

Wear Behaviour of Plasma Paste Boronized of AISI 8620 Steel with Borax and B₂O₃ Paste Mixtures

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In the present study, AISI 8620 steel was plasma paste boronized (PPB) by using paste mixtures with different borax and B₂O₃ percentages. The PPB process was carried out in a DC plasma system at 973, 1023 and 1073 K for 5 h in a gas mixture with 70% H₂–30% Ar under a constant pressure of 10³ Pa (10 mbar). The thickness of boride layers ranged from 15.9 to 53.6 μm depending on the temperature and process time. The hardness of the borides formed on the surface of the steel substrate and unborided steel substrate were 1648–1924 HV_{0.05} and 226 HV_{0.05}, respectively. X-ray diffraction analysis of the boride layers on the surface of the samples were obtained for FeB and Fe₂B phases. The wear tests on the steel were performed by using a ball-on-disc test device at room temperature with applied load of 10 N and with a shear velocity of 0.2 m/s. Wear test results showed that the highest value of wear rate was observed in the unborided sample while the lowest wear rate was observed in the PPB samples with 30% borax + 70% B₄C paste at 1073 K for 5 h.

KEY WORDS: Coating; Borax; B₂O₃; Wear; Coefficient of friction

1. Introduction

Boronizing is a thermo-chemical diffusion process through which boron is diffused into steel at high temperatures. In this diffusion process, a ferro-boron layer composed of a single-phase Fe₂B or two-phase Fe₂B + FeB is formed. Formation of the Fe₂B single-phase occurs more often than the two-phase Fe₂B + FeB layer. The FeB phase, which is rich in boron, is more brittle; therefore formation of the FeB phase is not favoured. The coefficient of thermal expansion of FeB ($23 \times 10^{-6}/^{\circ}\text{C}$) is higher than that of Fe₂B ($7.85 \times 10^{-6}/^{\circ}\text{C}$). Due to the difference in thermal expansion between Fe₂B and FeB phases formed in the two-phase system, cracks are observed on the surface. It is possible to prevent these cracks from forming by reducing the amount of the FeB phase or by avoiding its formation. Boronizing can also be applied to non-ferrous metals and alloys^[1–4]. Boronizing is carried out through methods such as solid, liquid, gas, plasma and ion implantation. Solid boronizing is generally performed with patent-protected agents that consist of

approximately 5% B₄C, 5% KBF₄ and 90% SiC (commercial Ekabor®). Although among these methods pack boronizing is used commonly for commercial purposes, its drawbacks are higher treatment temperatures and longer periods of processing^[4–6]. In liquid boronizing a firmly adhering salt layer forms on the workpieces which can be quite costly to be removed after boronizing has been completed. Traditional gaseous boronizing agents which are very sensitive to even the slightest traces of moisture are very poisonous, more costly and prone to exploding. The plasma boronizing process has a superior advantage as compared to conventional boronizing processes. However the gases (B₂H₆, BCl₃) used in plasma boronizing, which are expensive, poisonous and have explosive characteristics, are of disadvantages^[6–9]. The disadvantages in the gas and plasma boronizing process can be eliminated through the plasma paste boronized (PPB) surface process. What makes this process advantageous is the fact that the paste used consists of boron raw materials which are harmless to the environment, and the gases generally have inert characteristics, such as hydrogen, argon and nitrogen^[9–12]. The number of studies on plasma boronizing has increased in recent years^[12–14]. The plasma boronizing process has far more advantages compared to traditional boronizing processes. For example, due to the high energy generated by plasma boronizing process, the applications are performed at lower temperatures than traditional boronizing processes and distortion (shape distortion) can be reduced to the minimum^[14–16].

