



## Research Article

# Mechanical characterization of pack-boronized AISI 4140 and AISI H13 steels

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## ABSTRACT

Wear losses have a great importance in the world machinery industry. They cause billions of dollars in financial losses every year. Studies on surface treatments are increasing day by day in order to minimize the wear losses of materials. In this study, the pack boronizing process was applied to AISI 4140 and AISI H13 steels, which are frequently used in the manufacturing and molding industry, by using Ekabor II powder at 900 °C and 950 °C for 4 and 6 hours. Microstructural examinations of the samples subjected to metallographic processes were carried out. Afterwards, microhardness measurements were performed by applying 50 gf load for 10 seconds. Wear tests were carried out using pin-on-disk tribotests in a dry environment under 2 N and 5 N loads on the CSM Tribometer device. Wear losses were measured as volumetric loss. Thanks to the boronizing process, surface quality, surface hardness, and wear resistance of both steel materials were increased at a high rate.

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## 1. Introduction

In today's industry, significant investments are made in steel materials which have a wide application area. However, these materials have short service lives due to various wear caused by both mechanical factors and oxidative and corrosive ambient conditions. Material losses caused by wear lead to billions of dollars in financial losses each year in the world machinery industry. Therefore, the problem of wear should be taken into account not only in its technical aspect but also in its negative impact on the economy. In order to prevent these losses and to extend the service life of the steels, an appropriate surface coating process should be applied to these steels in addition to the correct design and appropriate steel selection. The surface coating has layers with high hardness and abrasion resistance by pack-boronizing on metallic surfaces. This thermochemical surface treatment is also known as a surface hardening method, and it has a wide usage area in the industry due to its easy applicability and economy. Boride layers with

high mechanical performance are formed on the surface of steels thanks to boronizing based on the diffusion of boron atoms to the base material surface. It has been reported in various studies that the friction coefficient of the borided surfaces obtained after the boriding process based on the thermal diffusion of boron atoms is very low [1-3]. In addition, thanks to the surface hardness that reaches up to 2000 HV, which it gives to the surface of the material, boronizing is seen as a solution against abrasive or adhesive wear [4-10]. Moreover, even at temperatures well above room temperature (up to 650 °C), the material surface can maintain this high hardness [8]. Boronized steels have high hardness (about 2000 HV) and high wear resistance [7, 11]. The combination of high surface hardness and low friction coefficient of the boron layer obtained on the substrate material surface by boronizing contributes significantly to the fight against wear [12]. AISI 4140 tempered steel is a medium carbon and low alloy steel material, and thanks to its high wear resistance and high toughness and a good hardness-ductility balance, it has a very wide usage area in automotive (e.g.,

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crankshaft, axle shaft, and gear cone, machining processes; machine tools, parts such as rams, spindles, studs, bolts, and nuts) [13, 14]. AISI H13 hot work tool steel, which is frequently used in the diesinking industry, is a high-strength and ductility tool material used especially in extrusion and injection molds of metals, plastic injection molds, hot pressing of copper alloys and steel forging dies [15, 16].

Both steel materials are exposed to high temperature and pressure due to the working environments in which they are used. Therefore, not only the service life will be prolonged by preventing deformation and wear on the surface of these materials, but also the expected high performance from the materials in aggressive working conditions will be achieved. Some studies have focused on improving the wear resistance of hot work tool steels to make them cope with these challenging conditions that they are exposed to [16-20]. The developments in industrial applications in recent years have led to the need for materials with superior properties [21]. Boride layer formed on matrix material surface with boronizing shall also serve as a thermal barrier on the surface of the machine elements operating under high temperature. Thus, direct contact of the material surface with mechanical and tribological factors will be prevented. In this way, boronizing will be an economical alternative method to thermal barrier coating (TBC), which requires a very complex assembly and high-cost initial investment. It has been reported that this thermal barrier coating formed on the surface of the machine elements reduces wear and significantly improves the performance of the machine element and its mechanism [22, 23]. In many studies in the literature, the effect of boronizing on hardness and wear in AISI 4140 and AISI H13 steels has been examined. Ulutan et al. [24] applied the pack boronizing process to AISI 4140 steel by using commercial EKabor2<sup>®</sup> powder for 2, 4, and 6 hours at 900, 950, 1000, and 1050 °C. The results of the study showed that compared to untreated samples, the hardness of boronized AISI 4140 steel increased approximately 6 times, while the abrasive wear resistance increased approximately 3-4 times, and the friction coefficient was very low. Kara et al. [15] came to the conclusion that the hardness of the material increased from 485 HV<sub>0.05</sub> to 1989 HV<sub>0.05</sub> as a result of the pack boronizing process applied to AISI H13 steel at 900 and 950 °C for 2, 4, and 6 hours. Cimenoglu et al. [25] investigated the tribological behavior of the boron layer obtained on AISI 4140 steel by pack boronizing at high temperature. As a result of the pack boronizing process carried out using EKabor2<sup>®</sup> powder for 12 hours at 750, 800, 850, and 900 °C temperatures, they obtained a very good wear resistance in all samples at room temperature; this superior wear resistance obtained in samples boronized at 850 °C and 900 °C and consisting of a double phase boron layer

(Fe<sub>2</sub>B+FeB) was also maintained at 300 °C. Cárdenas et al. [26] applied pack boronizing to AISI H13 tool steel at 1000 °C for 8 hours and examined the wear behavior by using ball-on-disc method. With boronizing, the hardness of AISI H13 steel reached 1803 HV and the friction coefficient decreased from 0.3 to 0.15. While cracks and grinding were observed on the unboronized sample surface after the wear test, only non-significant shear wear marks were observed on the boronized sample surfaces. The shear wear resistance of AISI H13 steel increased 13 times after boronizing.

