

# Kinetics of Plasma Paste Boronized AISI 8620 Steel in Borax Paste Mixtures<sup>1</sup>

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**Abstract**—In the present study, AISI 8620 steel was plasma paste boronized by using various borax paste mixtures. The plasma paste boronizing process was carried out in a dc plasma system at a temperature of 973, 1023 and 1073 K for 2, 5 and 7 h respectively in a gas mixture of 70% H<sub>2</sub>–30% Ar under a constant pressure of 10 mbar. The properties of the boride layer were evaluated by optical microscopy, X-ray diffraction, the micro-Vickers hardness tester and the growth kinetics of the boride layers. The thickness of the boride layers varied from 14 to 91 μm depending on the boronizing time and temperature. X-ray diffraction analysis of boride layers on the surface of the steel revealed the formation of FeB and Fe<sub>2</sub>B phases. Depending on the temperature and layer thickness, the activation energies of boron in steel were found to be 99.773 kJ/mol for 100% borax paste.

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## 1. INTRODUCTION

Boronizing refers to a surface diffusion treatment by which a boride coating is formed on the component surfaces. Formation of iron borides on steel surfaces is a well-known example of boronizing [1, 2]. It is a thermal diffusion treatment of boron compounds used to form iron borides which typically require process temperatures ranging from 833 to 1273 K in solid, salt, gaseous, paste, spark plasma sintering (SPS), electrochemical and plasma media [3–8]. The boronizing methods stated above have certain disadvantages. In gaseous boronizing, boron sources such as BCl<sub>3</sub>, TMB (trimethyl borate), TEB (triethyl boron) and BF<sub>3</sub> along with H<sub>2</sub> and Ar gases are used [9–11]. In the case of liquid boronizing, formation of a firmly adhesive salt layer on the workpieces constitutes one of its advantages; however this can be quite costly to remove after boronizing has been completed. Pack boronizing is used commercially by Ekabor<sup>®</sup>. Although pack boronizing is more widely applied for commercial purposes than other methods, higher treatment temperatures and longer periods of time constitute its drawbacks [12–14].

Plasma boronizing has many advantages over the conventional boronizing process. For example, high energy efficiency is expected as a high energy of source of plasma is utilized in the plasma boronizing process, and distortion can be minimized after the process since the processing temperature is relatively lower than that in the conventional processes. However, the plasma boronizing process also has its limitations. B<sub>2</sub>H<sub>6</sub> and BCl<sub>3</sub> gases have been used as boron source gases but these gases are relatively expensive, toxic and

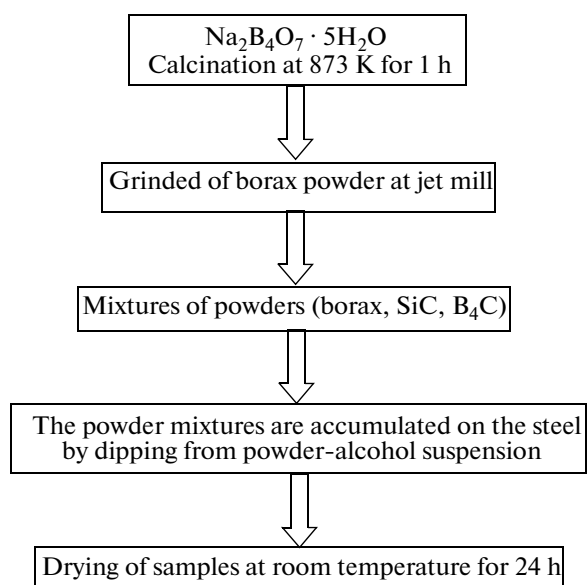
explosive. Corrosion in the vacuum chamber by boron chloride is another serious problem in plasma boronizing [10].