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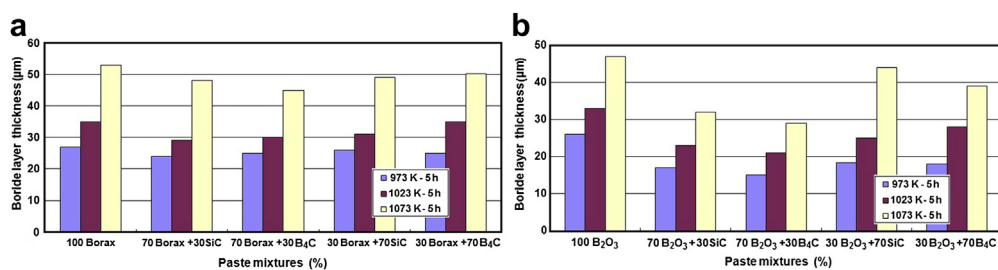


Fig. 1 Variation of thickness of boriding layer with time at different boriding temperatures: (a) borax paste mixtures, (b) B₂O₃ paste mixtures.

In this study, AISI 8620 steel was PPB in paste mixtures with different borax and B₂O₃ percentages, and the wear behaviour of these borided samples was examined. From the results of the wear tests, the effect of the PPB process on the friction coefficient, surface roughness and wear rate was studied. Worn surfaces of the samples were characterised by scanning electron microscopy (SEM), and the results were compared with those obtained from PPB samples.

2. Experimental

2.1. Material and plasma paste boriding

The substrate material, AISI 8620 steel, essentially contained: 0.19 wt% C, 0.4 wt% Cr, 0.7 wt% Mn, 0.4 wt% Ni and balance Fe. The samples were cylindrical with a diameter of 18 mm and thickness of 6 mm. AISI 8620 steel samples were ground by 800 mesh SiC paper and polished with a 0.1 µm alumina suspension to obtain a smooth surface. In this study, borax, B₂O₃, SiC and B₄C powder mixtures with various percentages were used as a paste. SiC and B₄C were added to borax and B₂O₃ pastes as it is otherwise difficult to clean the remains of borax and B₂O₃ pastes from the surface of samples borided with 100% borax and 100% B₂O₃ pastes, then prolongs the process. PPB was carried out in a DC-pulsed PPB system^[12] with a power supply frequency of 5 kHz and duty cycle of 80%. The samples were placed in the PPB chamber and the chamber was evacuated to 2 Pa (2×10^{-2} mbar). PPB samples were cleaned with hydrogen sputtering for 30 min and were then PPB for 5 h at 973, 1023 and 1073 K under a constant pressure of 10³ Pa (10 mbar) in gas mixtures of 70% H₂ + 30% Ar. The PPB temperature was measured by using a chromel–alumel thermocouple, placed at the bottom of the PPB samples.

2.2. Surface characterization

Cross-sections of PPB steels were prepared metallographically to observe the morphological details by using a BX60 Olympus microscope. The thickness of the layers formed on the steels was measured by an optical micrometer attached to the optical microscope. X-ray diffractograms were obtained by using a copper tube source through the conventional Bragg–Brentano ($\theta-2\theta$) technique with symmetric geometry with monochromatised CuK α radiation. The hardness of the boride layers was measured on the cross-sections by using a Micro-Vickers indenter (Shimadzu HVM-2) with a 50 g load.

2.3. Friction and wear tests

Wear tests of the PPB steels were performed by using a ball-on-disc test device. In the present study, the bottom movable flat surface was the treated steel and the upper fixed surface was the WC–Co balls with diameter of 8 mm. Errors caused by the distortion of the surface were eliminated by using a separate abrasion element (WC–Co ball) for each test. The wear experiments were carried out in a ball-disc arrangement under dry friction condition at room temperature with an applied load of 10 N and with a sliding velocity of 0.2 m/s at sliding distance of 500 m. Before and after each wear test, each sample and abrasion element was cleaned with alcohol. After the test, the wear volumes of the samples were quantified by multiplying the cross-sectional areas of the wear by the width of wear track obtained from the Tribotechnic Rugosimeter device. Wear rate was calculated with the following formula:

$$W_k = \frac{W_v}{MS} \quad (1)$$

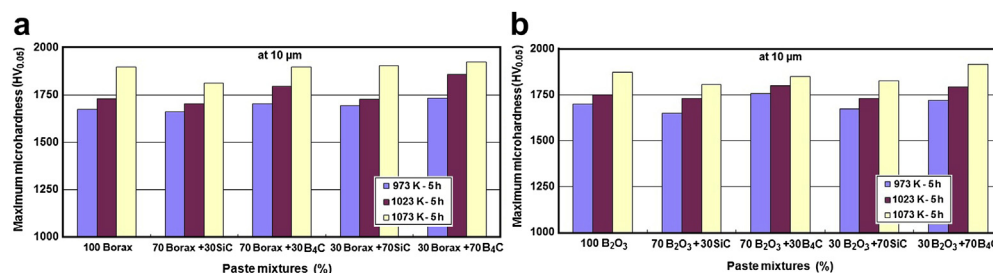


Fig. 2 Variation of microhardness of boride layer with time at different boriding temperatures: (a) borax paste mixtures, (b) B₂O₃ paste mixtures.

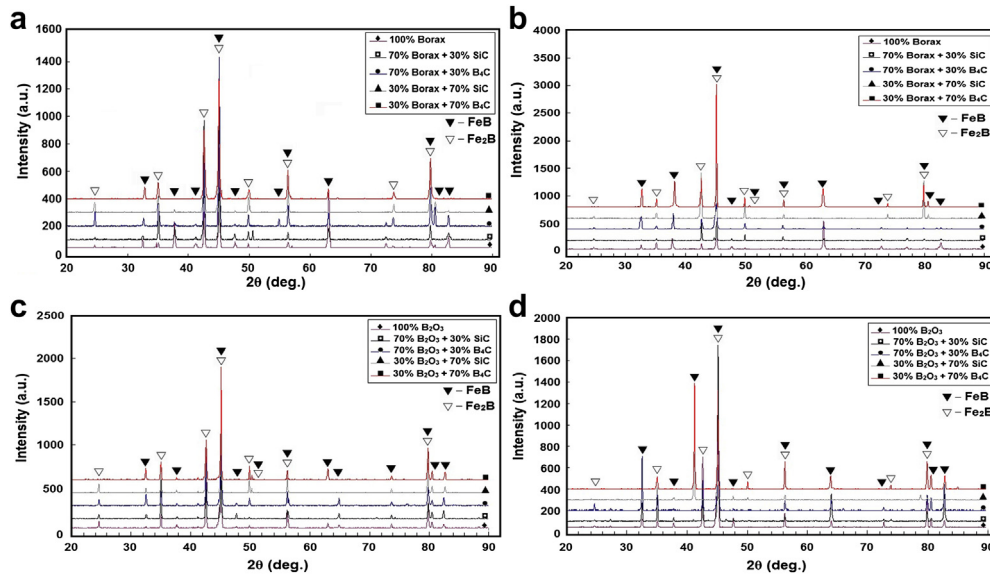


Fig. 3 XRD patterns of the samples plasma paste borided in different paste mixtures: (a) borax at 973 K, (b) borax at 1073 K, (c) B_2O_3 at 973 K, (d) B_2O_3 at 1073 K.

where W_k is the wear rate, W_v the worn volume, M the applied load and S is the sliding distance.

Coefficient of friction (COF) with respect to sliding distance was obtained through a COF program. As a result of the wear tests, graphs of the COF, surface roughness and wear rate were plotted against the temperature of the PPB. Surface profiles of the wear depth tracks on the samples and surface roughness were measured by a Tribotecnica Rugosimeter. The worn surfaces of the samples were characterised by SEM and the results were compared with those obtained for PPB samples by using different borax and B_2O_3 paste mixtures.