In this study, pack boronizing was performed on AISI 4140 and AISI H13 steels at 900 °C and 950 °C for 4 and 6 hours. After the boronizing process, the microstructural analyses of the samples were performed and surface roughness, microhardness, and wear strengths were measured. With increased strength and wear resistance, steel materials whose surface and mechanical properties are improved with pack boronizing will provide significant advantages to mold manufacturers and end-users. The most important of these advantages will be the economic gain to be achieved due to the prolongation of the mold and tool life of steel materials. In addition, as an alternative to hot work tool steels used in high-temperature applications, surface quality, hardness, and wear resistance were increased by boronizing, and a boronized AISI 4140 tempered steel with a lower cost was also proposed.

## 2. Experimental Details

### 2.1 Pack Boronizing Process

The chemical compositions of AISI 4140 tempered steel and AISI H13 hot work tool steel used in the study are shown in Table 1. Samples to be boronized were prepared in Ø12x7mm size shaped as discs. Before the boronizing process, the samples were polished, cleaned with ultrasonic bath, and air-dried, then placed in steel boxes filled with Ekabor II powder in a way that they would not contact each other, and covered with boronizing powder again. Before the lid was closed, it was completely filled with SiC powder to prevent boronizing powder from being affected by the air in the oven. Afterwards, the lid of the boxes was closed and the surroundings were plastered with mud to prevent air from getting into them. The pack boronizing process was carried out in a laboratory type ash oven at 900 °C and 950 °C for 4 and 6 hours. After the boronizing process was completed, the steel crucibles taken out from the oven were cooled at room temperature and the samples were removed from the crucible.

Table 1. Chemical composition of the samples (wt.%)

	C	Si	Mn	Cr	Mo	V
AISI H13	0.40	1.00	0.35	5.15	1.40	1.00
AISI 4140	0.38-0.45	0.15-0.40	0.50-0.80	0.90-1.20	0.15-0.30	-

## 2.2 Surface Roughness Measurement

Measurements of the surface roughness were performed with Mitutoyo SJ 301 brand roughness measuring device before and after the boronizing process to determine the effect of the boronizing process on surface roughness. This device was also used to extract wear surface profiles during the volumetric measurement of wear losses.

## 2.3 Characterization of Boride Layers

Before the microstructural analyses, the samples were sanded with 240, 400, 600, 800, 1000, and 1200 grit mesh sanding sheets respectively after molding in the hot bakelite extraction device for ease of grip. It was then polished with 3  $\mu\text{m}$  Struers DiaPro MolB3 and 1  $\mu\text{m}$  Struers DiaPro NapB1 solutions. Polished samples were examined with the NIKON Eclipse LV100 brand optical microscope. In the examinations of the coating layers, the coating layer thicknesses were measured using the Clemex brand image analysis program of the optical microscope.

## 2.4 Surface Roughness Measurement

Microhardness measurements of the samples with polished surfaces were performed by applying 50 gf load for 10 seconds on Future-Tech FM-700 brand device. Hardness measurements were made along a single line from the beginning of the coating to the base metal. Thus, in addition to the coating thickness, the hardness depth was also measured. The optimum parameter was obtained by keeping the samples at 950 °C for 6 hours after the microhardness process.

## 2.5 Wear Tests

In the study, the dry sliding wear tests were performed on the test samples which were pack boronized at 950 °C for 6 hours and unboronized by using CSM Tribometer Device. The dry sliding wear tests were performed in pin-on-disc assembly under dry friction conditions. WC balls with a diameter of 6 mm were used as the counter element. Wear tests were carried out at dry sliding paths of 250 m and 500 m at velocity of 11.77 cm/sec by applying 2 N and 5 N loads. Abrasion losses were calculated as volumetric loss. The profile of the wearing surface was measured with the surface roughness measuring device and volumetric losses were calculated.

## 3. Results and Discussion

### 3.1 Surface Roughness

The surface roughness of the samples was measured before the boronizing process and after 6 hours of boronizing at 950 °C, and the results shown in Table 2 were obtained. When the measurement results in Table 2 are examined, it is seen that the boronizing process has a positive effect on surface roughness. Besides the boronizing process is known to increase surface roughness values in the boronizing of

Table 2. Effect of Boronizing on Surface Roughness

	4140	B-4140	H13	B-H13
Ra ( $\mu\text{m}$ )	1.88	1.59	1.96	1.34

precision machined surfaces, it also causes the boronizing surface quality to increase in materials with higher surface roughness. The results confirmed this situation. Therefore, the use of boronizing in manufacturing will shorten the time of the machining process and thus reduce the production costs.

### 3.2 Characterization of Boride Layers

Microstructural images were taken after etching the polished samples with 4% nital solution. Figure 1(a) shows the microstructural images of AISI 4140 steel boronized at 900 °C for 4 hours. When the boronizing layers are examined, a structure with porous but having a columnar morphology is encountered as in the literature [27]. Since the outermost surface of the coating is damaged due to the polishing process, it seems to be porous. Due to the alloy elements in the chemical component of AISI 4140 material, the boronizing layer has not been able to develop further. Figure 2(a) shows the microstructural images of AISI H13 steel boronized at 900 °C for 4 hours. Since the alloy ratio of AISI H13 material is higher than AISI 4140 material, it is seen that the coating layer development is weaker at the same temperature and time. The growth of the coating did not occur columnar due to the density of the alloy elements in agreement with the literature [28].