The characterization of boronized steels by using various boronizing processes has been evaluated by a number of investigators [12, 15–17]. However, the literature on kinetic and characterization of paste boronized steels in a plasma environment is scarce. In this study, low alloyed AISI 8620 steel was plasma paste borided. The removal of the borax paste remaining on the surface of the specimen boronized with 100% borax paste is difficult and the process is time-consuming, therefore, SiC and B<sub>4</sub>C were added to the borax paste. In addition, the ways in which SiC and B<sub>4</sub>C provide a contribution to the formation of phases FeB and Fe<sub>2</sub>B were investigated. This occurs by a decrease or an increase of the boron concentration with SiC and B<sub>4</sub>C in the environment. The main objective of this study is to characterize plasma paste boronized AISI 8620 steel using borax pastes. The phase structure, microhardness and kinetics of the boride layers were investigated using X-ray diffraction (XRD), microhardness tester. In addition, the present study provides a good comparison between plasma paste boronizing and conventional boronizing.

## 2. EXPERIMENTAL DETAILS

AISI 8620 steel which contained 0.19% C, 0.4% Cr, 0.7% Mn and 0.4% Ni was used for the investigations. The samples had a cylindrical shape and were 18 mm in diameter and 6 mm in length. AISI 8620 steel samples were ground using 800 mesh SiC paper and polished with 0.1 alumina suspension to obtain a smooth surface. In this study, borax, SiC and B<sub>4</sub>C powder mixtures (each of 150 μm) with various per-

<sup>1</sup> The article is published in the original



**Fig. 1.** Flow chart of preparing of borax based pastes on the samples steel for plasma paste boronizing.

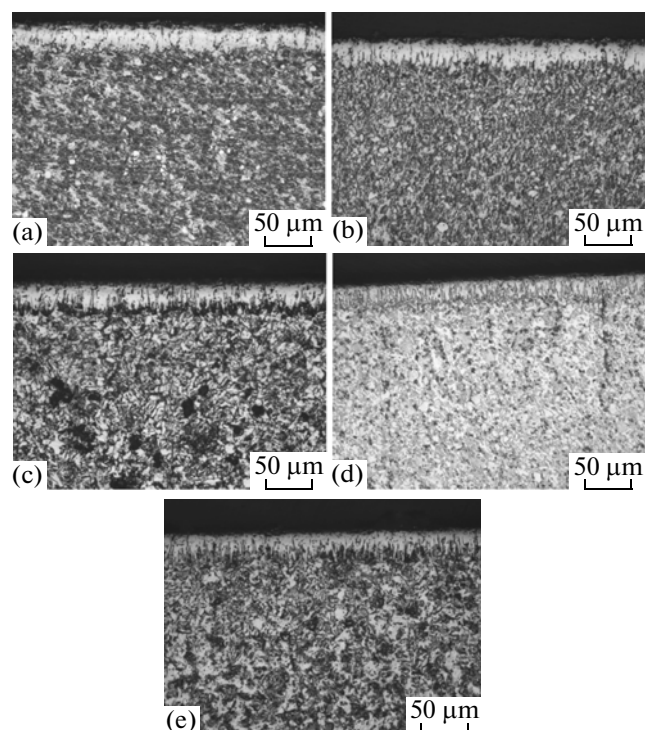
centages were used as a paste. Figure 1 shows a flow chart of the preparation of borax based pastes on the sample steels for plasma paste boronizing. The powder mixtures were accumulated on the steel that was immersed in a powder-alcohol suspension. Percentages of paste mixtures used in this study are shown in Table 1.

A plasma paste boronizing treatment was performed in a dc plasma system, which is described in [12]. The prepared samples were placed in the vacuum container and the container pressure was set to  $2 \times 10^{-2}$  mbar of vacuum. The samples were plasma paste boronized at 973, 1023 and 1073 K for 2, 5 and 7 h in a gas mixture of 70% H<sub>2</sub>–30% Ar under a constant pressure of 10 mbar. The temperature of the samples was measured by using a chromel-alumel thermocouple that was placed at the bottom of the treated samples.

