



Research Article / Araştırma Makalesi

A STUDY ON ELECTROTHERMOCHEMICAL BORONIZING OF AN IF STEEL

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ABSTRACT

Boronizing is one of the primary surface hardening methods that have been applied for several decades in order to increase the service life of components, tools and equipments made mainly of ferrous alloys. It is a thermochemical surface diffusion treatment which includes adhesion and penetration of boron atoms supplied from a source that can be in solid, liquid, gas or even plasma states. Though boronizing has been applied to many alloy systems including all ferrous alloys, titanium alloys, nickel alloys, cobalt alloys, refractory metal (chromium, molybdenum, vanadium, niobium, tantalum, tungsten and zirconium) alloys, etc., no studies exist in the literature on boronizing of an Interstitial-Free (IF) steel. In this work, IF steel, possessing very little amount of alloying elements, was boronized by electrothermochemical method. Boronizing treatment was carried out at 900 and 1020 °C temperatures for durations between 15 and 90 min by applying current densities between 0.05 and 0.40 A/cm² in dehydrated sodium borax bath. The boride phases of Fe₂B, FeB, Fe₃B and Fe_{3.5}B were formed as a result of the boronizing treatment. Boronizing treatment at 1020 °C for 45 min by applying a current density of 0.20 A/cm² caused the formation of a boride layer with a thickness of 135 µm. Diffusion mechanisms and mechanical properties of the layers formed by boronizing treatment were investigated by means of microstructure and phase analysis and hardness measurements.

Keywords: IF steel, boronizing, boride layer.

IF ÇELİĞİNİN ELEKTROTHERMOKİMYASAL YÖNTEMLE BORLANMASI ÜZERİNE BİR ÇALIŞMA

ÖZET

Daha çok demir esaslı alaşımlardan üretilmiş parça, araç ve gereçlerin kullanım ömrünü uzatmak amacıyla onlarca yıldır uygulanan belli başlı yüzey sertleştirme yöntemlerinden biri borlamadır. Borlama, katı, sıvı, gaz, hatta plazma durumundaki bir kaynaktan sağlanan bor atomlarının yüzeye temas etmesi ve yüzeyden içeriye nüfuz etmesini içeren termokimyasal bir yüzey difüzyonu işlemidir. Her ne kadar tüm demir esaslı alaşımlar, titanyum alaşımları, nikel alaşımları, kobalt alaşımları, refrakter metallerin (krom, molibden, vanadyum, niobyum, tantal, tungsten ve zirkonyum) alaşımlarını içine alan pek çok alaşım sistemi borlanmış ise de bir arayeratomsuz çeliğin (IF çeliği) borlanmasına ilişkin çalışma literatürde mevcut değildir. Bu çalışmada, bünyesinde çok az alaşım elementi bulunduran arayeratomsuz çelik elektrottermokimyasal yöntemle borlanmıştır. Borlama işlemi 900 ve 1020 °C sıcaklıklarda 15-90 dakika sürede 0.05-0.40 A/cm² akım yoğunluğu uygulanarak kalsine sodyum boraks banyosunda yapılmıştır. Borlama işlemi sonucu Fe₂B, FeB, Fe₃B ve Fe_{3.5}B fazları oluşmuştur. 1020 °C sıcaklıkta 45 dakika sürede 0.20 A/cm² akım yoğunluğu uygulanarak yapılan borlama işleminde 135 µm kalınlığa sahip bir borür tabakası oluşmuştur. Borlama işlemi ile oluşturulan tabakaların difüzyon mekanizmaları ve mekanik özellikleri, mikroyapı ve faz analizi ve sertlik ölçümü yoluyla araştırılmıştır.

Anahtar Sözcükler: IF çeliği, borlama, borür tabakası.

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1. INTRODUCTION

Metallic parts, tools and equipments are exposed to several effects due to their running conditions. These effects are stress, impact, fluctuating stress, friction, corrosion and combinations of these phenomena. Metallic components must be more resistant to these effects so that they may continue to be in service for a long time. Surface properties, besides with the properties of the overall material, have a great importance in parts that are exposed to friction, wear, corrosion and oxidation in both low and high temperature conditions. Thus, the improvement of the surface properties of metallic components has become an important subject in last decades.

Boronizing is one of the treatments that are carried out to improve the surface properties of steels. The term boronizing is used to describe the diffusion of boron atoms into the surface of metal objects in a quantity exceeding the solubility limit, so that metal boride is formed [1]. In this process, boron is diffused onto the metallic part through the surface in a medium having a boron source. The diffused boron atoms, dependent on their concentration, form compounds principally with iron and also some other elements in steel. The introduced boron atoms tend to take place at interstitial positions due to their atomic radius, but they can also occupy substitutional positions [2, 3].