When Figure 1(b) is examined, it can be concluded that the boride layer of AISI 4140 steel boronized at 900 °C for 6 hours is porous and continuous, and that FeB phase formation occurs due to the increase in diffusion time outside Fe<sub>2</sub>B phase compared to Figure 1(a). When the microstructural images of AISI H13 steel boronized at 900 °C for 6 hours in Figure 2(b) are compared with Figure 2(a), it is observed that the increased diffusion time causes an increase in the development of the coating, and a nonporous layer formation occurs compared to the 4-hour coating. In addition, FeB phase formation other than Fe<sub>2</sub>B phase occurred with the increase in time. In Figure 1(c), the boride layer of AISI 4140 steel boronized at 950 °C for 4 hours is seen as a partially columnar but porous boride layer, and no significant effect of boronizing temperature on the coating layer has been observed compared to Figure 1(a). Examining Figure 2(c), it is understood that after boronizing process performed in AISI H13 steel at 950 °C for 4 hours, the coating layer does not develop in columnar morphology and the boride layer has a porous structure. When compared with Figure 2(a), it is understood that boronizing temperature does not have a positive effect on boronizing behavior. As seen in Figure 1(d), although the boride layer of AISI 4140 steel boronized at 950 °C for 6 hours developed in the columnar morphology, it was extremely porous. Due to the increase in temperature and duration, a homogeneous

thickness and columnar-developed FeB phase were also encountered. The fact that the FeB phase was homogeneously distributed and had equal thickness caused the wear resistance of the material to increase. If the formation of the FeB phase were to occur in irregular dimensions, this would certainly be an undesirable problem causing a rapid increase in wear losses. However, in this parameter, the columnar development of the layer created a multi-layer coating effect. In Figure 2(d), it is observed that the AISI H13 steel boronized at 950 °C for 6 hours has developed in a homogeneous and porous structure. As a result of the boronizing process, no FeB phase was found.

Boride layer thickness measurement was measured from 5 different points in Clemex brand image analysis program and the results presented in Table 3 were obtained by taking the average values. When Table 3 is examined, it can be seen that the coating layer values obtained with the comments made in the microstructural images confirm each other. It is seen that the increase in the ratio of alloy element in the material has a negative effect on the development of the boride layer. For both materials, the highest boride layer thicknesses were reached by boronizing performed at 950 °C for 6 hours.

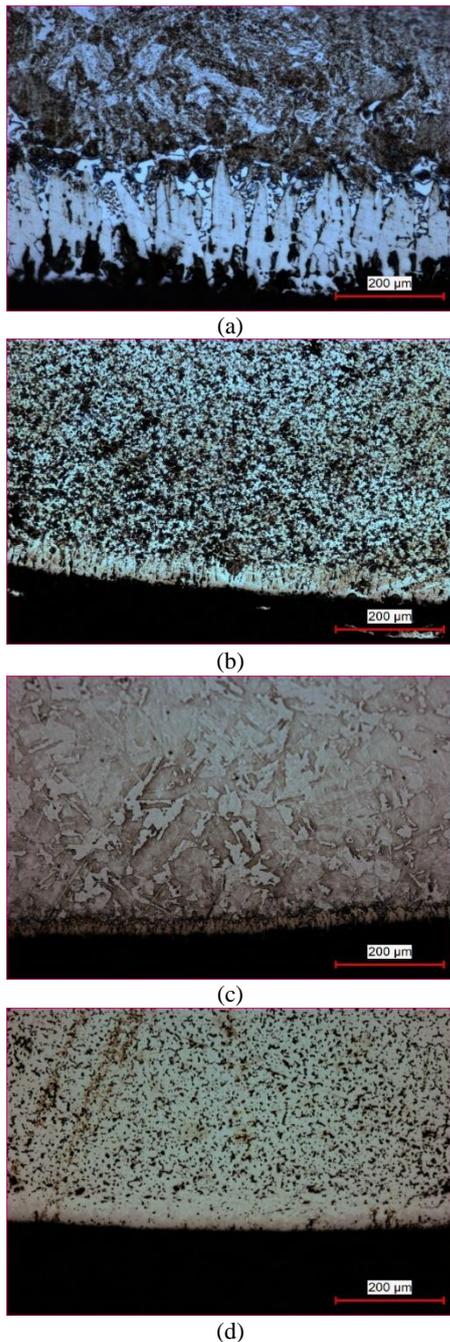


Figure 1. Microstructural images of AISI 4140 boronized a) at 900 °C for 4 hours b) at 900 °C for 6 hours c) at 950 °C for 4 hours and d) at 950 °C for 6 hours