Cross-sections of plasma paste boronized steels were prepared metallographically to observe morphological details using the BX60 Olympus microscope. The X-ray diffractograms were obtained by using a copper tube source as dictated by the conventional bragg-brentano ( $\theta$ – $2\theta$ ) technique having symmetric

**Table 1.** Ratios of borax, SiC and B<sub>4</sub>C paste mixtures used for plasma paste boronizing treatment

Borax	SiC	B <sub>4</sub> C
100%	–	–
70%	30%	%
70%	–	30%
30%	70%	–
30%	–	70%



**Fig. 2.** Optical cross-section microstructure of plasma paste boronized steel in different % borax paste mixtures for 5 h at 1023 K, (a) 100% borax, (b) 70% borax + 30% SiC, (c) 70% borax + 30% B<sub>4</sub>C, (d) 30% borax + 70% SiC, (e) 30% borax + 70% B<sub>4</sub>C

geometry with monochromatized radiation (Cu K $\alpha$ ,  $\lambda = 0.15418$  nm). The thickness of the layers formed on the steels was measured by an optical micrometer attached to the optical microscope.

The hardness of the boride layers was measured on the cross-sections using the Micro-Vickers indenter (Shimadzu HMV-2) with 50 g loads.

### 3. RESULTS AND DISCUSSION

#### 3.1. Surface Characterization

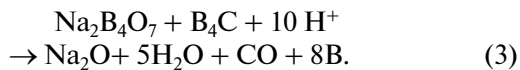
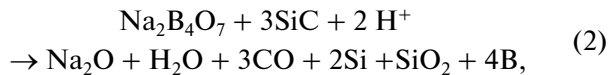
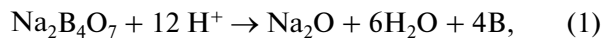
Figure 2 shows optical microstructures of plasma paste boronized steel at 1023 K for 5 h for 100% borax, 70% borax + 30% SiC, 70% borax + 30% B<sub>4</sub>C, 30% borax + 70% SiC, 30% borax + 70% B<sub>4</sub>C paste mixtures. The morphology of borides formed on the surface of the substrate has a columnar nature.

The thickness of the boride layers formed on the steels ranged from 14 to 91 μm. The thickness of boride layer increased with the increase in boronizing temperature and time [18–20] in all paste mixtures. While the maximum boride layer thickness value was obtained for 100% borax, the minimum boride layer thickness value was obtained for 70% borax + 30% B<sub>4</sub>C paste mixture. Since the plasma activates the chemical reaction to a higher extent, a thicker boride layer is formed than when using conventional boroniz-

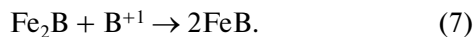
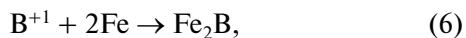
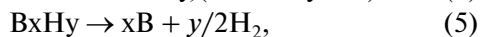
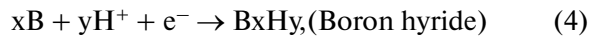
ing methods [12, 21, 22] at similar temperatures. It is possible to form a boride layer with the same thickness at lower temperatures in a plasma environment [14, 22–24]. Yoon et al. [10] reported that by using the plasma paste boronizing method for stainless steel, a thick boride layer with a flat structure could be obtained in a shorter time and at a lower temperature than that obtained using conventional thermal diffusion boronizing. Boronizing processes at lower temperatures (973, 1023 and 1073 K) and in less time (2 h) can be performed by plasma paste boronizing. This can be associated with plasma that reduces pollution of the environment [25] and reduces the activation energy required for the compound formation [10].

The X-ray diffraction (XRD) patterns of paste boronized AISI 8620 steel by plasma at various temperatures and times are given in Fig. 3. X-ray diffraction analysis of boride layers on the surface of specimens revealed peaks of FeB, Fe<sub>2</sub>B. While an increase was observed in the FeB phase with the increase in plasma paste boronizing temperature, a decrease occurred in the Fe<sub>2</sub>B phase. In the samples plasma paste boronized with SiC added to borax paste, the Fe<sub>2</sub>B phase was found to be slightly more dominant than the FeB phase. While the increase of boron concentration in the environment also increased the FeB phase formation, a single component Fe<sub>2</sub>B phase formed with the decrease in boron atom potential [26, 27].

