OKU Fen Bilimleri Enstitüsü Dergisi
6(2): 1175-1186, 2023

OKU Journal of The Institute of Science and Technology, 6(2): 1175-1186, 2023

Osmaniye Korkut Ata Üniversitesi
Fen Bilimleri Enstitüsü
Dergisi

Osmaniye Korkut Ata University
Journal of The Institute of Science
and Technology

Borlanmış AISI M2 Yüksek Hız Çeliğinde Tek Fazlı (Fe_2B) Borür Tabakası Oluşumu

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Araştırma Makalesi

Makale Tarihçesi:
Geliş tarihi: 27.04.2022
Kabul tarihi:14.11.2022
Online Yayınlanma: 05.07.2023

Anahtar Kelimeler:
AISI M2
Kutu borlama
Ekabor 2
Borür tabakası
Fe_2B fazı

ÖZ

Bu çalışmada, yüksek hız çeliği sınıfında yer alan AISI M2'nin kutu-borlama yöntemi ile borlanma işlemi gerçekleştirilmiştir. 20x20x15 mm boyutundaki numuneler, atmosfer kontrollü fırında, 900, 1000 ve 1100°C'de, 2,4ve 6 saat süre ile borlanmışlardır. Borlama ısıl işlemi için AISI 316 paslanmaz çelik pota ve borlama maddesi olarak ticari ismi EKABOR 2 olan toz karışımı kullanılmıştır. Borlama işlemi tamamlanan numuneler daha sonra metalografik etüt için hazırlanmıştır. Mikroyapı incelemeleri için, geri saçılan elektronlar yardımıyla taramalı elektron mikroskobu (SEM), kimyasal analiz için ise SEM'e entegre EDX (Energy Dispersive X-Ray Analysis) ünitesi ve ayrıca XRD (X-ray diffraction) cihazı kullanılmıştır. Mikroyapı ve kimyasal analiz işlemleri sonrasında, borlanmış numunelerin yüzeylerinde oluşan borür tabakalarının, Vickers sertlik cihazı ile, 100 gr'lık ağırlık kullanılarak sertlikleri ölçülmüştür. Gerçekleştirilen analizler neticesinde, AISI M2 yüksek hız çeliğinin borlanabildiği belirlenmiş, yüzeyinde oluşan borür tabakasının, pek çok çelik türünün aksine tek fazlı olduğu ve bu fazın Fe_2B olduğu belirlenmiştir. Ayrıca elde edilen sonuçlar literatür ile kıyaslanarak sonuçların doğruluğu teyit edilmiştir.

Single Phase (Fe_2B) Boride Layer Formation on Borided AISI M2 High-Speed Steel

Research Article

Article History:
Received: 27.04.2022
Accepted: 14.11.2022
Published online: 05.07.2023

Keywords:
AISI M2
Pack Boriding
Ekabor 2
Boride Layer
Fe_2B phase

ABSTRACT

In this study, the boriding process of AISI M2, which is in the high-speed steel class, was carried out with the pack-boriding method. Samples of 20x20x15 mm were borided in an atmosphere-controlled furnace at 900, 1000, and 1100°C for 2,4, and 6 hours. For the boriding heat treatment, AISI 316 stainless steel crucible and a powder mixture with the trade name EKABOR 2 as boa riding agent were used. After the boriding process was completed, the samples were prepared for the metallographic study. Scanning electron microscope (SEM) with the help of backscattered electrons (BE) was used for microstructure investigations, and EDX (Energy Dispersive X-Ray Analysis) unit integrated into SEM and also XRD (X-ray diffraction) device were used for chemical analysis. After the microstructure and chemical analysis processes, hardness measurements were carried out on the boride layers formed on the surface of the samples. After the microstructure and chemical analysis processes, the hardness of the boride layers formed on the surfaces of the boronized samples was measured with a Vickers hardness device, using a 100 g. weight.As a result of the analyzes carried out, it was determined that AISI M2 high-speed steel can be borided, and it was determined that the boride layer formed on the surface was single-phase, unlike many steel types, and this phase was Fe_2B. In addition, the results obtained were compared with the literature and the accuracy of the results was confirmed.

To Cite: Aydoğmuş T., Çiçek B., Topuz P., Aydın Ö. Single Phase (Fe_2B) Boride Layer Formation on Borided AISI M2 High-Speed Steel. Osmaniye Korkut Ata Üniversitesi Fen Bilimleri Enstitüsü Dergisi 2023; 6(2): 1175-1186.