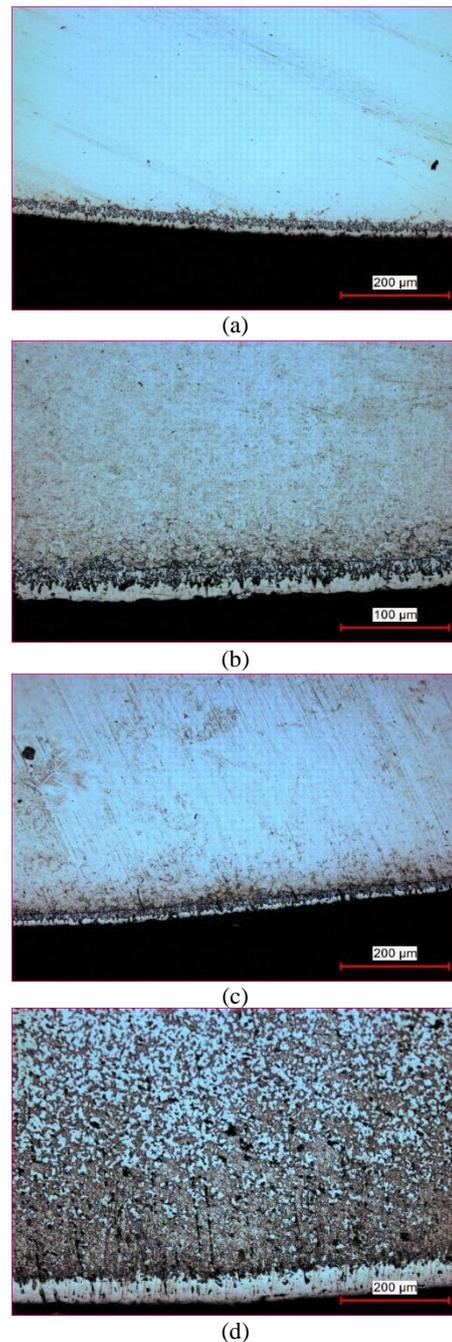


Figure 2. Microstructural images of AISI H13 boronized a) at 900 °C for 4 hours b) at 900 °C for 6 hours c) at 950 °C for 4 hours and d) at 950 °C for 6 hours

Table 3. Boride Layer Measurement Results

	Thickness ( $\mu\text{m}$ )
B-4140-900 °C-4h	40
B-H13-900 °C-4h	35
B-4140-950 °C-4h	60
B-H13-950 °C-4h	35
B-4140-900 °C-6h	40
B-H13-900 °C-6h	40
B-4140-950 °C-6h	80
B-H13-950 °C-6h	60

### 3.3 Microhardness

Microhardness measurements were carried out in such a way that they were closest to the starting point of the coating and the distance between the traces was at least 4 times the trace size. Figure 3 shows the microhardness changes of the boride layer of AISI 4140 material boronized at different temperatures and times. As the boronizing temperature and time increase, both the hardness and hardness depth of the AISI 4140 material (i.e. the diffusion layer) increase. The evaluation made with the microstructural images indicated that the highest hardness values (over 1200 HV) were reached in the parameters in which the FeB phase, which corresponded to the hardness values of the FeB phase, occurred. It is seen that the highest hardness and hardness depth has been reached in samples boronized at 950 °C for 6 hours. Table 3 shows that this corresponds to the highest boride layer.

Figure 4 shows the microhardness change of the boride layer of AISI H13 steel boronized at different temperatures and times. The examination of microstructural images of the boronized AISI H13 steel indicates that the FeB phase

definitely has not occurred. The fact that the highest microhardness value can reach up to 1100 HV is due to the fact that the FeB phase, which significantly increases the hardness in the boride layer, does not occur. Examining Table 3, it is seen that the thickness of the coating layer is in parallel with Figure 4. When Figure 3 and Figure 4 are examined together, it is figured out that the diffusion zone is not deep after passing the boride layer. The main reason for this is the alloy contents of both materials.

### 3.4 Abrasion Resistance

Figure 5 shows the graph of abrasion loss by material under 2N load. It is seen that the unboronized AISI H13 material has suffered from the greatest abrasion loss under 2N load. Abrasion losses of the boronized materials have reduced significantly. Abrasion loss after the boronizing process of the AISI 4140 material has decreased by 75%. It is seen that the decrease in abrasion loss reaches 83% in AISI H13 material. The best wear resistance has been achieved in boronized AISI 4140 material. The examination of microstructural images indicates that the FeB and Fe<sub>2</sub>B phases have caused this. The fact that the abrasion losses do not increase despite the formation of the FeB layer is proof that the FeB layer is connected to the substrate with a good adhesion force.

Figure 6 indicates the abrasion losses of the materials due to the sliding path. AISI H13 suffered the highest abrasion loss at a sliding path of 250m under a 2N load, while boronized materials showed the same abrasion losses. At a sliding path of 500m, boronized AISI 4140 material showed the best wear resistance due to its high surface hardness and the depth of the diffusion layer.

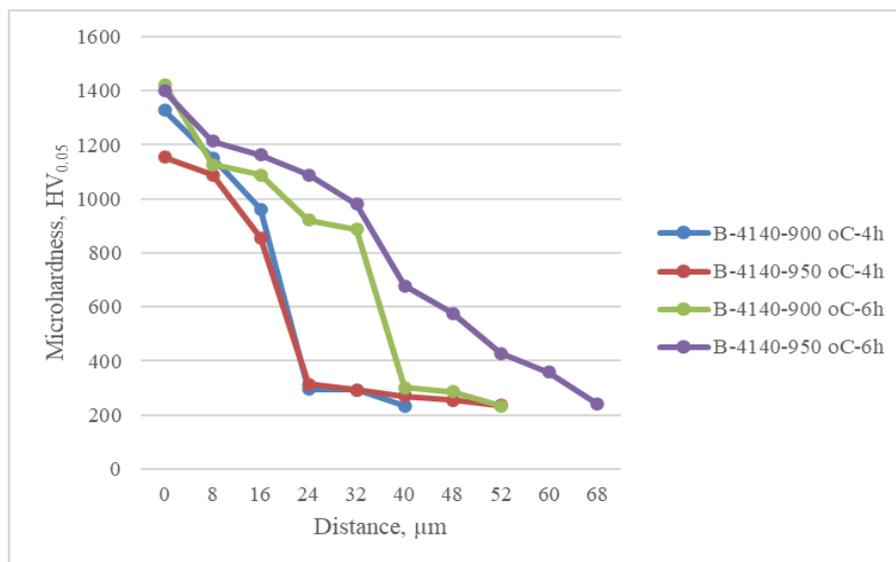


Figure 3. The change in microhardness as a function of depth below the surface of AISI 4140 steel samples boronized at different temperatures and times

