution was poured onto the samples with the help of droplets and the etching process was carried out. In the last part, the samples were washed with pure water, dried with ethanol and made ready for examination under a microscope.

2.3.1. Hardness Test

Samples were taken from all samples and measured with the Leeb Hardness Tester hardness device. The measuring device measures hardness by bouncing. Measurements were made at a right angle and in HRC to ensure the correct value and to affect the surface of the sample. For each sample, measurements were made from 5 different points and average values were taken.

The hardness values of the steels were measured after the heat treatment of the samples, but since the examination would be made after the nitriding process, the hardness measurements of the samples were made after the tenifer process. The samples have equal size and length on a flat surface. Hardness measurements were made from the same points of the samples.

3. RESULTS & DISCUSSION

3.1 Sample Analyzes

After the samples were processed with different parameters, metallographic examination and hardness measurements were made and they were made ready for discussion and selection of the most suitable parameter. As a result of the examination, it was determined that the selected parameter had the highest surface wear resistance value.

Surface wear of steel samples was compared after testing them in the same extrusion process. It has been determined that with appropriately determined parameters, the steel surface lasts longer than molds made with other parameters due to the effect of high temperature and friction, and no surface defects are observed. Since the applied forces were the same, the hardness values of the presses were measured and compared with a bouncing hardness measuring device.

Metallographic images and hardness measurement values of the examined samples are given below. Metallographic examinations and hardness measurements are listed according to process length parameters.

The examinations of the samples to which a process length of 360 minutes and a gas flow rate of 1600 to 1800 mbar were applied are given below;

Sample 1 - White layer image of the sample to which 1800 mbar gas flow rate was applied