1. Introduction

Boron is a light element obtained from compounds (Tinkal, Colemanite, Kernit, Ulexite, Pandermite, Borasite, etc.) extracted underground in the form of minerals. Boron is a harder element in terms of hardness as well as electrical conductivity than carbon (Adair, 2007). Because of these properties are used in many fields such as energy production, chemistry, and materials science (Yinghuai, 2022).

Boriding is a type of heat treatment applied to metals, their alloys, or non-metallic materials by diffusion methods to improve their surface properties (Tsipas and Tsipas, 2015). Boride layers formed on the surfaces of borided materials have particularly high hardness and wear resistance. Since they can form phases with many elements, they can also form polyphase boride structures on the surface of materials, depending on the composition of the material to which they are applied (Topuz, 2010). The boriding process, pack-boriding, paste boriding, plasma boriding, fluidized bed boriding, etc. can be applied in many ways (Sinha, 1999). The pack boriding method is based on the principle that the material embedded in the boriding agent is kept in the heat treatment furnace at the appropriate temperature and time using a heat treatment crucible and then cooling to room temperature. A schematic representation of the pack-boriding method is shown in Figure 1 below.

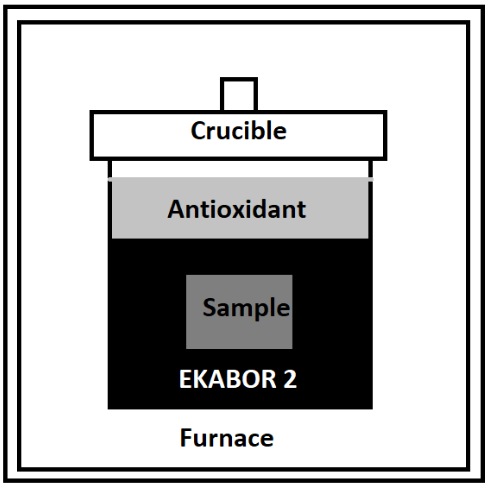


Figure 1. Schematic representation of the pack-boriding method

Today, the most commonly borided metallic materials are steels. There are many types of steel, and the alloying elements in it also affect the boriding process. But, as a result of the boriding process, a single-phase Fe_2B or a double-phased boride layer consisting of both FeB and Fe_2B is formed on their surfaces (Keddam and Chentouf, 2005). A schematic representation of boride layer formation is shown in Figure 2 below.

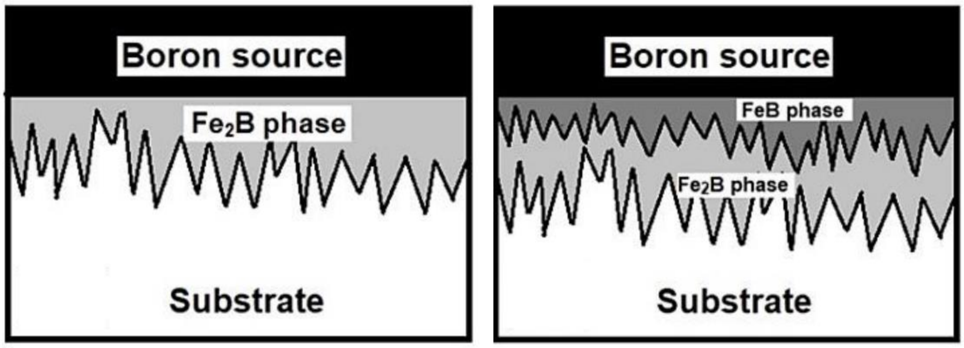


Figure 2. Schematic representation of single and double-layer boride phases (Velázquez et al., 2019)

The reason why the boride layer is single-phased or double-phased is related to the type and ratio of other alloying elements in the steel. The layer formed on the steel surface should be single-phased, because, in double-phased layers, undesirable situations such as cracking or separation between the phases may occur. The main reason for this is the tensile stress between these two phases because the coefficients of thermal expansion are different from each other (Sinha, 1999; Jain and Sundararajan, 2002). To prevent this situation, either the boriding temperature and time should be brought to optimal values or slow cooling treatment should be applied to the material after the boriding process (Dossett et al., 2013). The boride layers obtained by boriding of various steel types are given in Table 1 below.