Boronizing agent (source) can be applied to the samples in several means. Boronizing agent may be in solid, liquid, gas or even plasma states. The atmosphere of the environment is adjusted according to the boronizing agent and the applied method. The applied temperature is dependent on the melting temperature of the used boronizing agent in the case of boronizing in the liquid environment. Molten salts are generally used as the boronizing agent and an electrochemical circuit is composed by applying an electric current, in case of boronizing in a liquid source.

Boronizing can be applied to many metals including all ferrous alloys, titanium alloys, nickel alloys, cobalt alloys, refractory metal (chromium, molybdenum, vanadium, niobium, tantalum, tungsten and zirconium) alloys, etc. Some non-metals like cemented carbides can also be boronized. Copper cannot be boronized since copper atoms act as a barrier preventing the absorption of the diffusing boron atoms. Boronizing gets faster as the purity of the substrate increases, i.e. there is a small amount of alloying elements in the substrate. Further, alloying elements affect the thickness and the morphology of the boride layer [4-7].

During boronizing treatment monophase and polyphase boride layers can be generated. Usually, a monophase layer (Fe_2B) is more desirable for practical applications than a polyphase one (FeB and Fe_2B). The phase Fe_2B , being thermodynamically more stable and formed earlier than FeB , has generally lower hardness than FeB . The phase FeB , found at the external surface, is more fragile, due to its orthorhombic crystalline structure and its boron content, which is approximately 16 wt. %. The inner phase, Fe_2B , has a boron content of 8.33 wt. % and its crystalline structure is tetrahedral. Furthermore, the difference in the thermal expansion coefficient of Fe_2B and that of FeB causes vacancy during temperature changes which lead to brittleness and deterioration in mechanical properties. The polyphase layer formation can be inhibited, but this depends on the process parameters, which are temperature, time, boron potential and chemical composition of the substrate [8]. This situation has only been achieved in limited number of studies, most of which are protected by a related patent [9-11].

When the formation of FeB cannot be prohibited, boronizing of different types of steels having low carbon and alloy content, which may lead to reduction of the undesired effects of FeB , shall be carried out. Although there has been plenty of research on boronizing of Armco iron, no studies have been performed on boronizing of an Interstitial-Free (IF) steel [12].

IF steel is a special type of steel produced with very low amounts of interstitial elements (primarily carbon and nitrogen). Low amounts of titanium and/or niobium are added to tie up the remaining interstitial atoms. Interstitials (C, N) are removed from the ferrite solid

solution through precipitation by adding titanium and/or niobium. Without free interstitial elements, these steels are very ductile and soft, also they will not age or bake harden, and will not form strain (Lüder's) lines during forming due to the absence of YPE (yield point elongation). The IF ferrite matrix exhibits excellent cold formability. Interstitial free (IF) steels have been successfully developed in order to perform specific or complex deep drawing operations in the automotive industry. Interstitial free steels are currently being extensively used for manufacturing car bodies and different parts of the car, where effect of asymmetric cyclic loading and the consequent deformation behaviour of the steel after ratcheting are important issues [13-16].

In this work, boride layers formed on the surface of the Interstitial-Free (IF) steel were studied. The diffusion mechanisms of boron and the mechanical properties of the boride layers in lack of alloying elements were investigated by means of microstructure observations, phase analysis and hardness measurements. For this aim, optical microscope (OM), scanning electron microscope equipped with energy dispersive spectrometer (SEM-EDS), X-ray diffraction spectrometer and micro-hardness tester were utilized.

2. EXPERIMENTAL DETAILS

2.1. Samples

The chemical composition of the IF steel samples is given in Table 1. The steel plates were obtained in hot-rolled condition during the intermediate stages of the production which was carried out in Ereğli Iron and Steel Works Co., Zonguldak, Turkey. The thickness of the steel plates was 4 mm. The samples were prepared by cutting the plates into the dimensions of 50 × 23 mm before the boronizing processes.

Table 1. The chemical compositions of the IF steel, in weight per cent

Element	C	Si	Mn	P	S	Cr	Ni	Mo	Al	Ti	V	B
wt. %	0.0088	0.0110	0.2130	0.0136	0.0089	0.0250	0.0470	0.0010	0.0286	0.0597	0.0083	0.0004

2.2. Diffusion Coating Processes

The boronizing (boron diffusion) processes were carried out electrothermochemically in a chamotte ladle in a Protherm pit-type furnace (2.5 kW) by using commercially-pure nickel anode. The boronizing agent is liquid dehydrated sodium borax ($\text{Na}_2\text{B}_4\text{O}_7$). The boronizing processes were performed by applying 0.05, 0.10, 0.15, 0.20, 0.30 and 0.40 A/cm² current densities at 900 and 1020 °C temperatures for 15, 30, 45, 60 and 90 min. The Goodwill PPE 3323 power supply (sensitivity 0.05 mV) was used to obtain the desired electric current density during diffusion coating processes.

