

### **Iournal of New Results in Science**

https://dergipark.org.tr/en/pub/jnrs

Research Article

Open Access



E-ISSN:1304-7981 https://doi.org/10.54187/jnrs.1219301

# Coating of AISI 1018 steel with the pack-boriding method

Ramazan Erarslan 1 , Uğur Öztürk2 , Fikret Yılmaz3

#### Keywords:

AISI 1018 steel, Pack boriding, Wear properties, Hardness **Abstract** — In this study, AISI 1018 steel with low carbon content was coated with the pack-boriding method at 950 °C for 2h and 4h. A two-layer structure was observed in the 2h boronized material: the thin FeB phase and the more prominent Fe<sub>2</sub>B phase. In addition to this two-phase structure, undesirable phases (SiC and Fe<sub>3</sub>O<sub>4</sub>) were detected in the material boronized for 4h. It was observed that the wear resistance and hardness of the boronized materials increased greatly, which is explained by the hard FeB and Fe<sub>2</sub>B phases. In addition, as the heat treatment time increases, the thickness of the FeB phase increases while the thickness of the Fe<sub>2</sub>B phase decreases.

Subject Classification (2020): 74A55, 76R50

### 1. Introduction

Boronizing enriches the material surface with boron atoms through thermochemical diffusion [1-4]. In this process, boron atoms diffuse into the metal matrix on the material surface using thermal energy, forming boride compounds with the atoms of the main material [5]. Compared to traditional surface coating methods, the most crucial advantage of this method is that the coated material shows very high wear and oxidation resistance [6, 7]. Boriding can be carried out in solid, liquid, or gaseous mediums. Pack boriding is the most commonly used boronizing method, which is easier and cheaper than the others [8]. The coated surface can be tetragonal Fe2B and/or orthorhombic FeB depending on the concentration of diffused boron atoms [9]. Although the FeB phase has a higher hardness value than Fe2B, it shows lower toughness [10]. In this respect, the FeB phase, which has a more brittle structure, is undesirable because the coating layer is easily broken when exposed to an external force.

Öztürk et al. [11] obtained a 150  $\mu$ m Fe2B layer in thickness by coating the cylinder liner surface with the boronizing method. Another study found that very good thermal insulation is provided on boronized surfaces, thus improving performance parameters (thermal brake efficiency, brake-specific energy consumption, and fuel consumption) [12]. In addition, it has been observed that coated engines reduce harmful emission values. Türkmen et al. [13] coated SAE 1020 steel for 4, 8, and 12h for 850, 900, and 950 °C temperatures with the pack-boriding method. In that study, boric acid was used as a boron source. Homogeneous, mono-phase, and saw-tooth morphology Fe2B layers were observed in boronized materials. While the hardness value of the boronized layer was 1200-2000 HV0.1, the

<sup>&</sup>lt;sup>1</sup>erarslan60@hotmail.com; <sup>2</sup>ugurozturk@msn.com; <sup>3</sup>fikretyilmaz79@gmail.com (Corresponding Author)

<sup>1.3</sup> Faculty of Arts and Sciences, Department of Physics, Tokat Gaziosmanpaşa University, Tokat, Türkiye

<sup>&</sup>lt;sup>2</sup>Turhal Vocational School of Higher Education, Department of Electricity and Energy, Tokat Gaziosmanpasa University, Tokat, Türkiye

hardness value of the substrate was around 150 HV0.1. Motallebzadeh et al. [14] studied the high-temperature sliding wear characteristics of paste-borided 31CrMoV9 and X40CrMoV5-1 steels. A two-layer (FeB and Fe2B) structure was formed in both alloys. They found that the tribological properties at the test temperature of 500 °C were almost the same as at room temperature. In another study, AISI 316 L steel was coated with powder-pack boriding, and a two-layer (FeB-Fe2B) structure was detected on the surface [15]. These layers increased the tribocorrosion resistance of the steel by 1.5 times.

As can be seen from the studies, the properties, such as the structure, thickness, and hardness of the boron layers formed on the steel surface, are closely related to the process parameters and the steel composition. In addition, knowledge of materials' mechanical and tribological properties is crucial in determining their working conditions [16-18]. There are few studies on coating low-carbon steels by the boriding method in the literature. Among the low-carbon steels, AISI 1018 steel offers a good balance of toughness, strength, and ductility. However, they have low wear, corrosion resistance, and strength compared to high-carbon steels. In this respect, improving the surface properties of AISI 1018 steel is essential in increasing the cycle life.