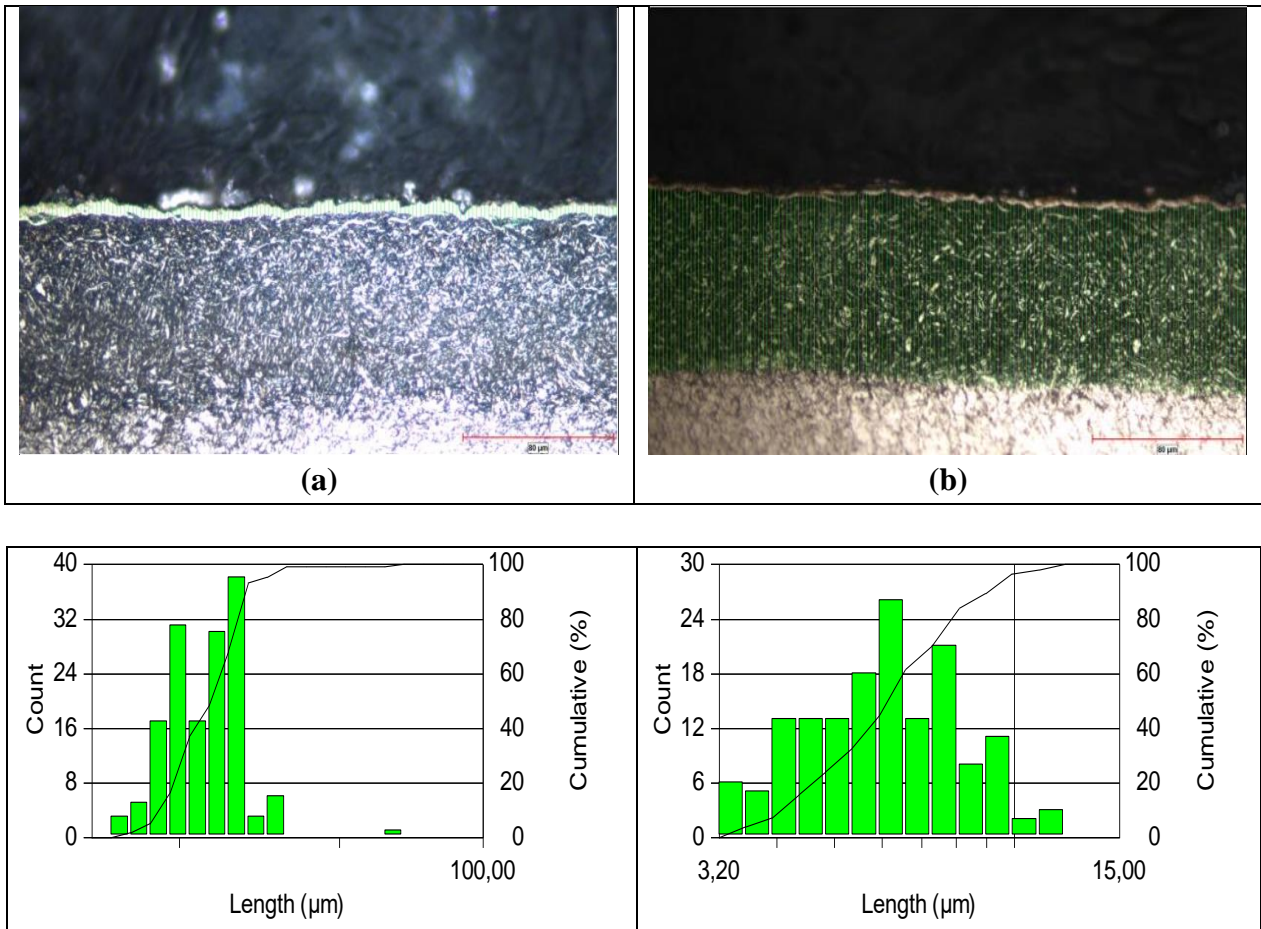
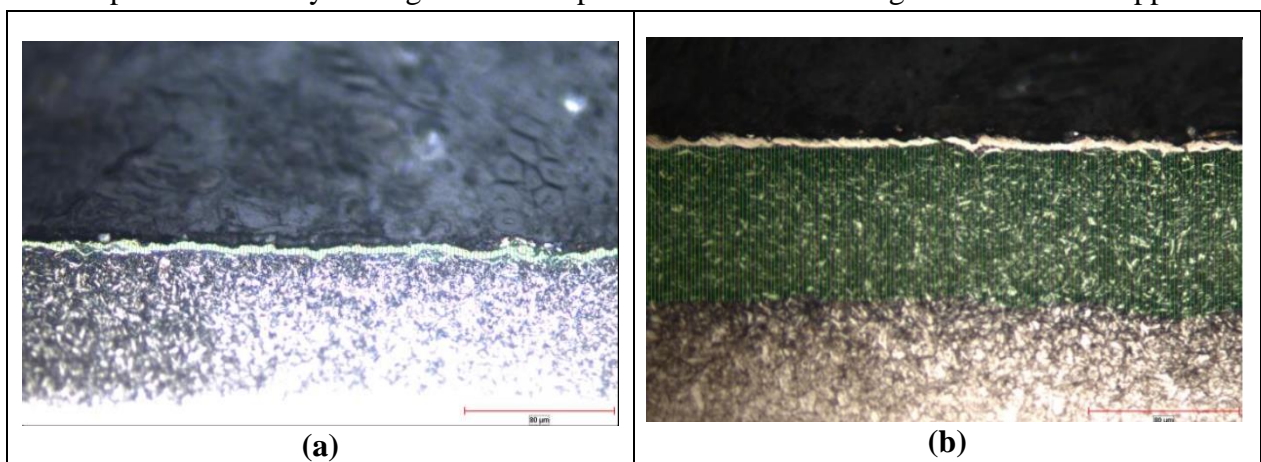


Figure 2. (a) is white layer and (b) is diffusion depth images of sample 1

Sample 2 - White layer image of the sample to which 1600 mbar gas flow rate was applied