During plasma paste boronizing, borax reacts with active hydrogen (H<sup>+</sup>) in glow discharge and the reaction occurs as follows (Equality 1–3);



Atomic boron was produced by the decomposition of the boron hydride (BxHy) from the paste, and this atomic boron became the active boron, B<sup>+</sup> within the molten borax or in the glow discharge. Finally, this active boron, B<sup>+</sup>, diffused and reacted with Fe to form the boride layer (Equality 4–7).



### 3.2. Microhardness

Figure 4 shows hardness values of AISI 8620 steel plasma paste boronized in paste mixtures with different percentages of borax for 2, 5 and 7 h at 973, 1023 and 1073 K. Surface hardness appeared to be high due to FeB and Fe<sub>2</sub>B phases formed as a result of the plasma paste boronizing treatment. It is known that

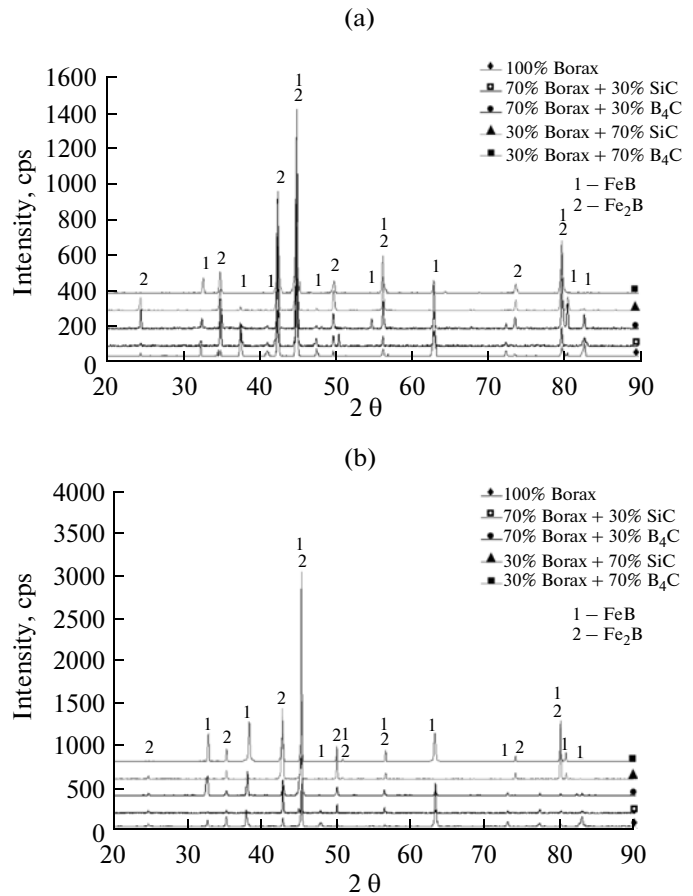


Fig. 3. XRD patterns of AISI 8620 steel that was plasma paste boronized for 5 h boronizing time and in different temperatures with different % borax based pastes, (a) 973 K, (b) 1073 K.

the FeB phase has more hardness values compared to the Fe<sub>2</sub>B phase [28, 29].

While the lowest hardness value was obtained at 1598 HV<sub>0.05</sub> for 70% borax + 30% SiC boron paste mixture for 2 h at 973 K, the highest hardness value was found to be 1976 HV<sub>0.05</sub> for 30% borax + 70% B<sub>4</sub>C paste for 7 h at 1073 K. Along with the increase in boronizing temperature and time, surface hardness of the boride layer increased [30–32].