In this study, low-carbon AISI steel was coated for different durations by the pack-boriding method. Boron compound layers formed on the surfaces were examined using scanning electron microscopy, x-ray diffraction method, hardness test, and wear test. The obtained results were evaluated in terms of the mechanical and tribological properties of the material.

## 2. Experimental

Commercial Ekabor-2 (Bortec) powder was used for coating AISI 1018 steel (Table 1). Firstly, the samples were cut with a diameter of 2 cm and a thickness of 3 mm and polished by metallographic methods up to 1 $\mu$ m. Then, the samples were buried in a 5x5x5 cm steel box filled with Ekabor-2 powder, and Ekrit (Bortec) powder was placed on it as a deoxidant. The samples were heat treated at 950 °C for 2 and 4h. The samples obtained are shown in Figure 1. The boronized products were molded with epoxy and polished by metallographic methods. It was then etched and prepared for Scanning Electron Microscopy (SEM) and hardness analysis. SEM (QUANTA 450 FEG) analyses were obtained in BSE (back scatter electron) mode. Hardness analyzes were performed with a Vickers hardness device (Highwood) under 200 gf force. Wear tests were performed with a pin on the disk set up for a total distance of 600 m at 150 rpm and 30 N loads. X-ray diffraction analysis (PANalytical Emperians) of the samples was performed under CuK $\alpha$  monochromatic beam in the range of  $2\theta$ =10-90°.

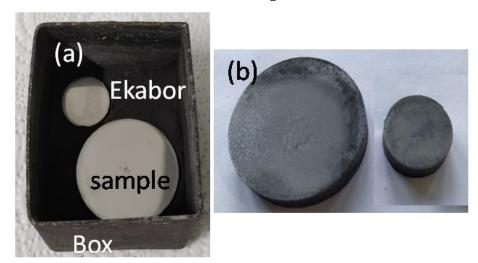


Figure 1. Samples (a) Before boronizing and (b) After boronizing

Sample	С	Fe	Mn	P	S	BiC	SiC	KBF <sub>4</sub>
AISI 1018	0.14-0.20	98.81-99.26	0.60-0.90	< 0.040	< 0.050	-	-	-
Ekabor-2	-	-	-	-	-	5	90	5

**Table 1.** Chemical composition of AISI 1018 steel and Ekabor-2 powder (wt.%)

### 3. Results and Discussion

Figure 2 shows the XRD patterns of untreated and boronized steels. In untreated 1018 steel, all possible diffraction peaks of the Fe (ICSD: 98-018-0969) phase were detected. A two-layered structure was observed in the 2h boronized sample, the Fe<sub>2</sub>B (ICSD: 98-060-3829) phase forming a large part of this structure. Apart from this, a small amount of FeB (ICSD: 98-042-5309), Fe<sub>3</sub>B (ICSD: 98-061-3889), Fe<sub>2</sub>Si (ICSD: 98-010-0094), and Fe<sub>3</sub>O<sub>4</sub> (ICSD: 98-026-3009) phases were detected. A large amount of SiC (ICSD: 98-004-3291) and Fe<sub>3</sub>C (ICSD: 98-001-6593) phases were observed in addition to a small amount of FeB phase in the 4h boronized sample. The XRD method can determine the material's crystal structure from the material surface. The penetration of X-rays on the surface is given by the formula [19],

$$I = I_0 e^{-\mu L}$$

where  $I_0$  is the incident X-ray intensity,  $\mu$  is the linear absorption coefficient, and L is the penetration distance. Since the X-ray intensity decreases exponentially, diffraction will not occur after a certain distance. In this respect, especially in the 4h boronized sample, the Fe<sub>2</sub>B compound could not be detected by XRD because it did not form on the surface. The SiC phase is contained in Ekabor powder and diffused to the surface during heat treatment. SEM images were obtained to see the compounds formed on the surfaces in more detail.