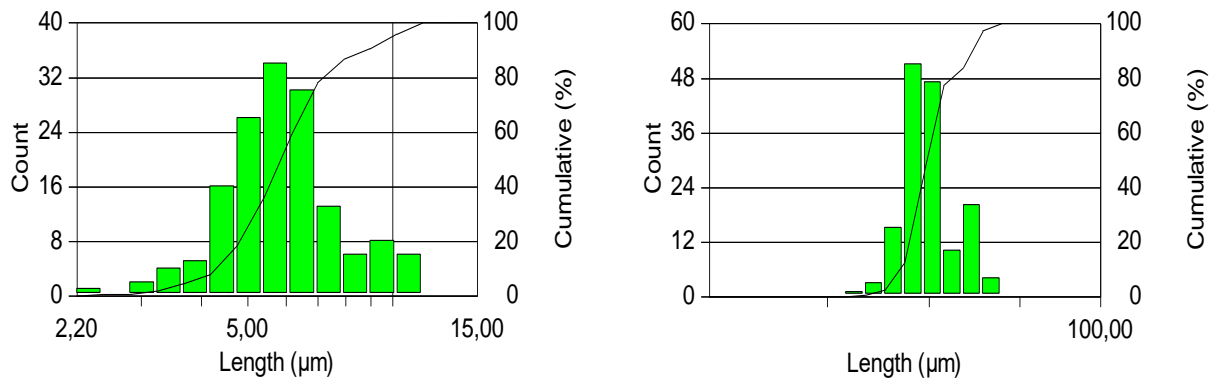
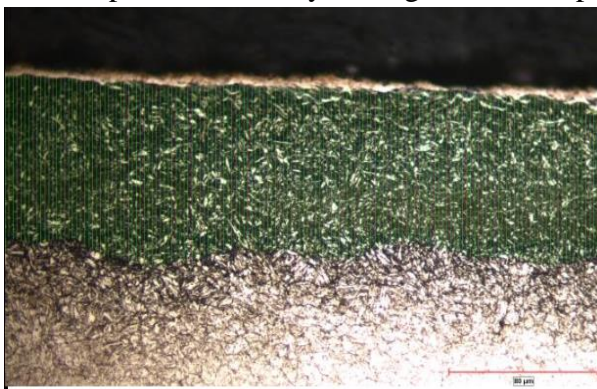


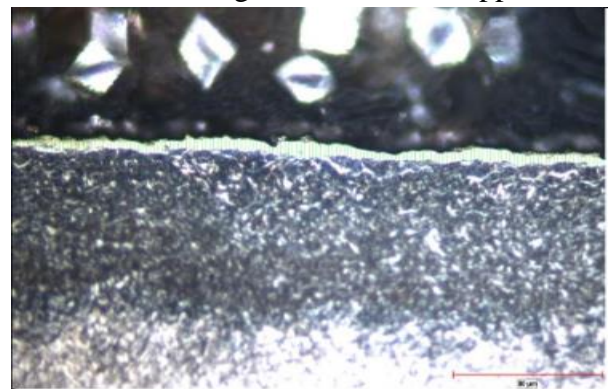
Figure 3. (a) is white layer and (b) is diffusion depth images of sample 2

The examinations of the samples to which a process length of 390 minutes and a gas flow rate of 1600 to 1800 mbar were applied are given below;

Sample 3 - White layer image of the sample to which 1800 mbar gas flow rate was applied



(a)



(b)

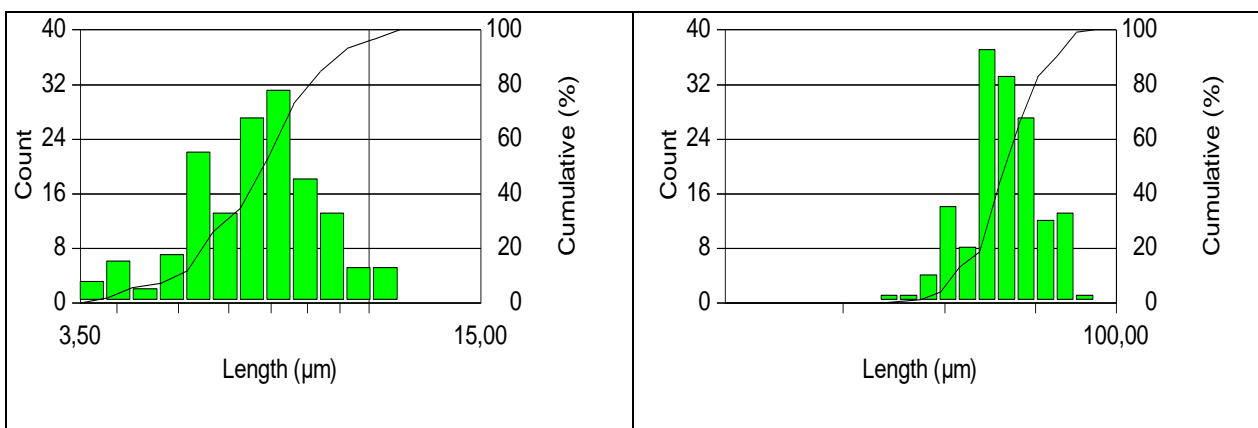


Figure 4. (a) is white layer and (b) is diffusion depth images of sample 3

Sample 4 - White layer image of the sample to which 1600 mbar gas flow rate was applied