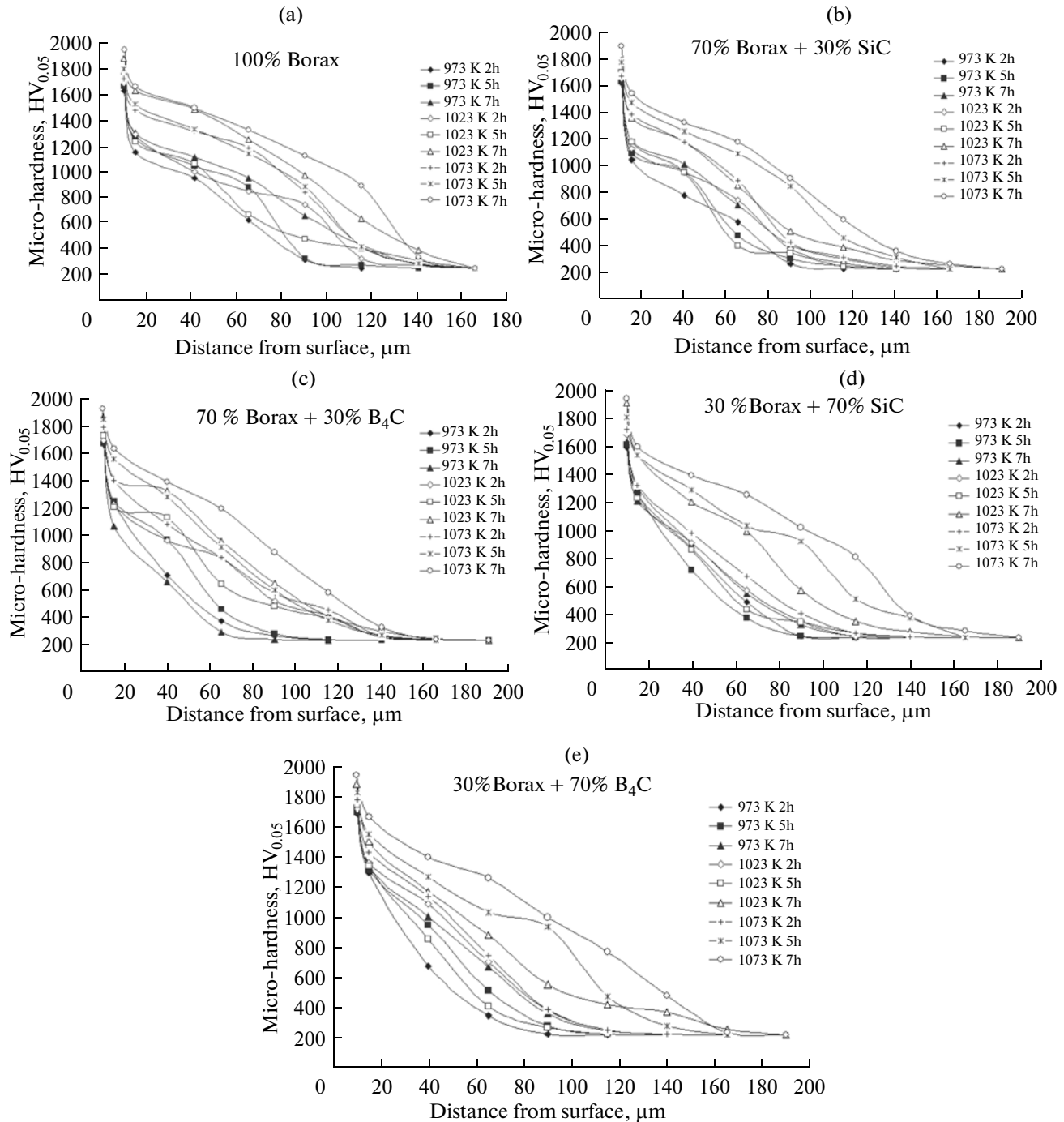
### 3.3. Kinetics

With the increase in boronizing temperature and time in borax paste mixtures and the thickness of the boride layers formed on the steel, the thickness of the boronizing layers ranged from 14 to 91 μm.