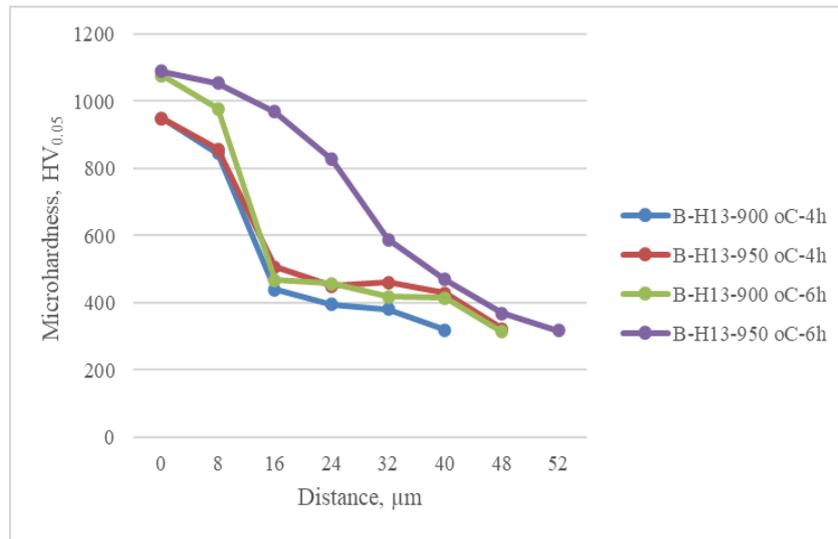


Figure 4. The change in microhardness as a function of depth below the surface of AISI H13 steel samples boronized at different temperatures and times

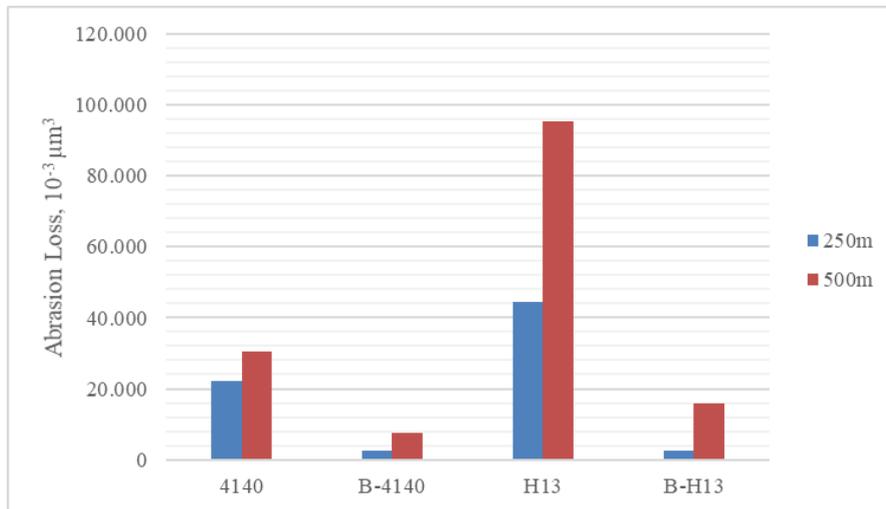


Figure 5. Abrasion losses of samples by material (under 2N load)

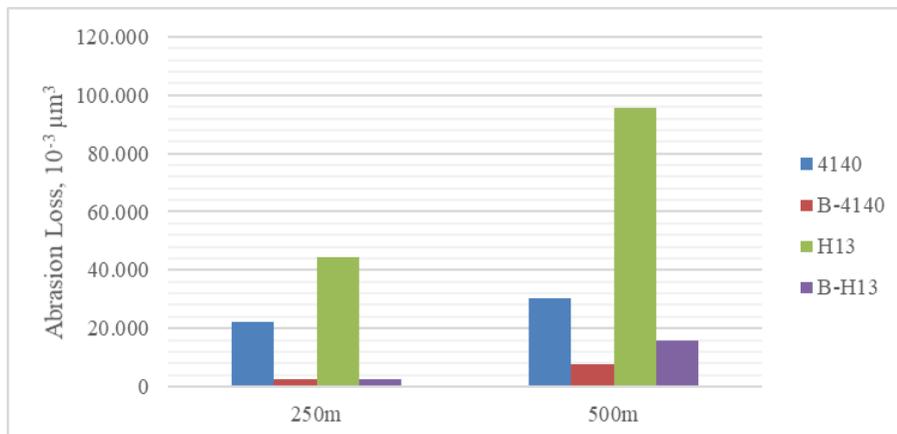


Figure 6. Abrasion losses of samples by sliding path (under 2N load)