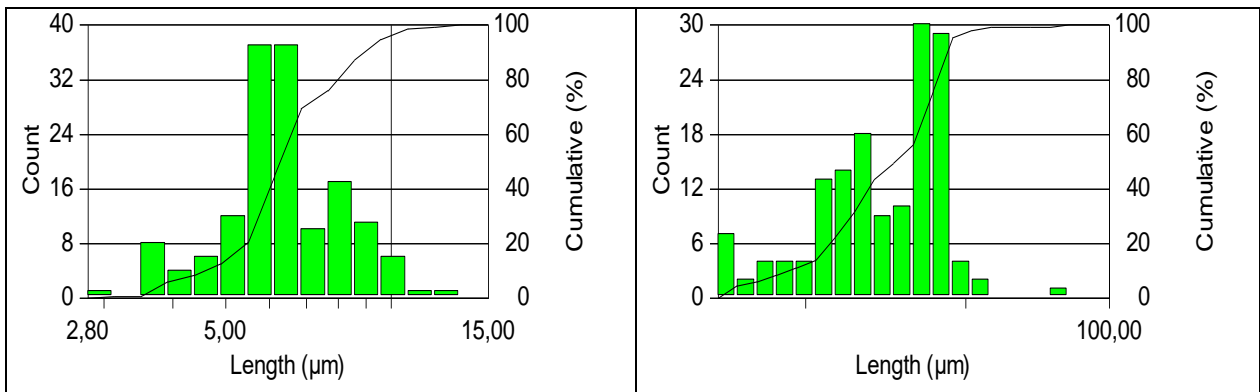
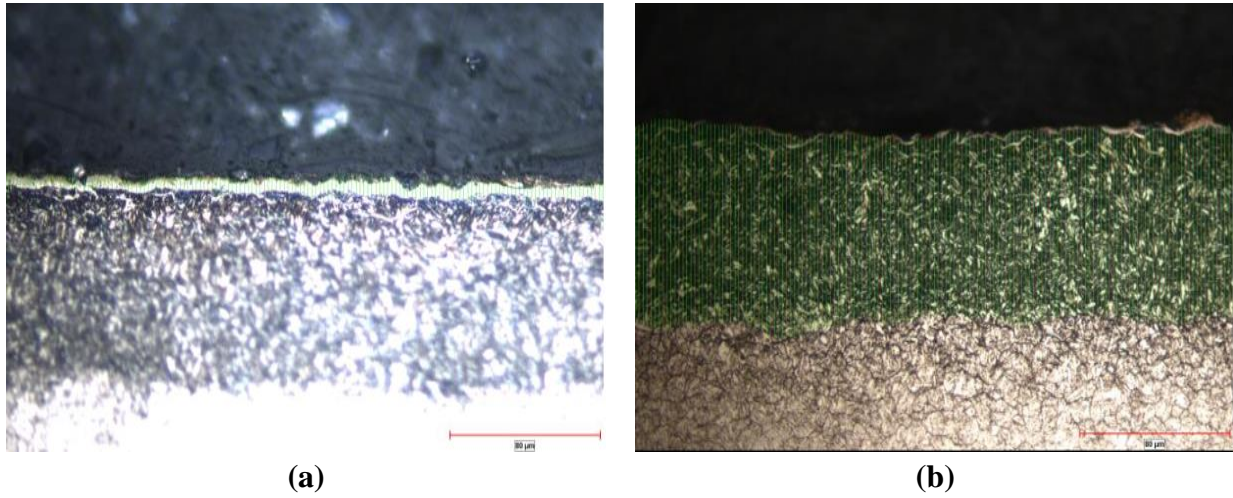
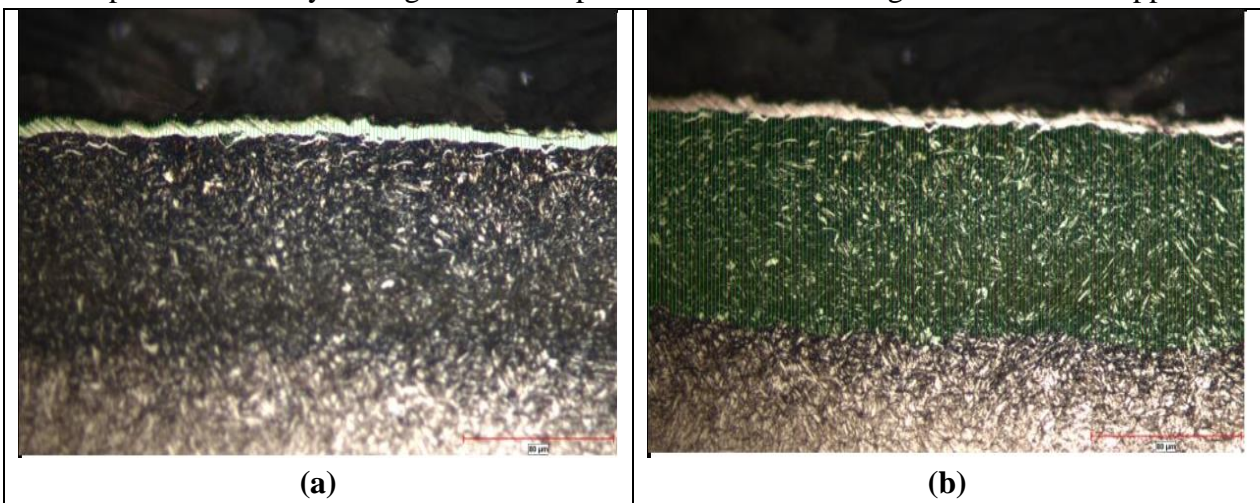