3. Results and Discussion

3.1. Surface characterization

The thickness of the boride layer formed on the PPB samples is shown in Fig. 1(a) and (b). The thickness of the boride layer increases with increasing boriding temperature. The largest thickness of boride layer was achieved with 100% borax, while the minimum thickness was achieved with the 70% B_2O_3 + 30% B_4C paste mixture. The thickness of the boride layer increases with increasing boriding temperature for all paste mixtures. Almost the same thickness of boride layer is obtained for same paste mixtures and temperature.

The hardness of borides formed on the surface of steel substrate and unborided steel substrate are 1648–1924 $HV_{0.05}$ and

226 $HV_{0.05}$, respectively. The hardness values of borides formed on the borided samples according to the hardness values of the unborided sample substrates, vary between 7.29 and 8.51 times with respect to the boriding temperature, as shown in Fig. 2(a) and (b). The lowest hardness value obtained is 1648 $HV_{0.05}$ for 70% B_2O_3 + 30% SiC paste mixture for 5 h at 973 K, and the highest hardness value is 1924 $HV_{0.05}$ for 30% borax + 70% B_4C paste for 5 h at 1073 K. For all paste mixtures, the hardness of the boride layer increases with increasing boriding temperature. Boride paste mixtures do not affect the boride layer hardness. Hardness values obtained are close to each other. Boriding temperature was found to be more effective than the paste mixture with regard to boride layer hardness. Ozbek and Bindal^[17] borided AISI W4 steel by Ekabor powders and found that higher boriding temperatures resulted in higher hardnesses due to the formation of the harder FeB phase.

X-ray diffraction (XRD) patterns of paste borided AISI 8620 steel by plasma with different borax and B_2O_3 paste mixtures are given in Fig. 3(a)–(d). XRD analysis of the boride layers on the surface of the samples reveals the peaks of FeB and Fe_2B . The boride layers mainly consist of intermetallic phases (FeB and Fe_2B) as a result of the diffusion of boron atoms from the boriding compound to the metallic lattice with respect to the holding time. The properties of these boride layers are known to be affected to a large extent by presence of these phases^[18,19]. While an increase is observed in the intensity of the FeB phase with increasing PPB temperature, a decrease occurs in the

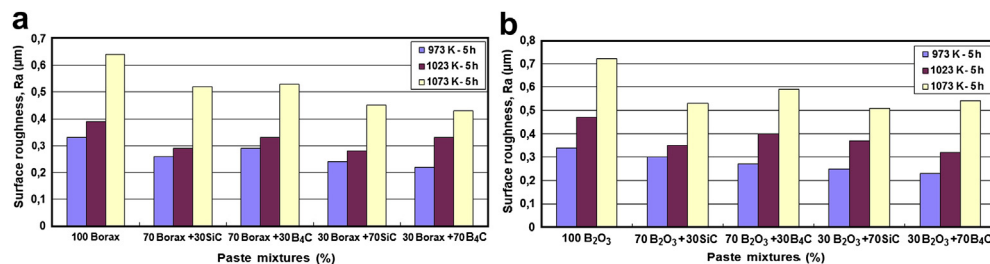
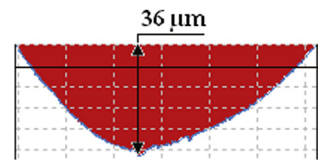


Fig. 4 Variation in the surface roughness of the plasma paste borided AISI 8620 steel depending on plasma paste boriding temperature: (a) borax paste mixtures, (b) B_2O_3 paste mixtures.