Table 1. Boride layer formation of various steel types

Material	**Boriding method**	**Temperature range (K)**	**Layer type**	**References**
AISI D2 steel	Pack	1223-1273	Double layer	(Keddam and Kulka, 2020)
AISI 316 steel	Pack	1123-1273	Double layer	(Campos et al., 2010)
AISI M2 steel	Pack	1173- 1323	Double layer	(Zouzou and Keddam, 2019)
Mild steel	Spark Plasma Sintering (SPS)	973–1273	Double layer	(Yu et al., 2002)
AISI D3 **AISI S1** **AISI 6F7** **AISI O2**	Pack	1123–1323	Double layer	(Topuz et al., 2014)
AISI 1080	Pack	973-1373	Single-layer	(Mandiang et al., 2013)
AISI M2	Paste	1223 – 1273	Single-layer	(Campos et al., 2007)
AISI M2	Ion implantation	873 – 1173	Single-layer	(Davis et al., 1998)

This study, it is aimed to determine whether a single-phased or double-phased layer will be formed on the surface of AISI M2 high-speed steel as a result of boriding treatment and to compare it with similar studies in the literature.

2. Experimental

The chemical analysis of AISI M2 high-speed steel of which used for the boriding treatments is shown in Table 2 below.

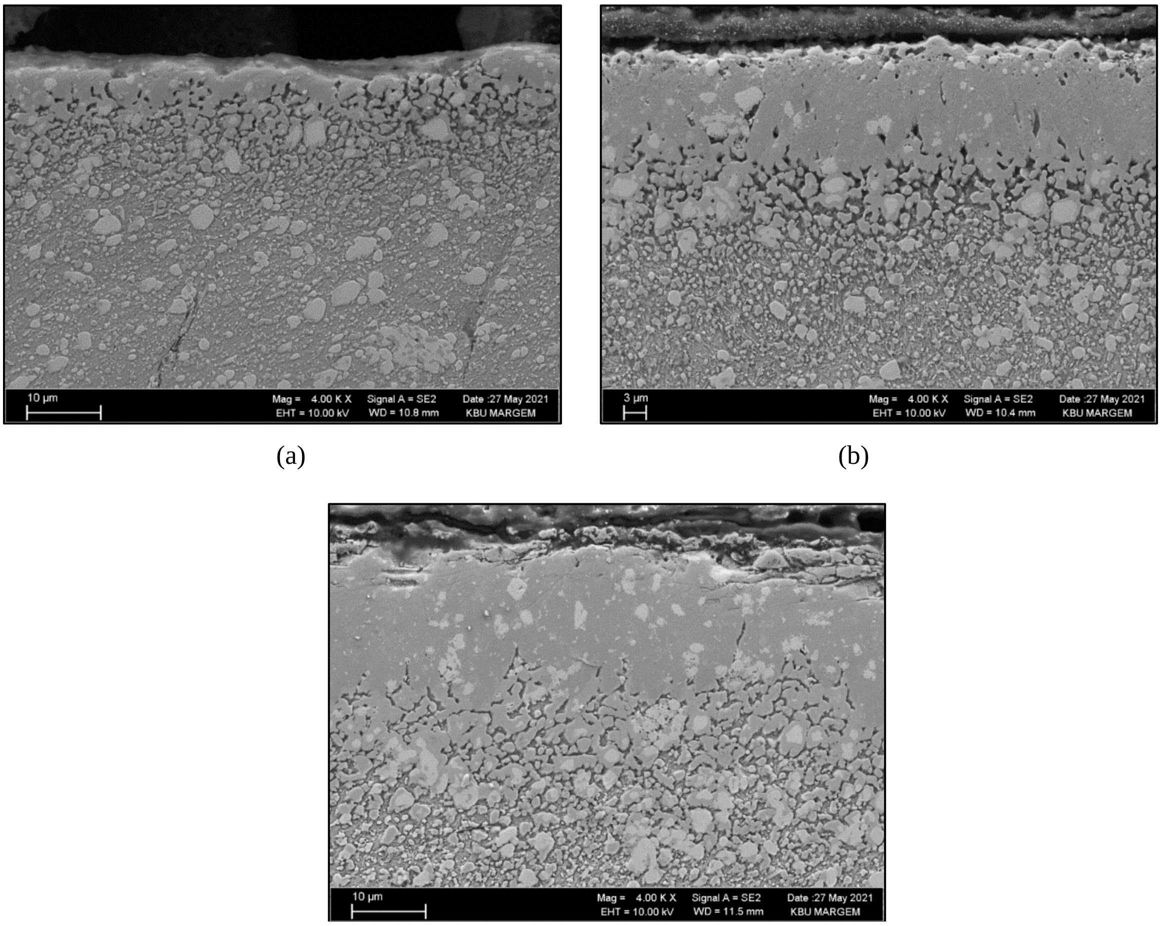
Table 2. Chemical composition of AISI M2 steel