Figure 7 shows the abrasion loss graph according to the material under 5N load. It is seen that AISI H13 material,

which has not been boronized, has suffered from the greatest abrasion loss under 5N load. The abrasion loss of

it is nearly 2 times the abrasion loss of the unboronized AISI 4140 material. Abrasion losses of boronized materials have decreased significantly. Abrasion loss of AISI 4140 material decreased by 75% after boronizing. It is seen that the decrease in abrasion loss has reached 73% for AISI H13 material. The best wear value has been achieved in boronized AISI 4140 material. When the microstructural images are examined, it is understood that the FeB and Fe<sub>2</sub>B phases have caused this. The fact that the abrasion losses do not increase despite the formation of the FeB layer is proof that the FeB layer is connected to the substrate with a good adhesion force. Figure 8 shows the materials' adhesion losses depending on the sliding path under 5N load. The unboronized AISI H13 was subjected to the highest abrasion loss at a sliding path of 250m, while the boronized AISI 4140 showed the least abrasion loss.

At a sliding path of 500m, boronized AISI 4140 material showed the best wear resistance due to its high surface hardness and the depth of the diffusion layer. In Figure 9-22, the wearing surfaces of the materials and the wearing surfaces of the counter elements are displayed with images taken by the Stereo Microscope.

These images indicate that the abrasion losses are high on the unboronized samples and adhesions from the unboronized material to the counter element occur. The width of the wear trace is also increasing. It is clearly seen in Figure 13 that there is an adhesive wear especially on the wear of unboronized AISI H13 material. Adhesive wear occurred especially on all unboronized samples. Because it is well known that the boride layer plays a significant role in improving the resistance to abrasion and adhesion [29, 30].

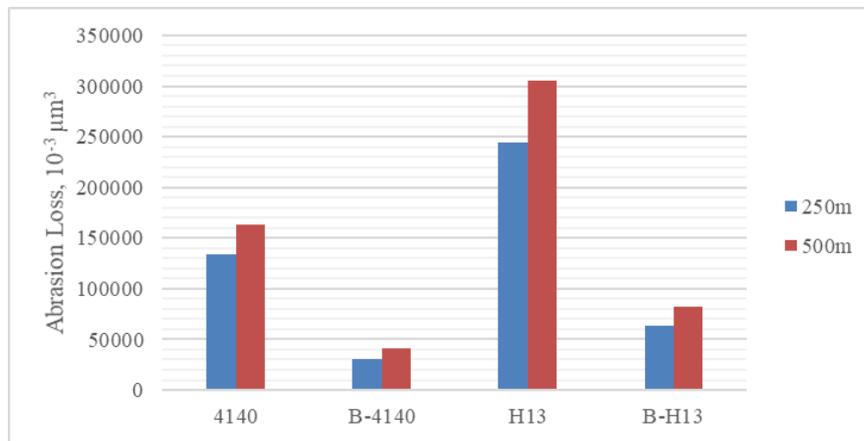


Figure 7. Abrasion loss of samples by material (under 5N load)

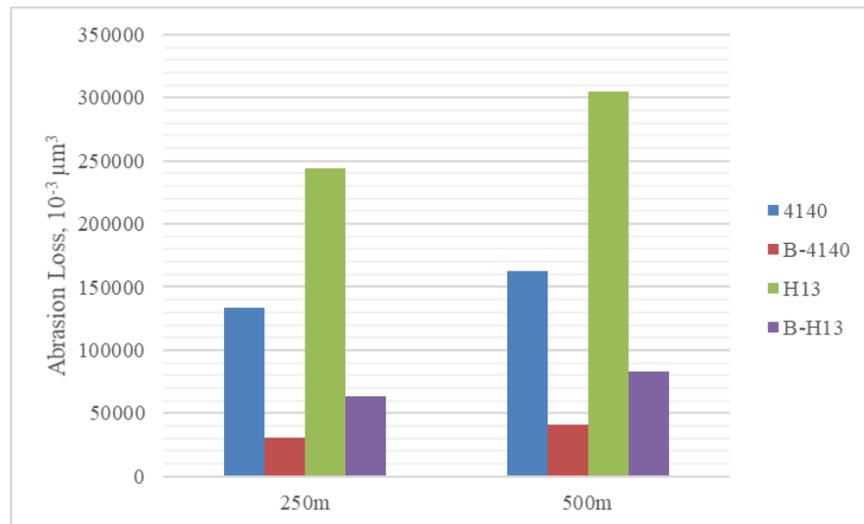


Figure 8. Abrasion loss of samples by sliding path (under 5N load)