2.3. Characterization

For optical investigation, all samples were metallographically prepared in standard manner and etched with Nital3. The prepared samples were investigated by using an Olympos GX71 optical microscope. The thickness of the diffusion layers were measured visually on the micrographs obtained by using this microscope. Additionally, the scanning electron microscope (Jeol JSM-7000F Field Emission SEM) equipped with energy disperse spectrometer (EDS) was utilised in order to examine the diffusion layers in more detail. The samples were coated with platinum and painted with AgCl₂ solution before the examination.

To carry out the phase analyses, Philips Pan Analytical X'Pert Professional type X-ray diffraction spectrometer having a tube supplying CuK α radiation was utilised. The scanning was

carried out between 10° and $90^\circ 2\theta$ values at a rate of $0.30^\circ/\text{min}$. The general phase analyses of the samples were done using Gonio method and the surface-intensive phase analyses of the samples were carried out using Omega method.

Hardness values of the samples were measured by using a Future-Tech FM 700 microhardness tester. The changing of the hardness values from the surface to the substrate was determined on Vickers indentation by applying a load of 50 gf for 7 sec.

3. RESULTS AND DISCUSSION

3.1. Results of the Microstructural Investigation Done by Optical Microscope

The effect of the current density is visualised in the micrographs given in Figures 1(a), 1(b), 1(c), 1(d), 1(e) and 1(f). Boronizing was carried out at 900°C for 45 min by applying current densities of 0.05, 0.10, 0.15, 0.20, 0.30 and 0.40 A/cm^2 , respectively.

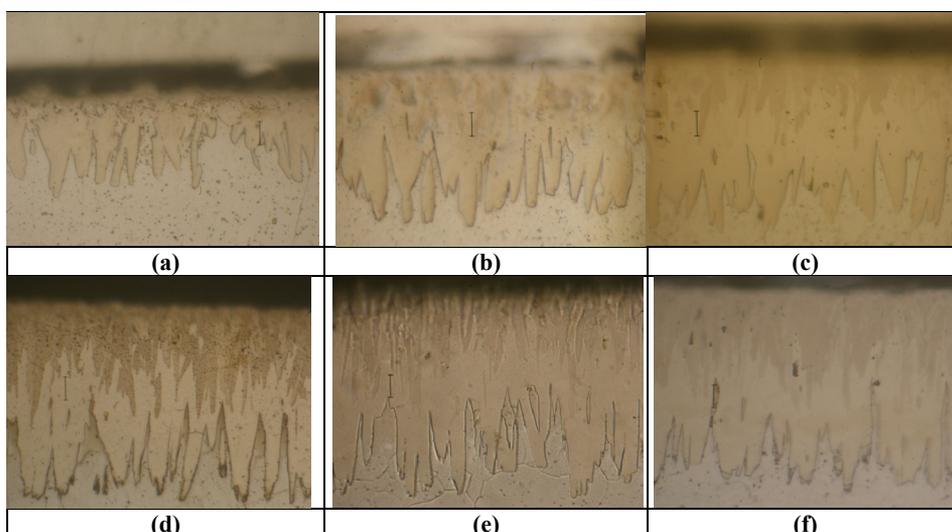


Figure 1. Cross-section micrographs of IF steel boronized at 900°C for 45 min by applying current densities of (a) 0.05, (b) 0.10, (c) 0.15, (d) 0.20, (e) 0.30 and (f) 0.40 A/cm^2 , respectively (Scale: $10\ \mu\text{m}$, Magnification: X1000)

The effect of time is visualised in the micrographs given in Figures 2(a), 2(b), 2(c), 2(d) and 2(e). Boronizing was carried out at 900°C for (a) 15, (b) 30, (c) 45, (d) 60 and (e) 90 min, respectively, by applying a current density of 0.10 A/cm^2 .

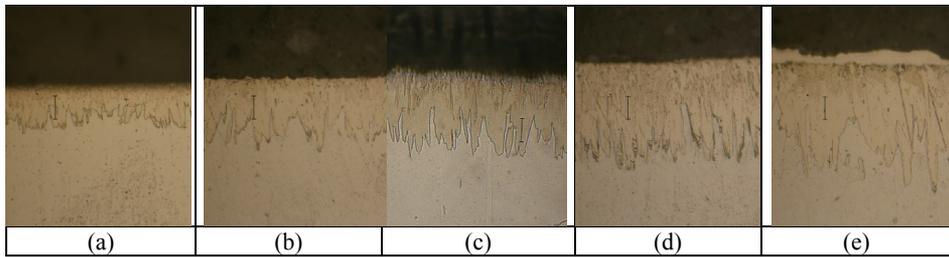


Figure 2. Cross-section micrographs of IF steel boronized at 900 °C by applying current densities of 0.10 A/cm² for durations of (a) 15, (b) 30, (c) 45, (d) 60 and (e) 90 min, respectively (Scale: 20 µm, Magnification: X500)

The curves indicating the effect of the current density and the duration on the thickness of the diffusion layer are given in Figures 3 and 4, respectively.