Borided Material	Alloying Elements (wt.%)			
AISI M2	C	Mn	Si	Mo
	0.89	0.18	0.02	4.99
	Cr	V	W	Al
	4.12	1.95	6.58	0.02

20x20x15 mm sized samples were prepared for boriding processes carried out in an atmosphere-controlled heat treatment furnace. The boriding experiments were carried out at 900, 1000, and 1100 °C for 2,4, and 6 hours, and a powder mixture with the trade name Ekabor 2 was used as a boriding agent. In the literature, it is stated that this boriding agent is composed of 5 wt.% B4C, 5 wt.% KBF4, and 90 wt.% SiC (Sinha, 1999). After the boriding processes were completed, the samples were then metallographically prepared. The samples were sanded using 80 to 1200 mesh sandpapers, then polished with a 1 μm grain size alumina paste. Nital 4 (vol.% 95 HNO_3 + vol. % 5 ethyl alcohol) reagent was used for the etching processes of the samples of which the polishing process was completed. By Carl Zeiss Ultra Plus branded Scanning Electron Microscope (SEM) was used for the microstructure investigations of the samples of which metallographic preparation processes were completed. While performing SEM microstructure investigations, point analyzes were also carried out to determine the elements on the boride layer of the samples using the EDX unit integrated into the SEM. In addition, XRD analysis of the borided sample was carried out at 1100 °C for 6 hours to determine the phases forming the boride layer. Rigaku Ultima IV branded device was used for XRD analysis. Finally, the hardness values of the boride layer formed on the surface of the samples, of which microstructure and chemical analyzes were completed, were measured with the Qness Q10 A+ Vickers hardness device using 100 g. weight.

3. Results and Discussion

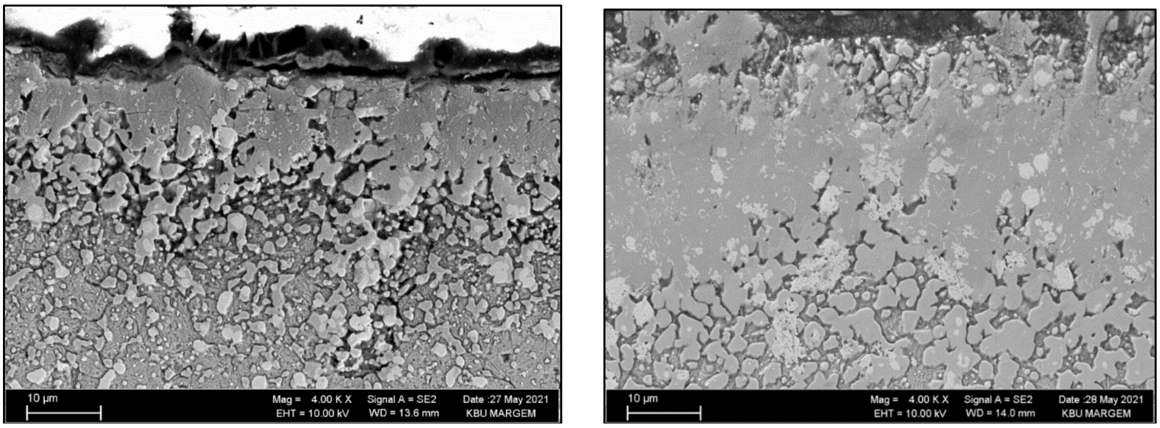
As a result of boriding experiments, it was understood from the images obtained by using backscattered electrons by SEM and also from the EDX and XRD analysis, that the boride layer has a single-phase (Fe_2B) structure. SEM (BE) images of the samples are shown in Figures 3 to 5 below separately.