The thickness of the boride layer as a function of squared time is described by:

$$x^2 = Kt, \quad (8)$$

where  $x$  is the depth of the boride layer,  $K$  is the growth rate constant, and  $t$  is the time. Generally, it is expected that the plot of boride layer thickness versus

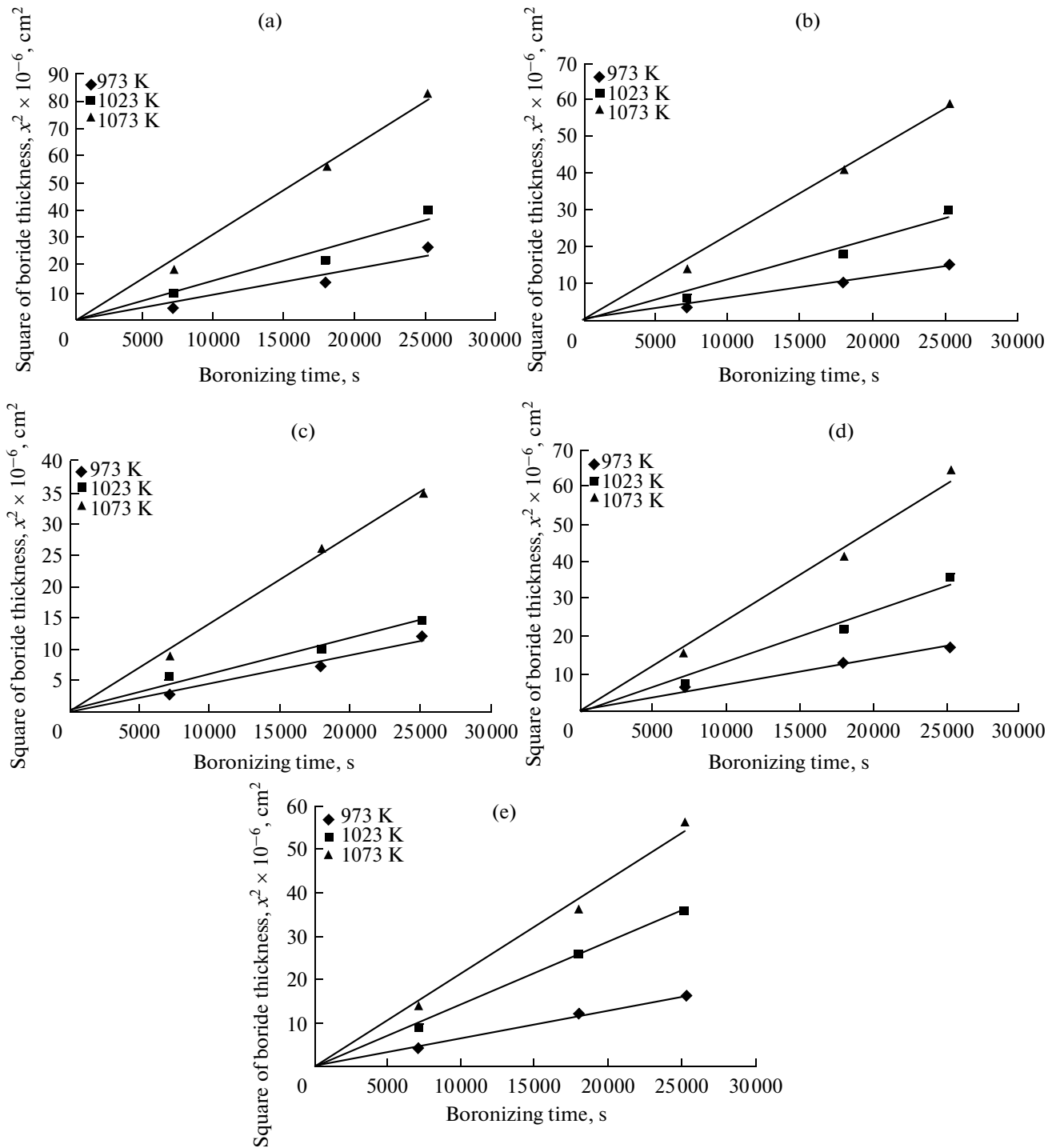


**Fig. 4.** From surface to core hardness distribution of the steel plasma paste boronized in different % borax paste mixtures, (a) 100% borax, (b) 70% borax + 30% SiC, (c) 70% borax + 30% B<sub>4</sub>C, (d) 30% borax + 70% SiC, (e) 30% borax + 70% B<sub>4</sub>C

