

## Investigation of tribological behaviour of plasma paste boronized of AISI 8620, 52100 and 440C steels

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In the present study, AISI 8620, 52100 and 440C steels were plasma paste boronized (PPB) by using 100% borax paste. PPB process was carried out in a dc plasma system at temperature of 700 and 800°C for 3 and 5 h in a gas mixture of 70%H<sub>2</sub>-30%Ar under a constant pressure of 4 mbar. Wear tests of the bearing steels were performed using a ball-on-disc test device. The applied loads on the ball were 5 and 20 N and the sliding time was 60 min for each test. Wear tests were carried out in the unlubricated and lubrication conditions at room temperature in air. Wear volume of the disc specimen was determined from the cross-sectional area of the wear track, obtained perpendicularly to the sliding direction. Worn surfaces of the samples were PPB characterized by scanning electron microscopy and the results were compared with those obtained for plasma paste boronized specimens.

**Keywords:** Plasma paste boronizing, Wear, Borax

Boronizing has been a considerably important surface hardening process for increasing the wear resistance of steels during recent years. Boronizing is a thermo-chemical surface hardening process in which boron atoms are diffused into the surface of a metal to form a hard layer of metal borides<sup>1,2</sup>.

Boronizing process involves heating the material in the range of 700-1000°C should be for 1-12 h, in contact with a boronaceous solid powder, paste, liquid, gaseous, plasma, plasma paste boronizing and fluidized bed boronizing<sup>3,4</sup>. Boronizing has been found to be an effective method for significantly increasing the surface hardness. Boronizing of steels is used against adhesive, sliding and abrasive wear and is recognized as an effective method to combat these effects<sup>5</sup>.

Depending on process temperature, chemical composition of substrate materials, boron potential of medium and boronizing time, single-phase Fe<sub>2</sub>B or two intermetallic phases (FeB, Fe<sub>2</sub>B) are obtained by diffusing boron atoms into the surface of metallic materials<sup>6</sup>. Since, the FeB phase is more brittle and harder, and has a higher coefficient of thermal expansion than the Fe<sub>2</sub>B one, the cracking of the double-phase boride layer is often observed<sup>7,8</sup>. The borided phases must fulfill properties that retard or

modify the proposed failure mechanisms in engineering components, which include low adhesion between the tool and the machining material, chemical stability, high hardness and abrasion resistance<sup>9-11</sup>.

Studies have been carried out in order to reduce the boronizing temperature and time for the last 40 years<sup>12</sup>. Pack boronizing is generally performed with patent protected agents that consist of approximately 5%B<sub>4</sub>C, 5%KBF<sub>4</sub> and 90%SiC (commercial Ekabor®). Although pack boronizing is used commonly for commercial purposes among these methods, higher treatment temperatures and longer periods of time constitutes its drawbacks<sup>13-15</sup>. Liquid boronizing, a firmly adhering salt layer forms on the workpieces and this can be quite costly to remove after boronizing has been completed<sup>16</sup>.

Lately, studies carried out on plasma boronizing (PB) have been increasing gradually<sup>17-19</sup>. PB process has a superior advantage when compared to conventional boronizing techniques because of its economical efficiency, lower processing temperature and easy control of experimental parameters<sup>13,17,18</sup>. However, the gases (B<sub>2</sub>H<sub>6</sub>, BCl<sub>3</sub>) used in plasma boronizing, which are expensive, poisonous and explosive characteristics, is a disadvantage. Moreover, in plasma boronizing process carried out in BCl<sub>3</sub> environment, the boride layer having pores

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poses a tremendous problems<sup>18-20</sup>. The disadvantages in PB process can be eliminated through plasma paste boronizing (PPB) surface process. In this study, the paste used having environment-friendly boron raw materials and gases generally being hydrogen, argon and nitrogen which have inert characteristics make this process advantageous. Plasma paste process caused lower activation energy for the formation of the boride layer than that for the conventional boronizing processes<sup>4</sup>. It is possible that formation of boride layer at lower temperatures in plasma is more effective than the other conventional processes. Boronizing temperatures and time decrease for plasma-assisted boronizing and boride layer forms at lower temperatures<sup>4, 22-24</sup>.

The wear of boronized steels by using various boronizing processes has been evaluated by a number of investigators. However, there is very little literature about wear of paste boronized steels in plasma environment. In this study, the steels were dried at room temperature for 24 h after approximate one millimeter of borax paste covered the samples. The prepared samples were placed in the vacuum container. Plasma paste boronizing treatment was performed in a dc plasma system, at gas mixtures of Ar and H<sub>2</sub> by using 100% borax paste. The microhardness of the specimens was measured on the surface and along the depth of the alloyed layer. Their wear resistance is estimated ball on disk tests. Worn surfaces of the samples were characterized by optical microscopy and scanning electron microscopy and the results were investigated with those obtained for plasma paste boronized specimens.

## Experimental Procedure

### Materials and plasma paste boronizing

For PPB treatment we used the circular AISI 8620, 52100 and 440C steel samples with size of Ø20×6 mm. The compositions of the AISI 8620, 52100 and 440C steels are presented in Table 1. The samples were mechanically polished up to 0.1 µm diamond paste in order to obtain a final mirror finish, and cleaned in alcohol. All the samples before PPB treatments covered by borax paste.