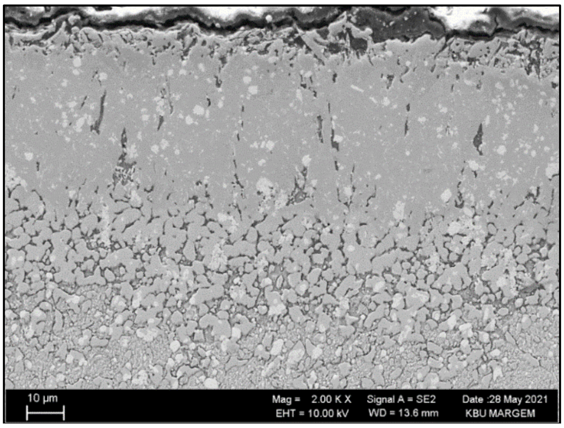
(a) (b)

(c)

Figure 3. SEM (BE) image of AISI M2 borided at 900 C. a) 2h, b) 4h, c) 6h

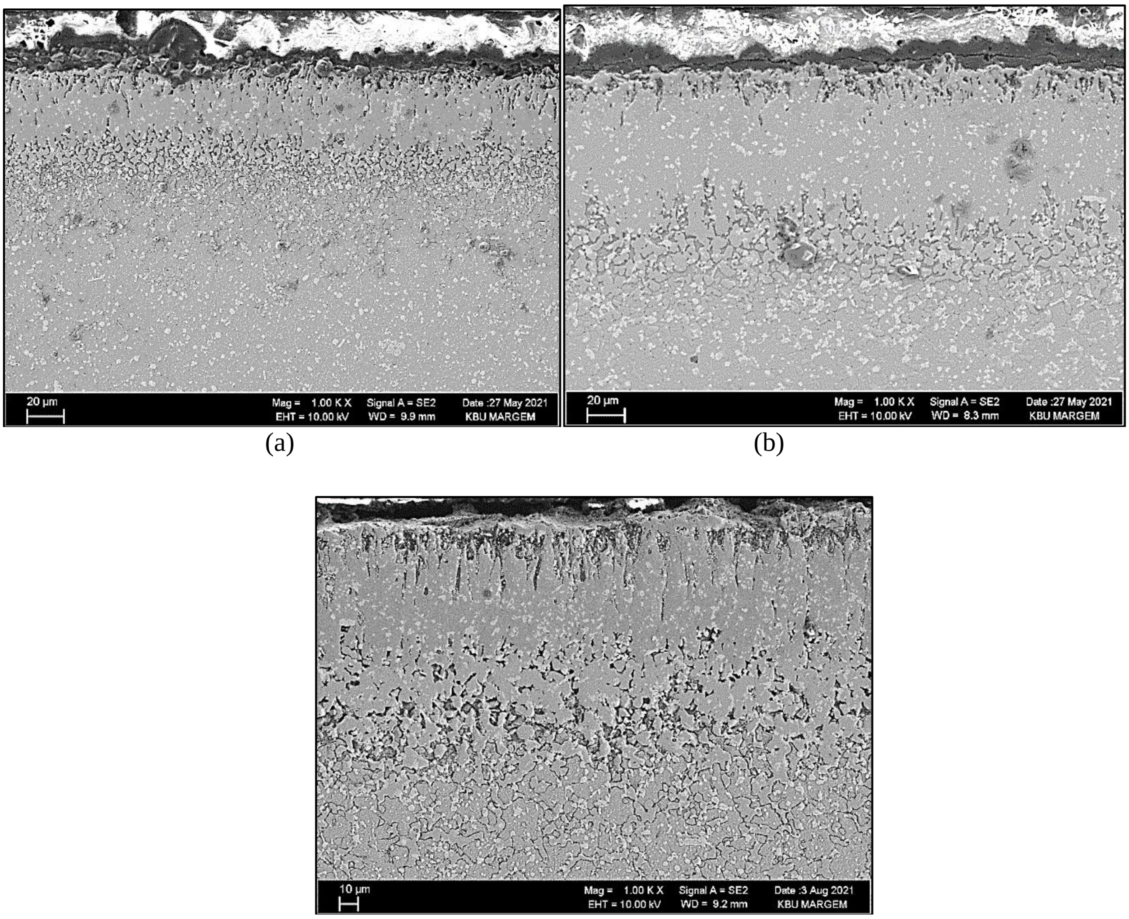


(a) (b)