squared treatment time gives a straight line which indicates that the growth of the layer has a parabolic dependence to time. The values of  $K$  were calculated from the slopes of the layer thickness versus squared treatment time graphs (Fig. 5). The relationship between the growth rate constant and the temperature can be expressed by an Arrhenius type equation such as:

$$K = K_0 \exp\left(-\frac{Q}{RT}\right), \quad (9)$$

where  $Q$  is the activation energy (J/mol);  $T$  is the absolute temperature (K) and  $R$  is the gas constant (J/mol K). Figure 6 shows the plot of the growth rate constant of the plasma paste boride layer on the steel in mixtures with different percentages of borax as a function of temperature. Consequently, the activation energy for the boron diffusion in the boride layer is determined by the slope obtained by the plot of  $\ln K$  vs.  $1/T$ . Making use of the least-squares analysis, the kinetics conclusions are obtained as:



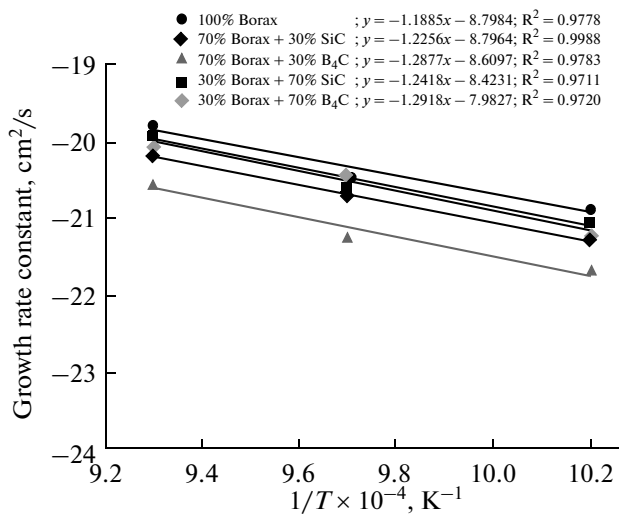
**Fig. 5.** Square of boride layer thickness in different % borax mixtures versus boronizing time at various temperatures for AISI 8620 steel: (a) 100% borax, (b) 70% borax + 30% SiC, (c) 70% borax + 30% B<sub>4</sub>C, (d) 30% borax + 70% SiC, (e) 30% borax + 70% B<sub>4</sub>C.

$$K_{Borax} = \exp\left(-\frac{99.773}{RT}\right), \quad (10)$$

$$K_{70\% Borax + 30\% B_4C} = \exp\left(-\frac{108.088}{RT}\right), \quad (12)$$

$$K_{70\% Borax + 30\% SiC} = \exp\left(-\frac{101.621}{RT}\right), \quad (11)$$

$$K_{30\% Borax + 70\% SiC} = \exp\left(-\frac{104.392}{RT}\right), \quad (13)$$



**Fig. 6.** Growth rate constant vs. temperature of % borax paste mixtures of plasma paste boronized the steel and correlation coefficients ( $R^2$ ).

$$K_{30\% \text{ Borax} + 70\% \text{ B}_4\text{C}} = \exp\left(-\frac{106.240}{RT}\right), \quad (14)$$

for the temperature range of 973 K to 1073 K. Table 2 shows the growth rate constant ( $K$ ) and activation energy ( $Q$ ) as a function of boronizing temperature

and on the samples boronized with different percentages of borax paste mixtures. Together with the increase of temperature from 973 K to 1073 K, an increase occurred in the diffusion coefficient values of boron in the boride layer. Since the diffusion process speeds up along with the increase in temperature, boride layers were formed. The samples boronized with borax paste with SiC and B<sub>4</sub>C were observed to have a decreased diffusion coefficient. B<sub>4</sub>C did not have much contribution since the plasma paste boronizing process was conducted in the lower temperature of 973 K. However, with the temperature increased to 1073 K, B<sub>4</sub>C was found to have contributed to the boronizing treatment. While the highest activation energy was observed to be 108.088 kJ/mol in 70% borax + 30% B<sub>4</sub>C paste, the lowest was obtained from 100% borax paste at 99.773 kJ/mol.