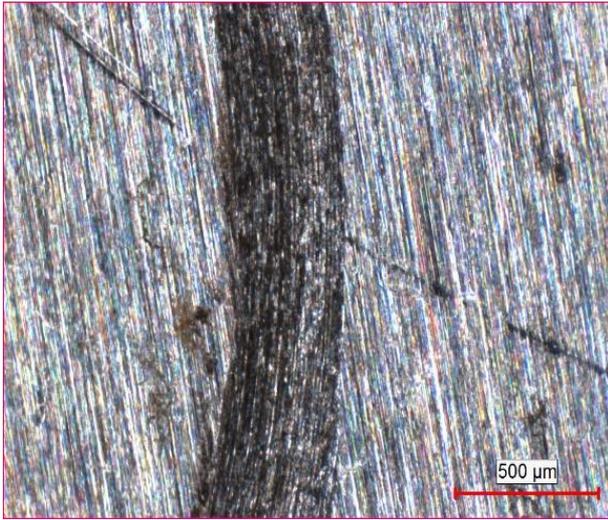


Figure 9. Wearing surface of AISI 4140 material abraded under 2N load

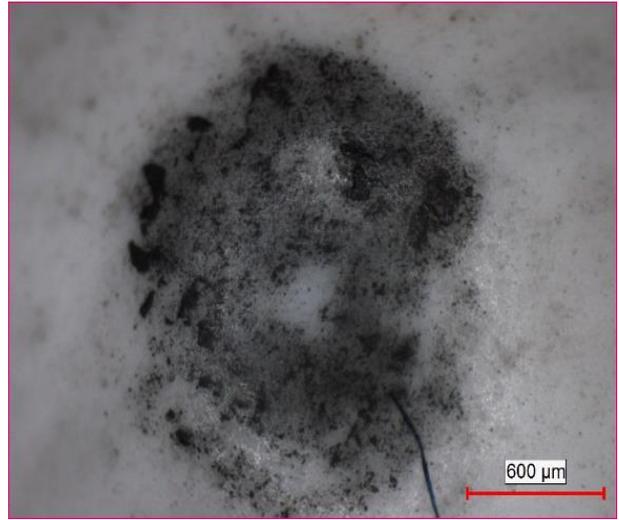


Figure 12. Wearing surface of counter element of boronized AISI 4140 material abraded under 2N load



Figure 10. Wearing surface of counter element of AISI 4140 material abraded under 2N load

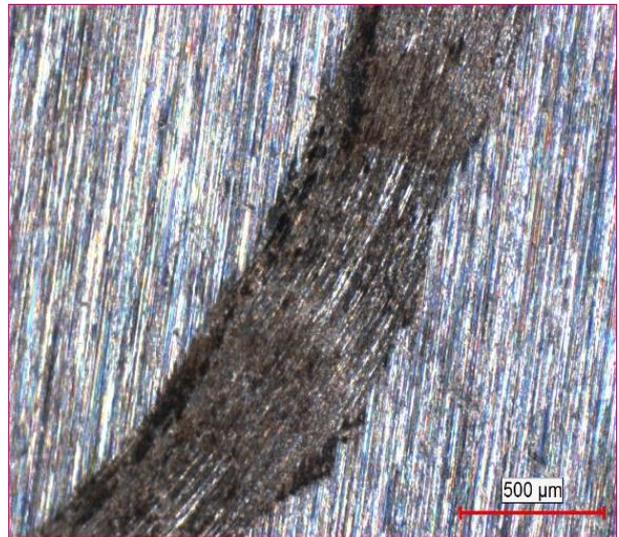


Figure 13. Wearing surface of AISI H13 material abraded under 2N load

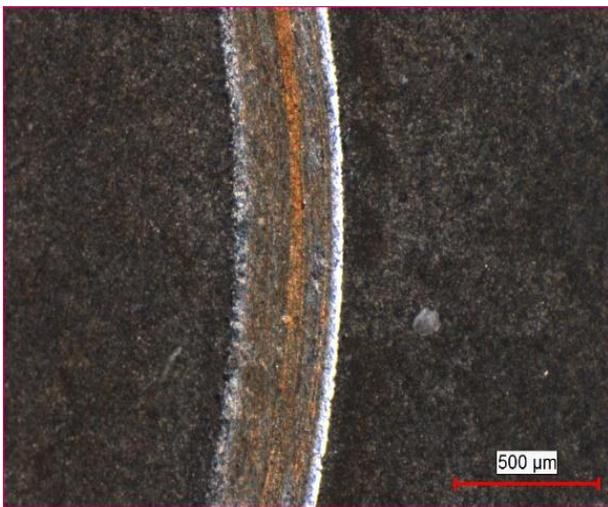


Figure 11. Wearing surface of boronized AISI 4140 material abraded under 2N load

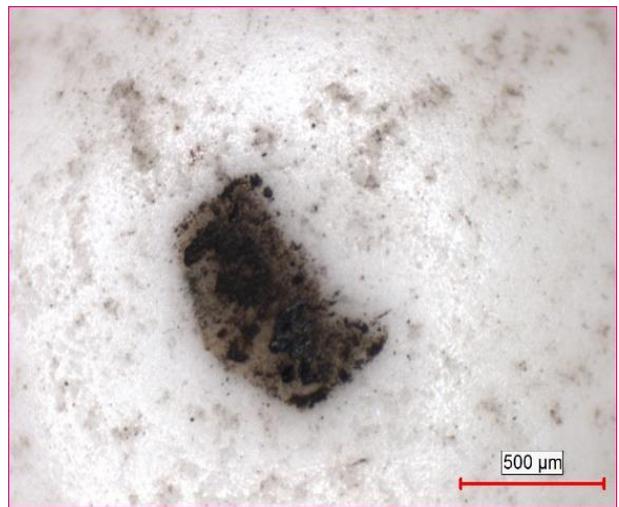


Figure 14. Wearing surface of counter element of AISI H13 material abraded under 2N load