Figure 5. (a) is white layer and (b) is diffusion depth images of sample 4

The examinations of the samples to which a process length of 420 minutes and a gas flow rate of 1600 to 1800 mbar were applied are given below;

Sample 5 - White layer image of the sample to which 1800 mbar gas flow rate was applied



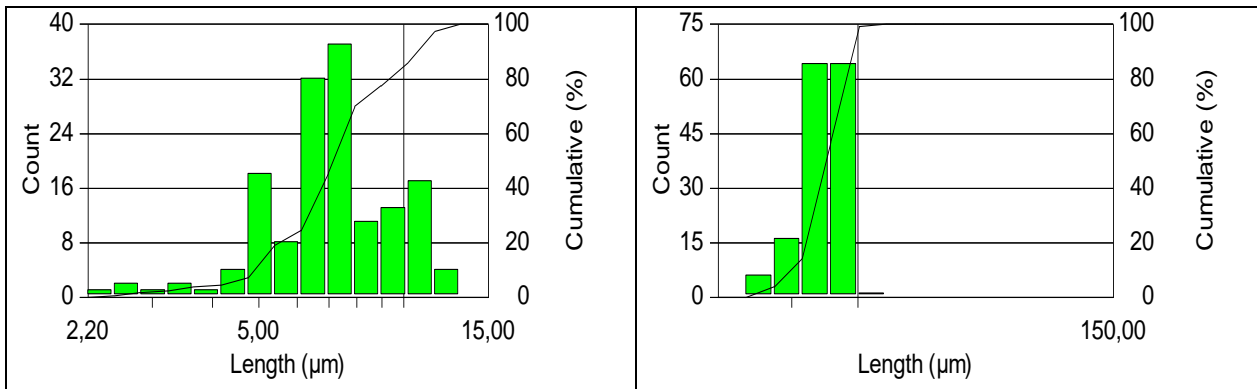


Figure 6. (a) is white layer and (b) is diffusion depth images of sample 5

Sample 6 - White layer image of the sample to which 1600 mbar gas flow rate was applied