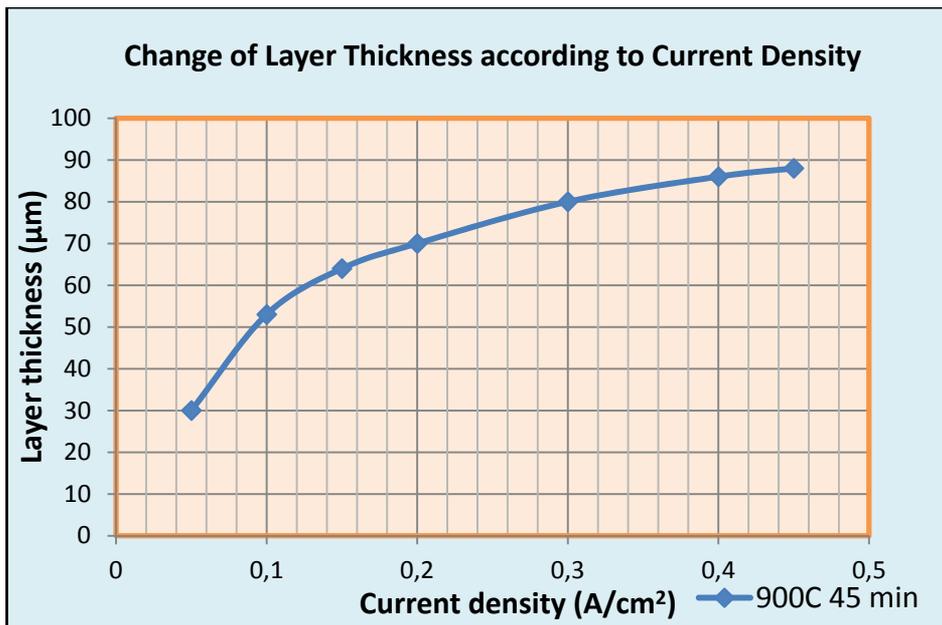


Figure 3. The curve indicating the effect of the current density on the thickness of the diffusion layer. The temperature was 900 °C and the duration was 45 min

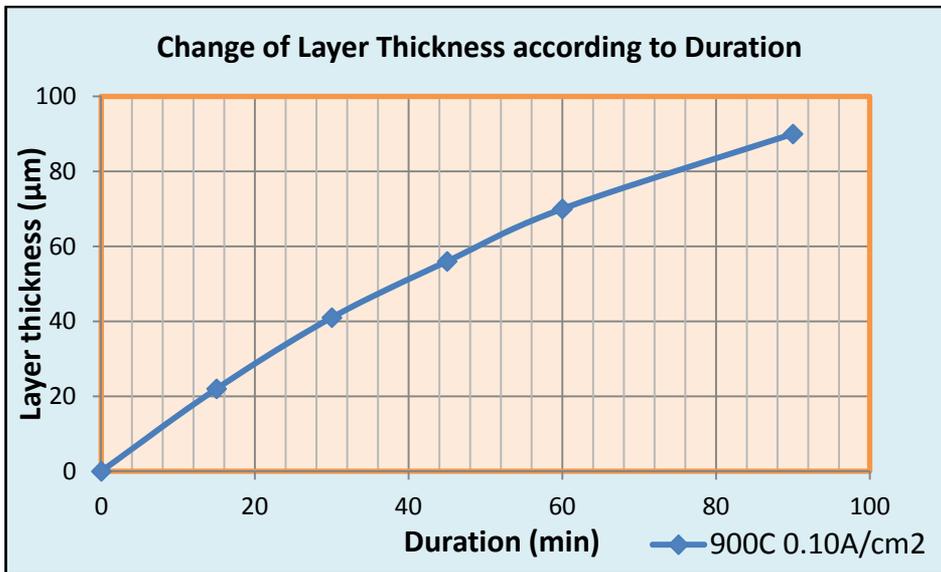


Figure 4. The curve indicating the effect of the duration on the thickness of the diffusion layer. The temperature was 900 °C and the current density was 0.10 A/cm²

The effect of temperature is illustrated in the micrographs given in Figures 5(a) and 5(b). Boronizing was carried out for 45 min by applying a current density of 0.20 A/cm² at 900 and 1020 °C, respectively.