(c)

Figure 4. SEM (BE) image of AISI M2 borided at 1000 C. a) 2h, b) 4h, c) 6h



(a) (b)

(c)

Figure 5. SEM (BE) image of AISI M2 borided at 1100 C. a) 2h, b) 4h, c) 6h

As can be seen from the images obtained with the help of backscattered electrons, the boride layers consist of a single layer. In addition, as a result of microstructural examinations, it was determined that the boride layer was in the form of a short sawtooth morphology. To show the formation of the single-phase layer more clearly, thermal image analysis was performed, and thus the approximate boundaries between the boride layer, the transition zone, and the matrix were determined. On the thermal image, the boride layer can be distinguished from each other by the light blue region, the transition region by the coarse-grained green region, and the matrix by the green region with much smaller grains than the transition zone. The thermal image of the borided AISI M2 is shown in Figure 6 below.

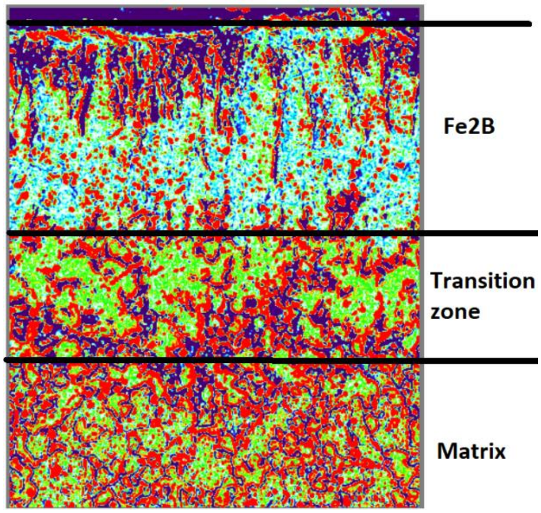
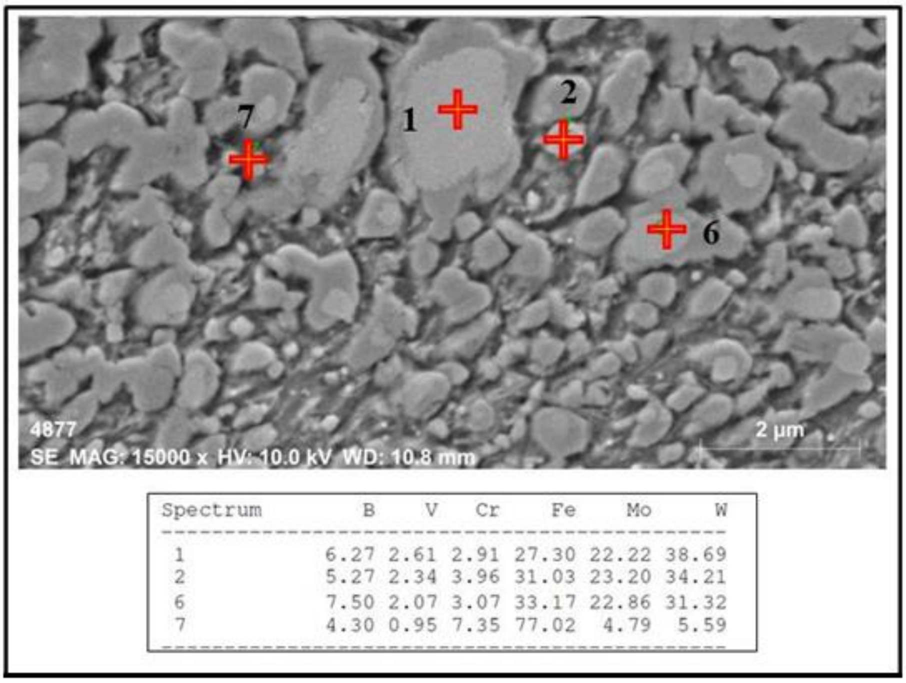


Figure 6. Thermal image of the borided AISI M2

As a result of the point EDX analysis performed on the images during SEM examinations, it was determined the ratio of boron element was between 4.3 wt.% and 6.27 wt.% and these values were compatible with the ratio of the Fe_2B phase on the iron - boron equilibrium diagram and literature (Krukovich et al., 2016). The point analysis regions with results on the SEM image and the iron-boron equilibrium diagram are shown in Figures 7 and 8 below respectively.