Table 1 Wear track depth of the plasma paste borided and unborided AISI 8620 steels in borax and B₂O₃ paste mixtures

Paste mixtures	Boride temperature (K)	Boride time (h)	Boride layer thickness (µm)	Wear track depth (µm)
100% borax	973	5	27	4
	1023	5	35	3.5
	1073	5	53	1.5
70% borax + 30% SiC	973	5	24	6.5
	1023	5	29	1.9
	1073	5	48	1.4
70% borax + 30% B ₄ C	973	5	25	4.4
	1023	5	30	3.1
	1073	5	45	1.7
30% borax + 70% SiC	973	5	26	4.4
	1023	5	31	4
	1073	5	49	1.4
30% borax + 70% B ₄ C	973	5	25	5
	1023	5	35	3
	1073	5	50.2	1.2
100% B ₂ O ₃	973	5	26	4.5
	1023	5	33	2.6
	1073	5	47	1.8
70% B ₂ O ₃ + 30% SiC	973	5	17	4.8
	1023	5	23	3
	1073	5	32	2.2
70% B ₂ O ₃ + 30% B ₄ C	973	5	15	5.2
	1023	5	21	3.8
	1073	5	30	2.3
30% B ₂ O ₃ + 70% SiC	973	5	18.2	5.5
	1023	5	25	4
	1073	5	44	2.6
30% B ₂ O ₃ + 70% B ₄ C	973	5	18	5.3
	1023	5	28	2.6
	1073	5	39	1.8
Unborided AISI 8620	—	—	—	—



intensity of the Fe₂B phase, as shown in Fig. 3. Uslu et al.^[20] borided AISI P20 steel using Ekabor 2 powders and found that higher boriding temperatures resulted in the FeB phase.

During PPB, B₂O₃ and borax react with active hydrogen (H⁺) in a glow discharge, the reaction occurs as follows:

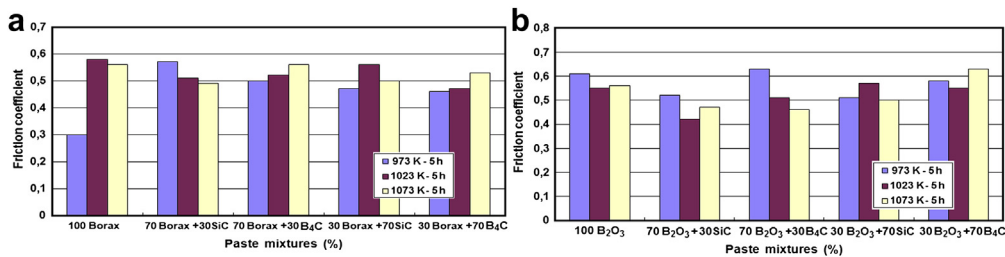
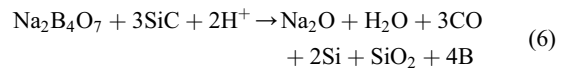
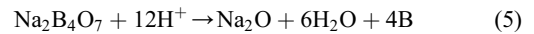
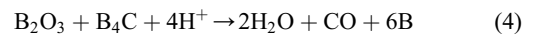
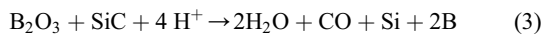
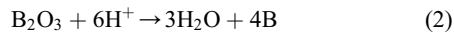


Fig. 5 Variation in the friction coefficient of the plasma paste borided AISI 8620 steel depending on plasma paste boriding temperature: (a) borax paste mixtures, (b) B₂O₃ paste mixtures.

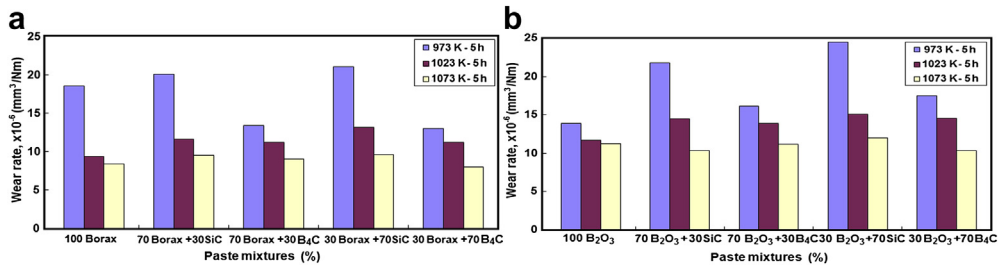
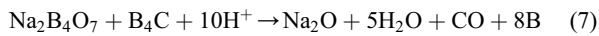
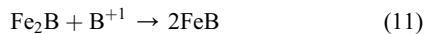
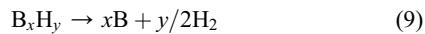
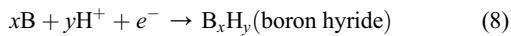


Fig. 6 Wear rate of the plasma paste borided AISI 8620 steel depending on the temperature of the plasma paste boriding: (a) borax paste mixtures, (b) B_2O_3 paste mixtures.