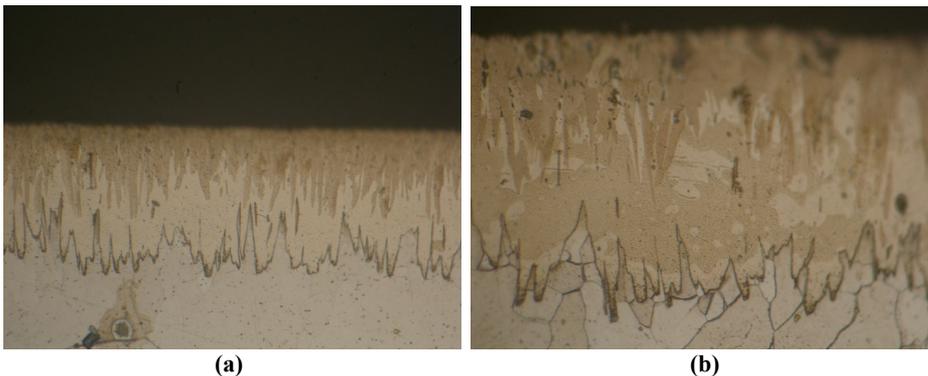


Figure 5. Cross-section micrograph of IF steel boronized at (a) 900 and (b) 1020 °C for 45 min by applying a current density of 0.20 A/cm² (Scale: 20 µm, Magnification: X500)

The boride phases of Fe₂B and FeB are identified in the micrographs easily. When the micrographs are examined, it is seen that, unlike the usual boronizing treatments, no transition zone was formed between the boride layer and the substrate. The reason for this is thought to be the very small amount of carbon content in the IF steel. The carbon content was not sufficient to form the transition zone, which can be roughly defined as Fe₃(B,C) phase [17].

On the evaluation of the effects of the boronizing parameters, i.e., the applied current density, time and temperature, it is seen that the thickness of the boride layer was increased while any of these parameters was being increased.

The increasing of the applied current density and the duration resulted in an increase in the continuity of the diffusion layer; that is, partial filling up the unboronized regions between the saw-like boronized morphologies by boron diffusion and increasing the continuous thickness of the diffusion layer.

Layer thickness changed almost linearly until the duration of 60 min and then the effect of time tended to decrease. This behaviour might be explained by filling up the interstitial lattice positions by boron atoms and decreasing of the number of available positions for diffusion. In addition, at durations higher than 60 min, the grain size of the substrate decreased as a result of recrystallization. This situation affected the morphology of the boride layer, which gained a sharper shape. When the temperature was 900 °C and the applied current density was 0.10 A/cm², the thickness of the boride layer reached about 24 µm for 15 min duration, as well as it reached about 40, 58, 70 and 88 µm for 30, 45, 60 and 90 min durations, respectively. The effect of the applied current density on the thickness of the diffusion layer started to slow down at the first value of the current density, i.e., 0.05A/cm². When the temperature was 900 °C and the duration was 45 min, the thickness of the diffusion layer was about 30 µm at the current density of 0.05 A/cm², as well as it reached about 50 µm, 60 µm, 70 µm, 80 µm and 86 µm at 0.10, 0.15, 0.20, 0.30 and 0.40 A/cm² current densities respectively.

3.2. Results of the Scanning Electron Microscope Analyses

Figure 6 shows the SEM micrograph of the cross-section of the IF steel sample boronized at 900 °C for 45 min by applying a current density of 0.10 A/cm². It was observed that the borides formed on the IF steel sample surfaces have a saw-tooth and porosity-free morphology. The thickness of the boride layer on the boronized IF steel sample was obtained as about 57.70 µm, at 900 °C for 45 min by applying a current density of 0.10 A/cm². Sen et al. [18] investigated the thickness of boride layer as a function of the boronizing time within a temperature range of 850-950 °C for a series of steel alloys and reported that the thickness versus iso-thickness diagrams which exhibited the approximate boride layer thickness were in the range of 52-60 µm, at 900 °C for 2h for AISI 5140, AISI 4340 and AISI D2 steels. These observations verify the finding that the thickness and proportion of each boride layers (single or double phase) formed depend on the chemical composition of the substrate material, boronizing medium, process temperature and duration of the treatment [11, 19-22].

Energy dispersive X-ray spectroscopy (EDS) point analysis (marked as 1 through 9 in the SEM micrograph given in Figure 6) results of the IF steel sample boronized at 900 °C for 45 min by applying a current density of 0.10 A/cm² are given in Table 2. The EDS analysis exhibited that iron content in the boride layer was lower at the outer layer than that at the inner layer, referring these surface layers consisted of FeB and Fe₂B phases, respectively. Different tones of gray colour correspond to the different stoichiometric ratios of boron content. These findings conform with previous studies [23-26].