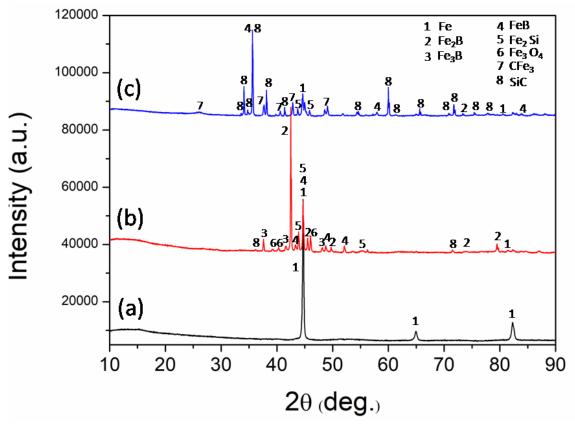


Figure 2. XRD pattern of (a) 1018 steel, (b) 2h boronized, (c) 4h boronized steel

Figure 3 shows the SEM images obtained in BSE mode. A two-phase structure is seen in the material that has been heat treated for 2h (Figure 3b). A clear transition zone was not detected between the boron compound and the base material due to the low C content of the steel used in the study. The transition zone is formed by the repulsion of carbon atoms during the diffusion of boron atoms. A three-layer structure is seen in the material, subjected to 4h heat treatment. When the SEM images and XRD analyses are evaluated together, it is understood that the upper layer is 10μm thick SiC and Fe<sub>3</sub>C. Since these phases are undesirable, they were removed by sanding before hardness and wear analysis. The middle layer is the FeB phase, and its thickness is around 20µm. The bottom layer is the Fe2B phase and has a thickness of 75μm. In addition, the saw-tooth structure of the Fe<sub>2</sub>B phase in the 4h boronized material grew more unevenly compared to the 2h boronized material. As it can be understood from the SEM and XRD analyses, the increase in the heat treatment time caused the formation of undesired phases of the material and decreased the thickness of the Fe<sub>2</sub>B phase. In general, the development of the boride layer increases with increasing time and/or temperature. Therefore, boron diffusion, which initially develops rapidly, slows down and finally stops with the decrease of boron in the powder media. In addition, the previously formed boride layer makes the diffusion of boron atoms difficult. In our case, the fact that the thickness of total boride layers (FeB and Fe<sub>2</sub>B) are nearly the same in both samples indicated that the boron diffusion had taken place completely. On the other hand, the thickness of the FeB layer increases with increasing time while the thickness of Fe<sub>2</sub>B decreases. This can be interpreted that increasing boronized time negatively affects the formation of the Fe<sub>2</sub>B phase.

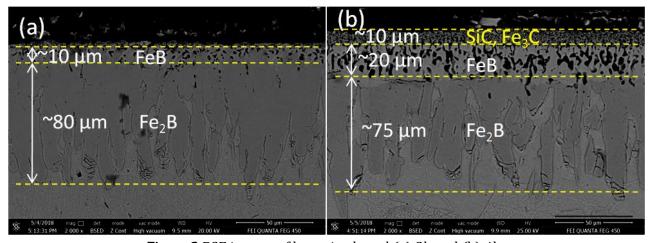


Figure 3 BSE images of boronized steel (a) 2h and (b) 4h

Figure 4 shows the mass loss plot of boronized samples and untreated steel for different distances. As seen in Figure 4, the mass loss of untreated steel increased up to about 0.16 grams for a sliding distance of 600 m. Mass loss in boronized samples is quite low, 0.002, and 0.004 g for 2h and 4h heat-treated samples, respectively. The results show that the wear resistance of the boronized samples is significantly increased. This can be explained by the hard boron compounds, FeB and Fe<sub>2</sub>B, in agreement with the literature [20]. High wear resistance is attributed to the boride layer's saw-tooth and columnar morphology. This structure improves the bonding strength with a substrate. The higher mass loss of the 4h sample compared to the 2h sample can be explained by the more formation of brittle FeB phase formed on its surface. In addition, since the thermal expansion coefficient of FeB is higher than that of Fe<sub>2</sub>B, cracks and fractures may occur easily under mechanical stress. Therefore, forming a single-phase Fe2B layer in boride coatings is desirable.

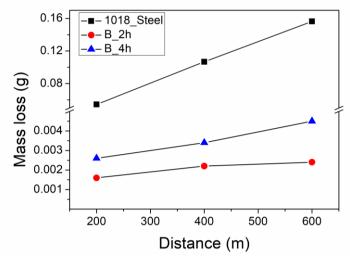
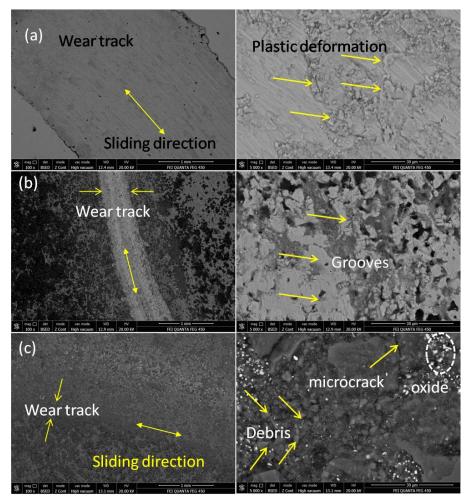