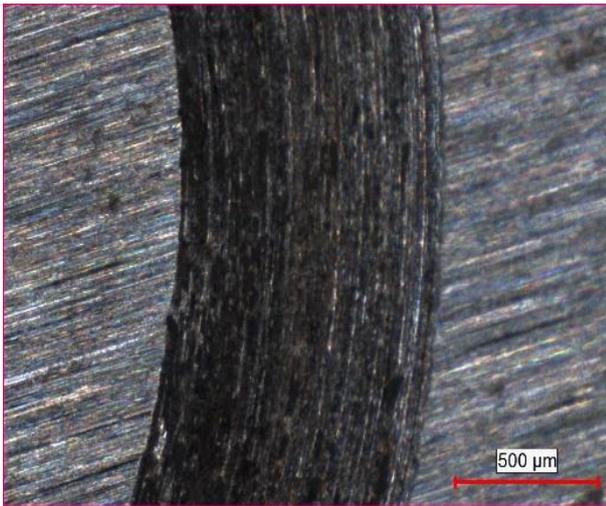


Figure 15. Wearing surface of AISI 4140 material abraded under 5N load

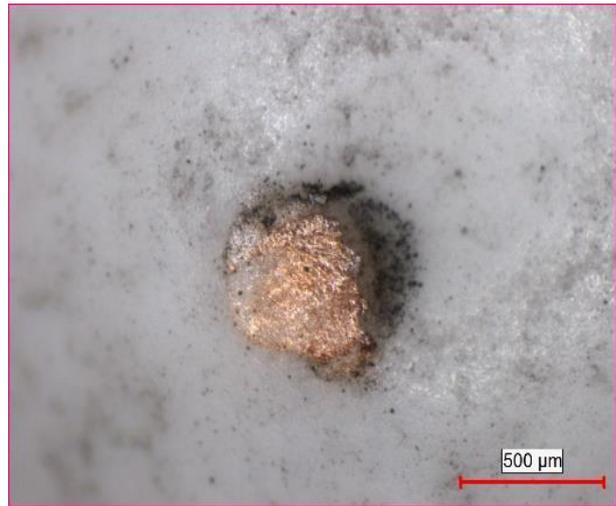


Figure 18. Wearing surface of counter element of boronized AISI 4140 material abraded under 5N load



Figure 16. Wearing surface of counter element of AISI 4140 material abraded under 5N load

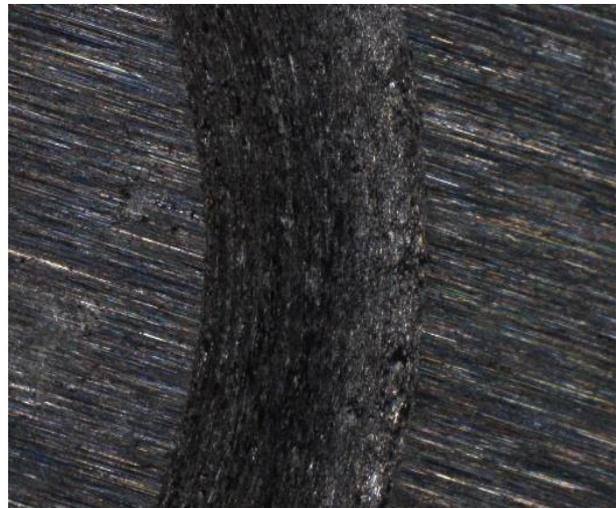


Figure 19. Wearing surface of AISI H13 material abraded under 5N load



Figure 17. Wearing surface of boronized AISI 4140 material abraded under 5N load



Figure 20. Wearing surface of counter element of AISI H13 material abraded under 5N load



Figure 21. Wearing surface of boronized AISI H13 material abraded under 5N load



Figure 22. Wearing surface of counter element of boronized AISI H13 material abraded under 5N load

#### 4. Conclusions

Within the scope of this study, boronizing process was performed on AISI 4140 and AISI H13 steel materials and appropriate boronizing parameter was determined. In order to determine the appropriate boronizing parameter, the materials were subjected to boronizing process at different times and temperatures. Optimum boronizing parameters were reached as a result of microstructural examinations and microhardness tests. Common parameters for both types of materials are determined as 950 °C temperature and 6 hours retention time. Surface roughness, hardness, and wear tests were applied to the samples, which were boronized with these parameters by using the pack boronizing process. The results and recommendations are given below.

- FeB phase was formed on AISI 4140 material boronized for 6 hours at both temperature values. This layer is highly homogeneous and columnar dependent on the substrate material.
- Boride layer thickness of 80 μm was reached in AISI 4140 material and 60 μm in AISI H13 material boronized at 950 °C for 6 hours.
- Microhardness values up to 1400 HV were reached in the process parameters where FeB phase occurred.

- As a result of the wear tests, the material with the lowest wear resistance was determined as AISI H13. However, by applying boronizing process, the properties of this material can be improved by 75%.
- The material with the highest wear resistance was determined as boronized AISI 4140. Thanks to the duplex coating on the surface, the wear resistance was improved up to 70%.
- The comparison of the wear resistance of the two boronized materials indicated that the boronized AISI 4140 material showed 2 times more strength than the boronized AISI H13.

In this study, as an alternative to AISI H13 hot work tool steel, surface quality, hardness, and wear resistance of the samples were increased by using boronizing. Finally, the choice of boronized AISI 4140 tempered steel, which is more cost-effective, is recommended.

#### Declaration

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article. The authors also declared that this article is original, was prepared in accordance with international publication and research ethics, and ethical committee permission or any special permission is not required.

#### Author Contributions

All authors conceived and conducted the study. D. Arslan. performed the experiments, analyzed the data, and wrote the paper. S. Akgün Kayral supervised and improved the study. All authors contributed to manuscript revisions. All authors read and approved the final version of the manuscript.

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