When the temperature of plasma is increased to about 1073 K, the B_2O_3 and borax powders are firstly melted and then atomic boron (B) is formed. Atomic boron is produced through the decomposition of the boron hydride (B_xH_y) from the paste, and this atomic boron becomes the active boron, B^{+1} , within the molten B_2O_3 and borax, or in the glow discharge. Finally, this active boron, B^{+1} , diffuses and reacts with Fe to form the boride layer^[12].



The graphs of variations in the surface roughness of the PPB samples in different borax and B_2O_3 paste mixtures with respect to boriding temperature can be seen in Fig. 4(a) and (b). It is determined that the surface roughness values of unborided samples (R_a , 0.18) would increase with increasing PPB temperature. The surface roughness of the coating increases

relatively from approximately 0.23 to 0.71 μm due to the paste composition. Surface roughness values of borided steel increase with increasing boriding temperature and time. This result indicates that the increase in boride coating thickness affects the surface roughness. The relationship between roughness and thickness in this study is in good agreement with that of many coating methods^[21–24]. This case can be attributed to the intrinsic properties of the phases formed. But it is difficult to clarify the reason for the relation between roughness and phases in boride layers revealed in this experiment, so it is not discussed here.

3.2. Wear and friction

Results of the wear tests, boride layer thickness and wear track depth of the PPB and unborided AISI 8620 steels in borax and B_2O_3 paste mixtures obtained from Tribotechnic Rugosimeter device are given in Table 1. With the increase in temperature of the PPB process, a decrease in the wear track depth was observed. The highest value of wear rate was observed in the unborided sample, while the lowest wear rate was observed in the PPB samples with 30% borax + 70% B_4C paste at a temperature of 1073 K for 5 h. Reviewing Table 1, it is clear that wear tracks deepen in the borided samples as the boriding temperature decreases. It is clear that the boride layer has the highest hardness as it presents the shallowest track depth^[25].

The effect of paste mixture and temperature on the COF of PPB samples can be seen in Fig. 5. COF values of the PPB steel samples featured boride layers are lower than those of the unborided steel samples. While the COF value of the unborided

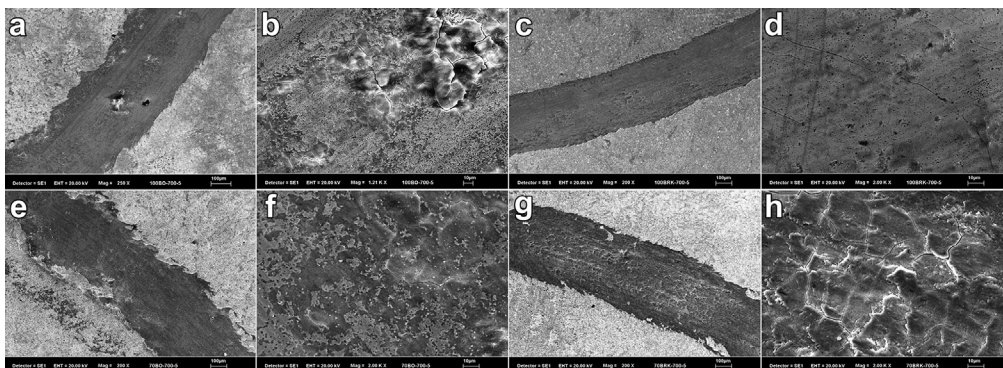


Fig. 7 Worn surface SEM images of AISI 8620 steel plasma paste borided at 973 K for 5 h: (a) and (b) 100% B_2O_3 , (c) and (d) 100% borax, (e) and (f) 70% B_2O_3 + 30% B_4C , (g) and (h) 70% borax + 30% B_4C .