Figure 4. Mass losses of the samples for different sliding distances

Figure 5 shows the worn surface of the samples. Plastic deformation appears to predominate in untreated steel. The ductile nature of untreated steel can explain this. It is seen that grooves are dominant in the 2h boronized sample due to the hard and tough  $Fe_2B$  phase formed on the surface. On the other hand, in the 4h boronized sample, it is seen that microcracks are formed on the surface, as well as debris and oxides. This is due to the fact that the brittle FeB phase and an oxide layer formed on the surface were more dominant in the 4h boronized sample.



**Figure 5.** Worn surface of the samples (a) Untreated steel, (b) 2hboronized, (c) 4hboronized (100x and 5000x magnification)

Figure 6 shows the Vickers hardness values of the samples. It was determined that the hardness values of the boronized samples were approximately two times higher than those of the untreated steel, in agreement with the literature [21]. In addition, it was observed that the hardness of the 2h boronized sample was slightly higher than the 4h boronized sample. The results obtained can be explained by the fact that the  $Fe_2B$  phase is more dominant in the 2hboronized sample, in agreement with the wear tests and SEM images.

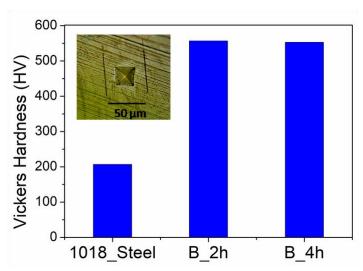


Figure 6. Vickers hardness of the samples. The inset picture shows the imprint image after indentation

## 4. Conclusions

In this study, AISI 1018 steel was coated with the pack boronization method for 2 and 4hours. The results obtained are summarized below:

- From the XRD analysis, a two-phase structure was formed in the 2hboronized material (FeB and Fe<sub>2</sub>B), while undesirable phases were detected from the surface of the 4hboronized material (SiC and Fe<sub>3</sub>O<sub>4</sub>).
- ➤ Multilayer structures were observed in SEM analysis, consistent with XRD. In addition, it was observed that the thickness of the FeB layer increased while the thickness of the Fe₂B layer decreased in the 4h boronized material.
- From the wear tests, it was determined that the wear resistance of boronized materials was greatly increased. While the plastic deformation mechanism dominates untreated steel, grooves dominate in 2h boronized material. In the 4h boronized material, debris and microcracks are evident, which is explained by the more fragile FeB and oxide phases.
- ➤ The hardness values of the boronized samples are approximately 2 times that of the untreated sample due to the hard boron-iron compounds.
- ➤ According to experimental studies, 2h boronized steel exhibited very promising results.

In this study, we obtained very promising results in the field of pack boriding. In future studies, we intend to investigate the effects of the mixture of Ekabor-2 and different powders, such as iron, cobalt, titanium, etc., on forming the boron layer.

## **Author Contributions**

All the authors equally contributed to this work. They all read and approved the final version of the paper.

## **Conflicts of Interest**

All the authors declare no conflict of interest.

## Acknowledgment

This study was supported by Tokat Gaziosmanpaşa University Scientific Research Projects Commission Grand number: 2018/13. The authors would like to thank Dr. Cengiz TEMİZ for SEM and XRD analysis.