Diffusion coefficient values obtained through plasma paste boronizing methods were established as higher compared to those obtained through the traditional boronizing methods. At the end of plasma paste boronizing, diffusion coefficient values were observed to change in the range of  $2.8 \times 10^{-10}$  and  $22.9 \times 10^{-10}$ . The calculated diffusion coefficient of the present study is in good agreement with values reported in the literature [33, 34].

**Table 2.** Growth rate constant ( $K$ ) and activation energy ( $Q$ ) as a function of boronizing temperature and steel

Steel	Growth rate constant, $\text{cm}^2 \text{s}^{-1}$			Activation energy, $\text{kJ mol}^{-1}$ ( $\text{kJ mol}^{-1}$ )
	Temperature, K			
	973	1023	1073	
100% Borax	$6.6 \times 10^{-10}$	$12.1 \times 10^{-10}$	$20.9 \times 10^{-10}$	99.773
70% Borax+ 30% SiC	$5.3 \times 10^{-10}$	$9.7 \times 10^{-10}$	$17.1 \times 10^{-10}$	101.621
70% Borax + 30% B <sub>4</sub> C	$2.8 \times 10^{-10}$	$5.5 \times 10^{-10}$	$9.9 \times 10^{-10}$	108.088
30% Borax+ 70% SiC	$5.4 \times 10^{-10}$	$10.2 \times 10^{-10}$	$18.2 \times 10^{-10}$	104.392
30% Borax + 70% B <sub>4</sub> C	$6.7 \times 10^{-10}$	$12.8 \times 10^{-10}$	$22.9 \times 10^{-10}$	106.240

**Table 3.** The comparison of activation energy for diffusion of boron with respect to the different boronizing medium and steel

Steel	Temperature range, K	Boronizing medium	Activation energy, $\text{kJ mol}^{-1}$	References
AISI 52100	1123–1223	Solid	269.6	2
AISI 440C			340.4	
AISI 304	1023–1223	Plasma paste	123	10
AISI H13	1073–1223	Salt bath	244.3	16
Mild Steel	973–1273	Spark Plasma Sintering (SPS)	145.8	22
AISI 1045	1193–1273	Paste	226.7	34
AISI 4140	1123–1223	Solid	215	35
31CrMoV4	1123–1223	Solid	230	36
AISI D2	1123–1273	Electrochemical	137.8	37
Low carbon steel	1023–1173	Electrochemical	175.5	38
AISI 8620	973–1073	Plasma paste (borax based pastes)	99.7–108.8	Present study

The kinetics results of the present study are effectively comparable with Refs. as seen in Table 3. Activation energy values obtained through plasma paste boronizing method were established as lower compared to those obtained through traditional boronizing methods. Yoon et al. [10] boronized AISI 304 steel by plasma paste consisting of borax ( $\text{Na}_2\text{B}_4\text{O}_7$ ) and amorphous boron, at different temperatures in Ar/H<sub>2</sub> gases and examined the diffusion kinetics and morphology of the layer. They reported that using the plasma paste process caused lower activation energy for the formation of the boride layer than that obtained when using conventional boronizing processes. The calculated activation energy of the present study is in good agreement with values reported in the literature [10, 16, 22, 34–38].

#### 4. CONCLUSIONS

The following conclusions can be drawn:

1. The AISI 8620 steel was successfully paste boronized in 70% H<sub>2</sub>–30% Ar gas mixture plasma at temperatures of 973, 1023 and 1073 K for 2, 5 and 7 h with various borax paste mixtures.
2. Maximum values of boride layer thickness (91 μm) were obtained for 100% borax.
3. The hardness of boride layers on the plasma paste borided specimens with borax paste mixtures were varied between 1598–1976 HV<sub>0.05</sub>.
4. FeB + Fe<sub>2</sub>B phases were obtained after plasma paste boriding as a result of X-ray diffraction analysis.
5. The lowest activation energy was obtained at 99.773 kJ/mol in 100% borax paste.

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