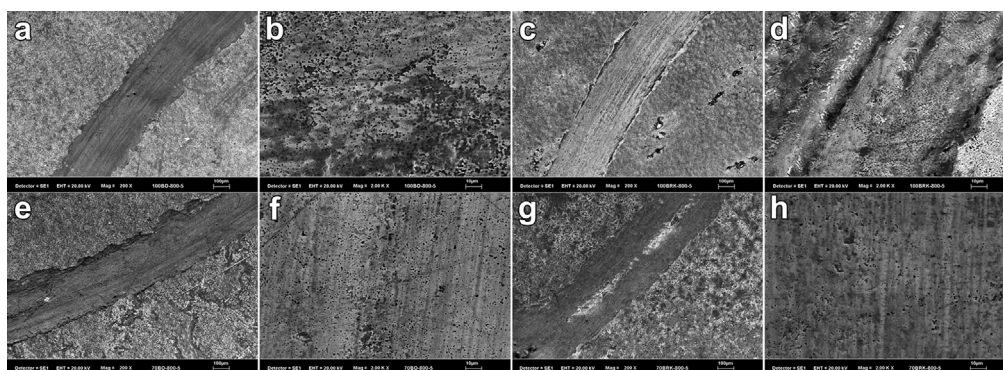


Fig. 8 Worn surface SEM images of AISI 8620 steel plasma paste borided at 1073 K for 5 h: (a) and (b) 100% B_2O_3 , (c) and (d) 100% borax, (e) and (f) 70% B_2O_3 + 30% B_4C , (g) and (h) 70% borax + 30% B_4C .

sample was 0.72, a decrease in COF values as a result of the boriding process was observed. The COF values of plasma borided samples (Fig. 5(a) and (b)) are found to vary between 0.54 and 0.66. The COF values obtained from borax and B_2O_3 paste samples are about the same. Sen et al.^[26] borided AISI 4140 low alloy steel and observed that the COF of unborided (hardened + tempered) and borided steels ranged from 0.50 to 0.60. Taktak^[27] investigated the tribological properties of borided AISI 52100 and 440C bearing steels and determined that the COF values of bearing steels at room temperature ranged between 0.68 and 0.58.

In Fig. 6(a) and (b), the variation in the graphs of the wear rate of PPB AISI 8620 steel in paste mixtures with different borax and B_2O_3 percentages can be seen with respect to the boriding temperature. In the PPB samples in paste mixtures with different borax and B_2O_3 percentages, a decrease in the wear rates with increasing temperature of the boriding process was observed. With increasing temperature in the boriding process, the FeB phase increased more than Fe_2B phase, and this, at the same time, led to an increase in the surface roughness^[28,29]. The highest value of wear rate was observed in AISI 8620 unborided steel. The lowest wear rate was observed in the samples that were plasma borided with 30% borax + 70% B_4C paste at a temperature of 1073 K for 5 h. With this method it is important that the useful life of the material can be extended by achieving a considerable decrease in the wear rate of the AISI 8620 steel. The wear rate of borax paste samples was slightly lower than that of B_2O_3 paste samples. It is well known that the hardness of the boride layer plays an important role in improving the wear resistance. This is due to the high wear resistance of the thicker boride layer formed by an increased boriding period^[30]. The surface microhardness (see Fig. 2) and the wear rate (Fig. 6) of borided samples also confirm that the relative wear resistance is improved with increasing hardness. The wear rates depend closely on the boriding duration. In other words, the longer boriding temperature and duration result in more excellent wear

resistance. It is well known that the hardness of boride layer plays an important role in improving the wear resistance. This is in agreement with the previous studies reported in literature^[25,31,32]. The borided samples showed wear resistance for a long period of time due to the fact that the boride layer had a higher hardness and low COF^[33,34].