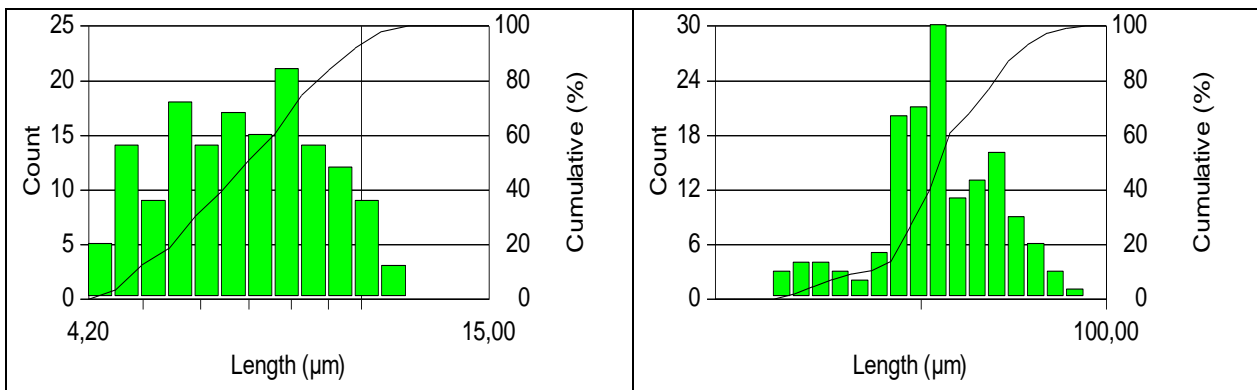
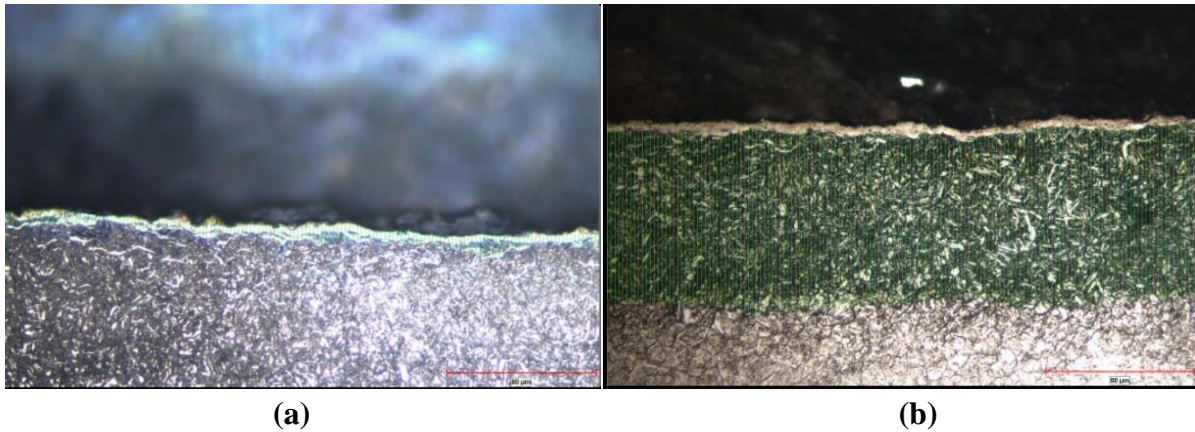


Figure 7. (a) is white layer and (b) is diffusion depth images of sample 6

The white layer and thickness of the samples were measured in micron size. Images of measurements made with an optical microscope are given in the analysis section. Measured white layer, diffusion depths and hardness values are given in the table below. Table 4 shows the microstructure measurements and hardness values of the samples.

Table 4. Microstructure measurements and hardness values of the samples

Sample No	White Layer (μm)	Difusion Depth (μm)	Hardness (HRC)
Sample 1	6,32	81,62	52,1
Sample 2	6,12	80,05	51,7

Sample 3	6,87	86,49	55,1
Sample 4	6,54	84,52	54,5
Sample 5	7,36	94,82	56,6
Sample 6	7,12	90,98	56,1

After the measurements, in addition to the scale values, the surface wear resistance was also examined during the extrusion process in order to determine the appropriate parameters. It has been calculated that a mold steel can absorb an average of 1800 to 2000 meters of aluminum in the drawing process after the initial nitriding. Following the transactions, it was aimed to increase this amount. Following this experiment, profiles with a certain wall thickness were extruded to determine how long the surface would wear. The values given have been tested with 6xxx series aluminum. The tensile strength of 6xxx aluminum alloy is 215 MPa and above, and the yield strength is 170 MPa and above. For the research, experiments were carried out on sample steels in line with the determined parameters and it was determined that the most suitable parameter was surface wear resistance. Table 5 shows the aluminum profile lengths drawn according to tenifer values.