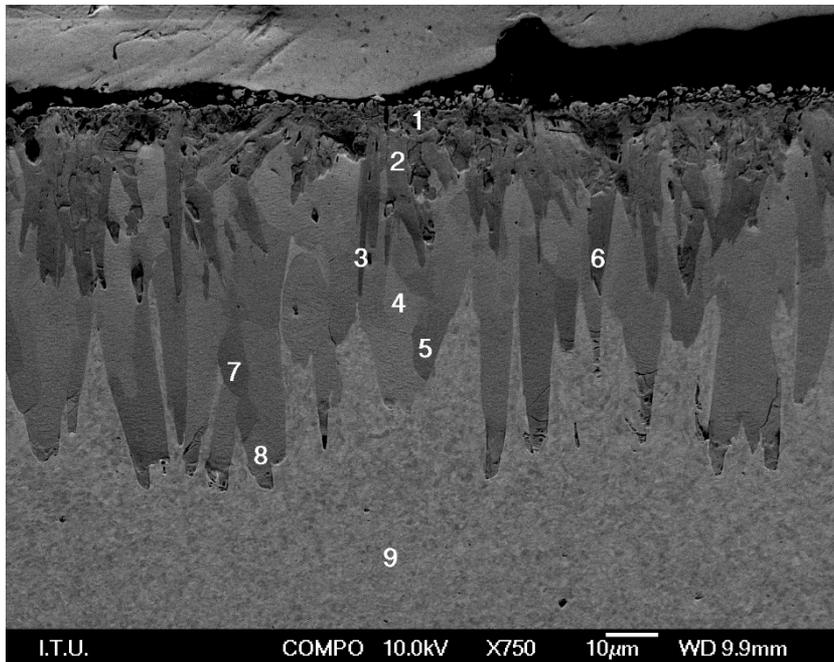


Figure 6. SEM micrograph of the IF steel sample boronized at 900 °C for 45 min by applying a 0.10 A/cm² density electric current. The marked numbers indicate the points from which EDS analyses were taken

Table 2. SEM/EDS spectra results taken from the IF steel sample boronized at 900 °C for 45 min by applying a 0.10 A/cm² density electric current (marked via numbers in Figure 6)

Point (#)	B (wt. %)	Fe (wt. %)	C (wt. %)
1	17.50	82.50	-
2	18.22	81.78	-
3	18.94	81.06	-
4	11.69	88.31	-
5	9.97	90.03	-
6	18.48	81.52	-
7	10.70	89.30	-
8	9.73	90.27	-
9	-	98.95	1.05

3.3. Results of the X-Ray Diffraction Analyses

Figure 7 shows the X-ray diffraction analyses results of the IF steel sample boronized at 900 °C for 45 min by applying a current with a density of 0.10 A/cm². As shown in Figure 7, the present phases of borides on the surfaces of boronized IF sample were iron boride (FeB, an orthorhombic structure with lattice parameters a=4,0530 Å, b=5,4950 Å, c=2,9460 Å, PDF no. 002-0869 [27]), boron iron (Fe₂B, a tetragonal structure with lattice parameters a=b=5,0990 Å, c=4,2400 Å, PDF no. 003-1053[28] and Fe₂B, a tetragonal structure with lattice parameters a=b=5.1317 Å, c=8.5321 Å, PDF no. 039-1314 [29]), boron iron (Fe₃B, a tetragonal structure with lattice

parameters $a=b=8.6210 \text{ \AA}$, $c=4.3130 \text{ \AA}$, PDF no. 034-1037 [30]), and boron iron ($\text{Fe}_{3.5}\text{B}$, a tetragonal structure with lattice parameters $a=b=8.6200 \text{ \AA}$, $c=4.2800 \text{ \AA}$, PDF no. 034-1032 [31]). It should be mentioned here that since both Fe_3B and $\text{Fe}_{3.5}\text{B}$ phases have the same crystal structure with the lattice parameters very close to each other, the respective X-ray peaks for both phases are coincident with each other. The obtained X-ray diffraction analyses verify the SEM/EDS spectra results given in Table 2. However, it is well known that during boronizing of ferrous alloys boron atoms are inserted into the metal lattice at the surface of the substrate by thermal effect to create borides, then a diffusion layer containing either one iron boride (Fe_2B) or two iron borides ($\text{FeB} + \text{Fe}_2\text{B}$) is formed [4, 32-36]. Also, observation of some peaks corresponding to metastable Fe_3B and $\text{Fe}_{3.5}\text{B}$ phases in the current study conforms with the findings in previous researches on boronizing of mild steel C15 [37], AISI 1018 steel [38], AISI 1040 steel [39], and DC04 low carbon steel [40]. The presence of metastable phases such as Fe_3B and $\text{Fe}_{3.5}\text{B}$ might be attributed to the amount of the diffused boron from surface through matrix as the following: $\text{FeB}_x (x>1) \rightarrow \text{FeB} \rightarrow \text{Fe}_2\text{B} \rightarrow \text{Fe}_3\text{B} \rightarrow \text{Fe}_y\text{B} (y>3) \rightarrow \text{Fe}$ [40], consequently, occurring of the competitive nucleation and growth by diffusion controlled characteristics during the boronizing process.