Worn surface SEM micrographs of the PPB AISI 8620 steel under the 10 N load can be seen in Fig. 7. As can be seen in the surface morphology of the wear regions, during the wear process of the samples borided at 973 K, cracks are observed on the boride layer, and as a result of the cracks spreading delamination of the boride layer occurs.

Fig. 8(a)–(h) shows the SEM surface morphologies of the wear process of the PPB samples with 100% B_2O_3 , 100% borax, 70% B_2O_3 + 30% B_4C and 70% borax + 30% B_4C pastes at 1073 K for 5 h, respectively. The wear track thickness of the samples borided at 1073 K is lower than that of the samples borided at 973 K. This is due to the fact that the hardness and thickness of the borided layers are higher. Borided steels are extremely resistant to abrasion on account of their great hardness^[29,35]. It is observed that in all borided steels, a rather low level of wear occurs. Furthermore, it is observed that the burrs on the surface are reamed and many small pores are created. By examining Figs. 7 and 8, it is clear that the wear surfaces of the borided AISI 8620 are roughened and debris particles and cracks (shown with arrows) are present, indicating brittle and fine delamination fractures on the worn surface of the boride coating. Due to the differences in internal stress of the steels with the two different boride layers (FeB and Fe_2B), it is possible to observe the separation of boride layers, the advance of cracks through these regions and debris particles leading to new abrasive wear on the boride layer. In other studies, it has been reported that the wear resistance of two different non-homogeneous boride layers is much worse than that of a single-layer and it is only the wear resistance of the Fe_2B layer that is more highly resistant than that of the two-layer structure^[36–38].

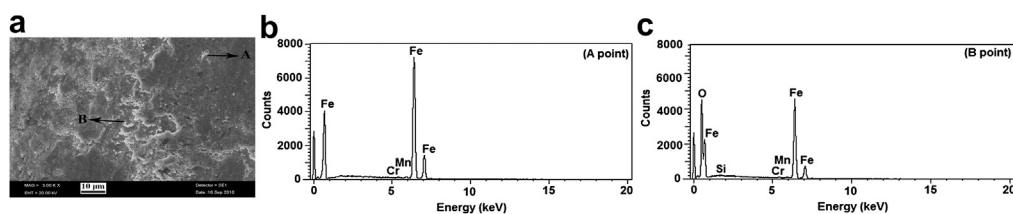


Fig. 9 (a) SEM micrograph of worn surfaces for the unborided AISI 8620 steel, (b) and (c) EDS spectra of points A and B in (a), respectively.

Fig. 9(a)–(c) shows typical morphologies of the worn surface of the unborided AISI 8620 steel and the EDS spectrum of illustrated points with A and B. Fe-based oxide layers form as a result of the wear test. The spallation of the oxide layers in the sliding direction and their orientation extending along the wear track are identified. When the SEM images of the worn surfaces of the unborided samples are examined, it can be seen that the wear marks in Fig. 9(a) are larger and deeper.

4. Conclusion

In this study, AISI 8620 steel was PPB in paste mixtures with different borax and B₂O₃ percentages, and the wear behaviour of these borided samples was examined. Boride layer thickness obtained in all borax and B₂O₃ paste mixtures varied between 26–53.6 and 15.9–45 μm, respectively. A double phase (FeB + Fe₂B) was obtained after PPB as determined by XRD analysis. The hardness of the boride layers on the PPB samples with borax and B₂O₃ paste mixtures varied between 1648–1916 HV_{0.05} and 1661–1924 HV_{0.05}, respectively. Surface roughness of the coating increased from approximately 0.23 to 0.71 μm with respect to time of boriding. Coefficient of friction values (0.54–0.66) for the PPB samples were lower than that (0.72) of the unborided steel sample.

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