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Spectrum          B     V    Cr     Fe     Mo      W
----------------------------------------------------
 1             6.27  2.61  2.91  27.30  22.22  38.69
 2             5.27  2.34  3.96  31.03  23.20  34.21
 6             7.50  2.07  3.07  33.17  22.86  31.32
 7             4.30  0.95  7.35  77.02   4.79   5.59
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Figure 7. EDX point analysis regions and analysis results of the borided AISI M2

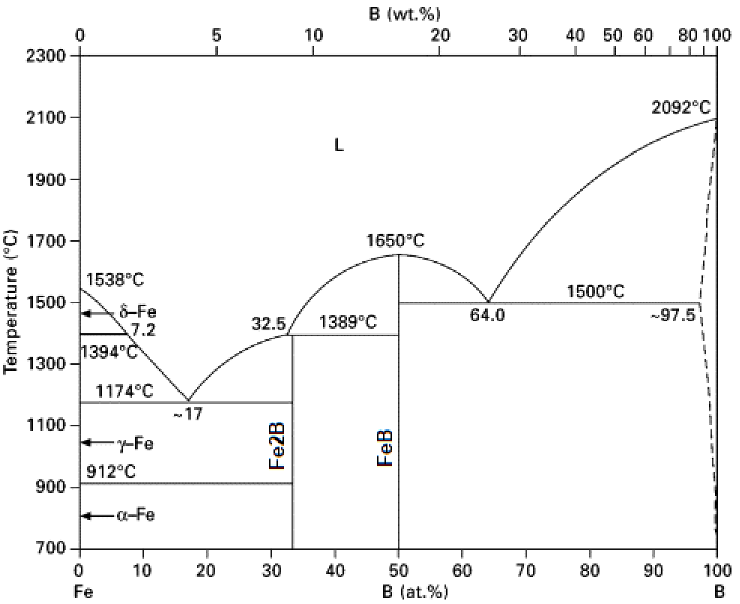

Figure 8. Iron-Boron equilibrium diagram (Massalsky, 1990)

If the iron-boron equilibrium diagram is examined, it can be seen that a double-phased (FeB+Fe2B) boride layer is formed in the region containing approximately 8 wt.% to 16 wt.% boron, and a single-phased boride layer is formed in the region containing up to a maximum of 8 wt.% boron.

To confirm this situation, XRD analysis was performed on the borided sample at 1100 C for 6 hours. For the XRD analysis, only the compounds formed by the element Fe and element B were taken into account. XRD analysis result was shown in Figure 9 below.

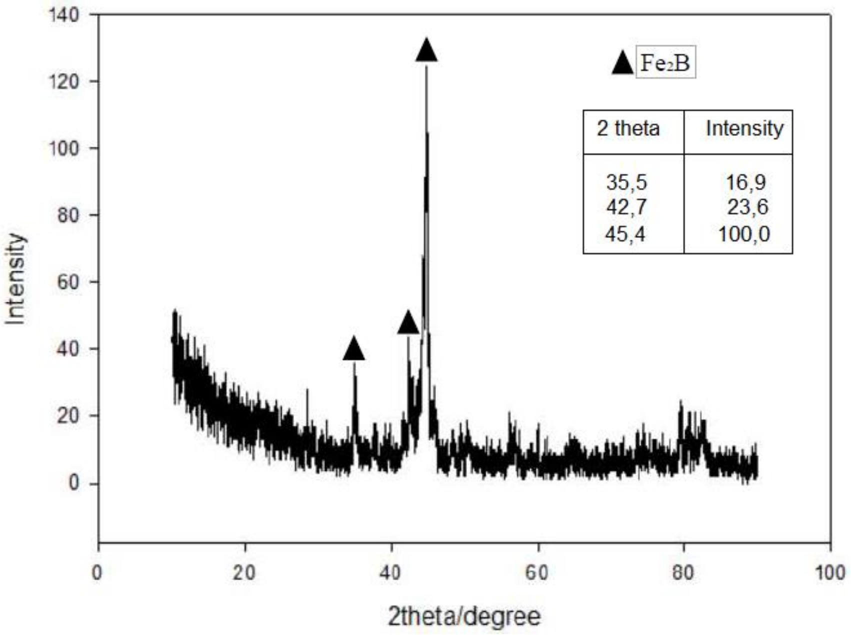

Figure 9. XRD analysis result of the boride layer formed on AISI M2

According to the XRD analysis result, after confirming that the boride layer formed on the surface of the samples was single-phase, layer thickness measurements were carried out on the SEM images. For the measurement of boride layer thicknesses, 5 measurements were taken from each sample and the average results are given in Table 3 below.