#### References

- [1] A. Erdoğan, *Investigation of high-temperature dry sliding behavior of borided H13 hotwork tool steel with nanoboron powder*, Surface and Coatings Technology 357 (2019) 886-895.
- [2] M. Usta, I. Ozbek, M. Ipek, C. Bindal, A. H. Ucisik, *The characterization of borided pure tungsten*, Surface and Coatings Technology 194 (2-3) (2005) 330-334.
- [3] E. Ekmekçiler, A. Polat, M. Usta, *Hard boride coating on iron aluminide (FeAl)*, Surface and Coatings Technology, 202 (24) (2008) 6011-6015.
- [4] I. Gunes, Y. Kayali, *Investigation of mechanical properties of borided Nickel 201 alloy*, Materials and Design 53 (2014) 577-580.
- [5] T. Balusamy, T. S. N. Sankara Narayanan, K. Ravichandran, Il Song Park, M. H. Lee, *Effect of surface mechanical attrition treatment (SMAT) on pack boronizing of AISI304 stainless steel*, Surface and Coatings Technology 232 (2013) 60–67.
- [6] M. Kulka, N. Makuch, A. Pertek, *Microstructure and properties of laser borided 41Cr4 steel,* Optics and Laser Technology 45 (2013) 308–318.
- [7] H. Yang, X. Wu, G. Cao, Z. Yang, *Enhanced boronizing kinetics and high temperature wear resistance of H13 steel with boriding treatment assisted by air blasts hot peening*, Surface and Coatings Technology, 307 (Part A) (2016) 506–516.
- [8] O. Ozdemir, M. A. Omar, M. Usta, S. Zeytin, C. Bindal, A. H. Ucisik, *An investigation on boriding kinetics of AISI 316 stainless steel*, Vacuum, 83 (1) (2009) 175–179.
- [9] I. Campos-Silva, M. Ortiz-Dominguez, N. Lopez-Perrusquia, A. Meneses-Amador, R. Escobar-Galindo, J. Martinez-Trinidad, *Characterization of AISI 4140 borided steels*, Applied Surface Science 256 (8) (2010) 2372–2379.
- [10] A.K. Sinha, Metals Handbook-Heat Treating-Boriding (boronizing) of Steels, Vol. 4 (ASM International, Materials Park, 1991) pp. 978–999.
- [11] U. Öztürk, H. Hazar, Y. S. Arı, *Investigation of using pumpkin seed oil methyl ester as a fuel in a boron coated diesel engine*, Energy 186 (2019) 115871.
- [12] U. Öztürk, H. Hazar, F. Yılmaz, *Comparative performance and emission characteristics of peanut seed oil methyl ester (PSME) on a thermal isolated diesel engine*, Energy 167 (2019) 260-268.
- [13] İ. Türkmen, E. Yalamaç, M. Keddam, *Investigation of tribological behaviour and diffusion model of Fe<sub>2</sub>B layer formed by pack-boriding on SAE 1020 steel*, Surface and Coatings Technology 377 (2019) 124888.

- [14] A. Motallebzade, E. Dilektasli, M. Baydogan, E. Atar, H. Cimenoglu, *Evaluation of the effect of boride layer structure on the high temperature wear behavior of borided steels*, Wear 328-329 (2015) 110-114.
- [15] I. C. Silva, M.P. Pardave, R. P. P. Borja, O. F. Kahvecioğlu, D. B. Barcenas, C. L. Garcia, R. R. Helguera, *Tribocorrosion and cytotoxicity of FeB-Fe<sub>2</sub>B layers on AISI 316 L steel*, Surface and Coating Technology 349 (2018) 986-997.
- [16] S. Ergen, *The effect of indentation temperature and load on mechanical characterization and transformation behavior of high temperature Ti-V-Al-Cu shape memory alloy,* Materials Chemistry and Physics 268 (2021) 124757.
- [17] O. Uzun, F. Yılmaz, S. Ergen, C. Emeksiz, U. Kölemen, *Correlation of hardness and silicon morphology for Al-Si-Sb alloy*, Archives of Metallurgy and Materials 63 (1) (2018) 467-472.
- [18] S. Ergen, F. Yılmaz, S. Gül, U. Kölemen, M. F. Kılıçaslan, O. Uzun, *Structural and mechanical properties of hypereutectic AlSiFe powders and a new method for determination of sintering temperature,* Advanced Powder Technology 29 (3) (2018) 563-572.
- [19] J. Liu, R. E. Saw, Y. H., Kiang, *Calculation of effective penetration depth in X-Ray diffraction for pharmaceutical solids*, Journal of Pharmaceutical Sciences 99 (9) (2010) 3807-3814.
- [20] R. C. Espinoza, U. F. Lopez, J. M. Trinida, I. C. Silva, E. H. Sanchez, A. Motallebzadeh, *Tribological behavior of borided AISI 1018 steel under linear reciprocating sliding conditions*, Wear 362-363 (2016) 1-7.
- [21] E. J. Hernández-Ramírez, A. Guevara-Morales, U. Figueroa-López, I. Campos-Silva, *Wear resistance of diffusion annealed borided AISI 1018 steel,* Materials Letters 277 (2020) 128297.