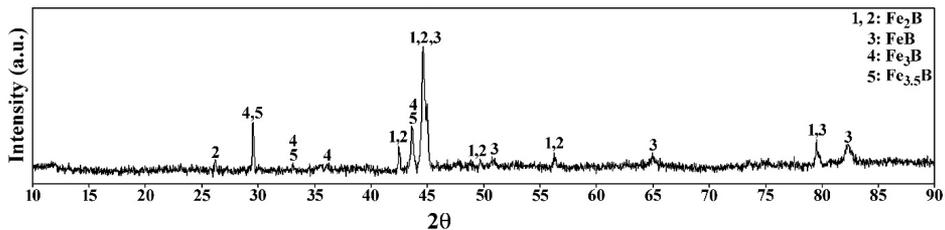


Figure 7. X-ray diffraction spectrum of the IF steel sample boronized at 900 °C for 45 min by applying a current density of 0.10 A/cm²

3.4. Results of the Hardness Measurements

Figure 8 shows hardness variation as a function of the applied current densities for the IF steel sample boronized at 900 °C for 45 min. The hardness of the outside boride layer was about 1550 kg/mm², for the IF steel sample boronized at 900 °C for 45 min by applying a current density of 0.10 A/cm², whereas the hardness of the unborided substrate was about 110 kg/mm². Since the presence of hard FeB , Fe_2B and Fe_3B phases, the hardness of the boride layer is significantly higher than that of the substrate. In addition, the hardness of FeB layer located in the surface of the boride layer is higher than those of Fe_2B and Fe_3B layers. This is in accordance with the observations of Sen et al. [26], Matushka [23], and Kartal et al. [40].

As a comparison, the measured hardness values belonging to the FeB , Fe_2B and Fe_3B phase regions from the surface through the matrix of the IF steel sample, are listed in Table 3 including the results obtained by other researchers [41-44]. It is clear that hardness values of the current study are nearly in the same intervals given in the literature. Based on the boronizing conditions, the hardness intervals are 1970-1550 kg/mm² for FeB and 1664-1240 kg/mm² for Fe_2B . Thus, hardness variation in the boride layer compounds might be attributed to the chemical composition of the substrate, stoichiometry of the formed borides or the formation of metastable boride (Fe_3B) [45], and boronizing conditions. Further, increasing the applied current density from 0.1 A/cm² to 0.4 /cm², resulted in a significant increment in hardness of the outside boride layer, i.e., 1550 kg/mm² to 2500 kg/mm², as seen in Figure 8.

Table 3. Hardness data of the boride layers obtained from both the literature [41-44] and the current study

Hardness of boride layers (kg/mm ²)		Substrate	Boronizing environment	Reference
FeB	Fe ₂ B			
1970-1550	1664-1240	IF steel (0.1 A/cm ²)	Liquid	Current study
2500-1613	1780-1301	IF steel (0.4 A/cm ²)	Liquid	Current study
2050	1500	Low carbon steel	Liquid	[41]
2250	1500	Mn steel	Liquid	[42]
2100-1950	1450-1380	Various chemical composition of Steel	Various environment	[43]
2140-1665 (FeB+Fe ₂ B)		Ductile iron	Liquid	[44]

The effect of boronizing time on the hardness of boride layer of IF steel is presented in Figure 9. It is seen that the maximum hardness value for 30 min duration was about 1772 kg/cm² at 10 μm distance, while the maximum hardness values for longer durations, i.e., 60 min and 90 min, were about 1745 and 1765 kg/cm² at 20 μm distance, respectively. This result clearly indicates that the shorter duration (30 min) causes thinner boride layer formation (FeB). Similarly, for each duration, reaching the same hardness value of about 400 kg/cm² was achieved at about 40, 64 and 84 μm distances, respectively. Thus, when the duration was increased from 30 min through 90 min, the formation of the boride phases (FeB, Fe₂B, and Fe₃B) was observed at different distances depending on the diffusion of boron atoms, consequently, changing the position of the boride layer-substrate interface. As a thermo-chemical diffusion process, during boronizing, the boride layer thickness changes depending both on the treatment temperature and duration. Further, the similar behaviour has been reported by previous researchers [17, 21, 46-49].