Table 3. Average boride layer thicknesses of borided AISI M2 high-speed steel

Borided material	Boriding temperature (C)	Boriding time (h)	Average Layer thickness (µm)
AISI M2 High-speed steel	900	2	5 ± 2
		4	14 ± 3
		6	19 ± 5
	1000	2	10 ± 3
		4	19 ± 4
		6	44 ± 7
	1100	2	25 ± 5
		4	45 ± 8
		6	70 ± 11

As can be seen in Table 3, the increase in boriding temperature and time causes the boride layer to be thicker. This is related to the penetration of boron atoms into the material at longer distances from the surface as the boriding temperature and time increase.

Finally, to determine the change in terms of the hardness of the sample, measurements were taken from the surface to the matrix using 100 g. with the help of the Vickers indentation method. Hardness measurements were made at a depth of 3 µm from the surface of the specimens and at a distance of approximately 10 µm between them. Three measurements were taken from each sample and their average hardness values are given in Table 4 below.

Table 4. Microhardness measurements of borided AISI M2 high-speed steel

Borided material	Hardness measured area	Average Hardness (GPa)
AISI M2 High-speed steel	Matrix	2.5 ± 0.08
	Transition zone	7.98 ± 0.11
	Boride layer	12.21 ± 0.09

When the hardness results are examined, the average hardness value of the boride layer is 1245 kg/mm^2 (12.2 GPa), and this value proves that the layer is composed of Fe_2B to the literature (Shaoming et al., 2016; Jiang et al., 2011).

As it has been demonstrated by many studies that double or single-phased layers are formed as a result of boriding of steel types, some literature examples regarding this are given in Table 1. In addition, the elements contained in the borided steel also affect the boriding process. As a result of these effects, the boride layer's morphology, thickness, and structure change (Davis, 2001; Goeuriot et al., 1982). Due to the high content of Cr, Mo, V, and W in the composition of AISI M2 high-speed steel used in this study, the morphology and thickness of the boride layer formed on the surface changed compared to the unalloyed steels.

When the microstructure images were examined, it was determined that the sawtooth morphology gradually shifted towards a columnar appearance and the saw teeth were formed in a rather short form. Likewise, if a comparison is made to the layer thickness measurements taken from the microstructure images, it has been determined that a thinner boride layer is formed than the borided, unalloyed, or low alloyed steels.

In addition, in many studies in the literature, it is stated that the average hardness value of the Fe_2B layer is 12 GPa, and it is seen that these results are compatible with the hardness value obtained in this study.

As a result, a single-phased boride layer was obtained on the surface of AISI M2 high-speed steel, unlike the double-phased boride layer formed on the surfaces of many steel types as a result of boriding treatment. Thus, in the introduction section, the negativities mentioned in detail (between phases or cracking) were prevented and a more stable boride layer was obtained.

4. Conclusion

It is possible to boriding of AISI M2 high-speed steel with the pack-boriding method. The boride layer formed on the surface, unlike many steel types, was formed as a single phase. Considering the SEM (BEI), EDX analysis, iron-boron equilibrium diagram, and hardness measurements, it was understood that this phase was Fe_2B. The formation of a single-phase boride layer instead of a double-phased boride layer is much more preferable in terms of mechanical properties, as stated in the literature. For this reason, it can be said that the single-phase boride layer formed on the surface of AISI M2 high-speed steel is much more useful than the two-phase boride layer formed in many other steel types.

Disclosure Statement

No potential conflict of interest was reported by the author(s).

Contribution Statement

In the preparation of this study, the authors; Tuna at the stages of the experiment, writing, and proof, Bünyamin and Özlem in the experiment and report phase, Polat writing, proof, and linguistic stages have contributed.

This study was supported by Hitit University Scientific Research Support Unit (BAP) within the scope of the project numbered TBMY19001.20.001.

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