Table 5. Aluminum profile lengths drawn according to Tenifer values

Sample (Die) No	Tenifer Values (µm)	Pulled Aluminum length (meters)
Sample 1	81,62 – 6,32	1892
Sample 2	80,05 – 6,12	1875
Sample 3	86,49 – 6,87	1932
Sample 4	84,52 – 6,54	1911
Sample 5	94,82 – 7,36	2172
Sample 6	90,98 – 7,12	2032

It was determined that the steel sample, which had the highest values among the samples examined, resisted the abrasion caused by the aluminum profiles on the surface for a longer time than the average values. While the diffusion depth provides durability to the mold in the extrusion process, the effects of the white layer thickness on surface wear were determined. In bridge molds, profiles with lengths that can exceed average values were obtained from steel molds with high white layer thickness.

It has been noticed that the thickness of the white layer on the surface decreases with the temperature and friction of the aluminum used. It has been determined that there is a direct proportion between the thickness of the white layer structure, which increases the surface resistance, and the meter length of the drawn profiles. It has been observed that the surface of the profiles drawn in steel molds with high surface thickness has a very smooth and homogeneous distribution.

4. CONCLUSION

The effect of the nitriding process on the surface and microstructure of 2344 heat treatment tool steels under different parameters such as temperature and gas flow rate was examined and the following results were obtained.

1. As a result of the relevant parameters in the tenifer process for 2344 steels, the highest diffusion depth is 94.82 µm and the thickest white layer thickness is 7.36 µm.

2. As a result of the relevant parameters, the highest hardness is 56.6 HRC and the lowest hardness is 51.7 HRC.
3. It has been determined that temperature parameters affect the diffusion depth during the nitriding process and the hardness of 2344 steels increases in direct proportion to the temperature.
4. According to the microstructure examined, the increase in thickness of 2344 steels is directly proportional to the gas flow rate in the nitriding process.
5. The values in the examination and the parameter with the longest wear resistance in the extrusion process are the sample 5 with a gas flow rate of 1800 mbar and a nitration time of 7 hours.
6. As a result of all examinations, among the parameters that will increase the surface wear resistance are temperature and diffusion depth, which increase the white layer thickness and hardness value.

5. REFERENCES

1. Ataş Bakdemir, S. , Özkan, D. , Türküz, M. C. , Uzun, E. & Salman, S. (2020). Effect of the Nitriding Process in the Wear Behaviour of DIN 1.2344 Hot Work Steel . Journal of Naval Sciences and Engineering , JOURNAL OF NAVAL SCIENCES AND ENGINEERING [en] , 45-70 .
2. Arif, A.F.M., Akhtar, S. S., Yilbas, B. S. (2010). Effect of Process Variables on Gas Nitriding of H13 Tool Steel With Controlled Nitriding Potential. International Journal of Surface Science and Engineering, 4, 396–415.
3. Yeh, S.-H., Chiu, L.-H., Chang, H. (2011). Effects of Gas Nitriding on the Mechanical and Corrosion Properties of SACM 645 Steel. Engineering, 3, 942–948.
4. Barrallier, L. (2015). Classical Nitriding of Heat Treatable Steel. Woodhead Publishing Limited, 393-412.
5. Birol, Y., Yuksel, B. (2012). Performance of Gas Nitrided and AlTiN Coated AISI H13 Hot Work Tool Steel in Aluminium Extrusion. Surface and Coatings Technology, 207, 461–466.
6. Baranowska, J. (2010). Importance of Surface Activation for Nitrided Layer Formation on Austenitic Stainless Steel. Surface Engineering, 26, 293–298.
7. Li, K. Y., Xiang, Z. D. (2010). Increasing Surface Hardness of Austenitic Stainless Steels By Pack Nitriding Process. Surface and Coatings Technology, 204, 2268–2272.
8. Berns, H. Advantages in solution nitriding of stainless steels. Met Sci Heat Treat 49, 578–580 (2007).
9. Mitsui, Hajime & Kurihana, Shinsuke. (2007). Solution Nitriding Treatment of Fe-Cr Alloys under Pressurized Nitrogen Gas. Isij International - ISIJ INT. 47. 479-485. 10.2355/isijinternational.47.479.
10. Telasang, G., Dutta Majumdar, J., Padmanabham, G., Manna, I. (2015). Wear and Corrosion Behavior Laser Surface Engineered AISI H13 Hot Working Tool Steel. Surface and Coatings Technology 261, 69–78.
11. Rad, H.F.; Amadeh, A.; Moradi, H. Wear assessment of plasma nitrided AISI H11 steel. Mater. Des. 2011, 32, 2635–2643