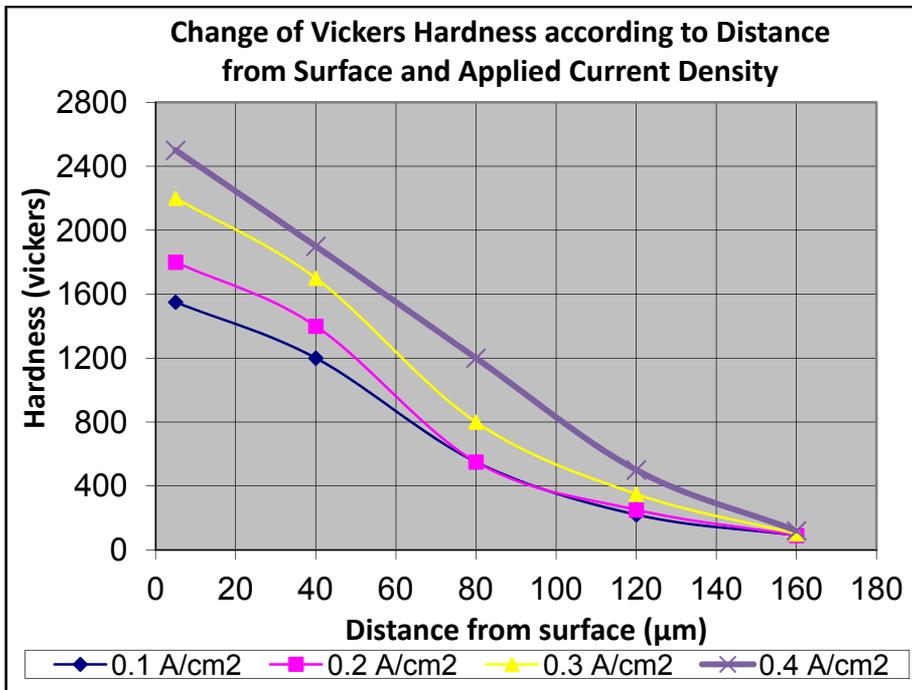


Figure 8. Hardness variation as a function of the applied current densities for IF steel boronized at 900 °C for 45 min

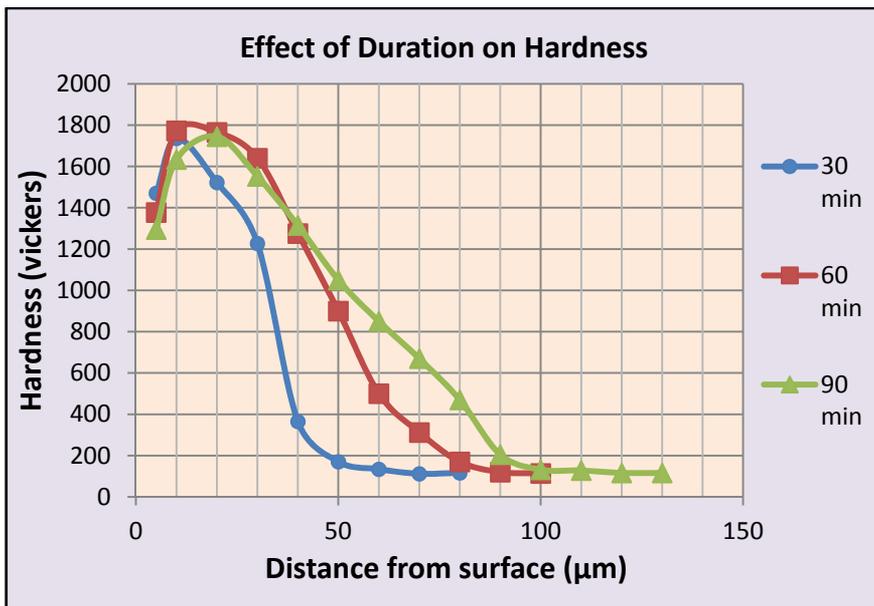


Figure 9. Hardness variation as a function of duration for IF steel boronized at 900 °C by applying a current density of 0.10 A/cm²

4. CONCLUSION

The results of the present study can be summarized as follows:

1. Interstitial Free (IF) steel was boronized electrothermochemically in a salt bath composed of merely calcined sodium borax ($\text{Na}_2\text{B}_4\text{O}_7$). Iron boride layers of Fe_2B , FeB , Fe_3B and $\text{Fe}_{3.5}\text{B}$ were formed. The thickness of the boride layer increased with temperature, duration and the applied current density. The thickness of the boride layer reached $135 \mu\text{m}$ in case of boronizing at $1020 \text{ }^\circ\text{C}$ for 45 min by applying a current having a density of 0.20 A/cm^2 .

2. No transition zone was observed between the boride layer and the substrate, unlike most boronizing treatments. The low amount of carbon is thought to be insufficient to form the transition zone, which can be roughly defined as $\text{Fe}_3(\text{B,C})$ phase.

3. Concerning boronizing parameters, the applied current density presented a directly proportional, but decreasing effect on the thickness of the boride layer beginning from the zero point, while duration almost exhibited a linear effect until about 45 minutes, where it started to slow down.

4. The hardness of the boride layer increased with the applied current density and duration.

5. In the current study, the obtained hardness of the boride layers for FeB and Fe_2B phases in the IF steel samples by applying the current density of 0.1 A/cm^2 exhibited lower values than those of the previous investigations including low carbon steel, Mn steel, and ductile iron, indicating the exploration of superior property about brittleness of the boride layers achieved by the IF steel as compared to the present literature.

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