PPB was carried out in a dc-pulsed PPB system with power supply frequency of 5 kHz and duty cycle of 80%. The specimens were placed into the PPB chamber and the chamber was evacuated to  $2.5 \times 10^{-2}$  mbar. PPB were subjected to cleaning by hydrogen sputtering for 30 min and the specimens were then PPB at 700°C and 800°C under a constant pressure of 4 mbar at gas mixtures of 30%Ar+70%H<sub>2</sub>. The PPB temperature was measured using a chromel-alumel thermocouple, placed at the bottom of the plasma paste boronized samples. The exposure times were 3 and 5 h, while a 100% borax paste was used to cover one side of the material surface.

### Wear and friction tests

Wear tests of the bearing steels were performed 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 ball with diameter of 8 mm. The ball was fixed and the disk sample was rotated at the speed of 300 rpm. The applied loads on the ball were 5 and 20 N and the sliding time was 60 min for each test. Wear tests were carried out in the unlubricated and lubrication conditions at room temperature in air. The friction force was detected by a load cell through a friction force measurement arm. The friction coefficient was continuously recorded during the tests. Surface profiles of the wear tracks on the discs were measured by a surface profilometer. Wear volume of the disc specimen was determined from the cross-sectional area of the wear track, obtained perpendicularly to the sliding direction. Worn surfaces of the samples were characterized by optical microscopy and scanning electron microscopy and the results were investigated with those obtained for plasma paste boronized specimens.

## Results and Discussion

### Surface characterization

Table 2 shows the boride layer thicknesses formed on PPB steels. In addition, the boride layer thicknesses of pack boronized steels are given in Table 2 for comparison. As seen in the Table 2, any measurable thickness of the boride layer was not

Table 1—Chemical compositions of AISI 52100, 440C and AISI 8620 steels (wt %).

Steels	C	Cr	Ni	Si	Mn	Mo	S	P
AISI 52100	0.95	1.45	0.107	0.194	0.318	0.014	0.0076	0.0143
AISI 8620	0.2	0.4	0.4	0.15	0.7	0.15	0.04	0.03
AISI 440C	0.91	16.5	0.318	0.42	0.417	0.46	0.001	0.024

Table 2—The boride layer thicknesses formed on plasma paste boronized steels at various temperatures and times

Steels	Boride layer thickness, $\mu\text{m}$			
	700°C		800°C	
	3 h	5 h	3 h	5 h
AISI 8620	27	30	36	44
AISI 52100	22	29	38	46
AISI 440C	.....	.....	8	18

observed on all three pack boronized steels at temperature of 700°C. However, except AISI 440C stainless steel, plasma paste boronized AISI 8620 and 52100 had the measurable boride layers at 700°C. The thickness of boride layer increased with the increase in the temperature and time. Additionally, it was seen that the boride layer thickness on the 8620 steel was greater when compared to other steels. At temperature of 800°C, the boride layers formed on both plasma paste boronized three steels. This suggested that boronizing process at lower temperatures and times (700°C) can be performed by PPB that provides the energy and time saving. Yoon *et al.*<sup>4</sup> reported that 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<sup>23,24</sup>.

While a maximum surface hardness of 1827 HV was obtained for the 52100 steel plasma paste boronized at 700°C for 3 h, a hardness value of 1897 HV was obtained at 700°C for 5 h. For the case of 8620 steel, the surface hardness values were 1730 and 1824 HV at same temperature for 3 and 5 h, respectively. The hardness of boride layers on plasma paste boronized steels at 800°C was higher than that at 700°C. It was also noticed that the longer boronizing time results in higher surface hardness values due to the formation of a harder FeB phase<sup>25,26</sup>. The highest hardness value at 800°C was found for 440C (1946 HV for 3 h, 1978 HV for 5 h). Surface hardness values obtained at 800°C in this study are close to values in previous studies carried out at 900°C using pack boronizing for 52100, 440C and 8620 steels<sup>27</sup>.

The roughness values formed on the steel as a consequence of plasma paste boronizing are given in Table 3. As seen in Table 3, increase of boronizing temperature and time in each three steels leads to increase in surface roughness values. This situation is in compliance with previous studies<sup>28</sup>.

Table 3—Surface roughness values in plasma paste boronized steels in various temperatures and times

Steels	Surface Roughness, Ra ( $\mu\text{m}$ )			
	700°C		800°C	
	3 h	5 h	3 h	5 h
AISI 8620	0.226±0.029	0.281±0.034	0.307±0.025	0.35±0.074
AISI 52100	0.222±0.012	0.235±0.065	0.273±0.043	0.384±0.056
AISI 440C	0.151±0.042	0.154±0.054	0.177±0.063	0.322±0.052

### Wear and friction

Wear tests were carried out in the unlubricated and lubrication conditions at room temperature in air. The friction coefficient values with the against WC-Co ball belonging to plasma paste boronized specimens at 700°C and 800°C is illustrated in Figs 1 and 2, respectively.

The friction experiments have been performed in lubrication and unlubrication medium. In Figs 1 and 2, the friction coefficient values against WC-Co ball of the plasma paste boronized samples in 700°C and 800°C in lubrication and unlubrication mediums. As seen from the graphics, the friction coefficient values in lubrication mediums changes between 0.09-0.14. While the friction coefficient changes between 0.09-0.12 in 5 N load, it changes between 0.11-0.14 in 20 N load. As seen in Fig. 2, the friction coefficient of the same samples in dry medium is between 0.39-0.77. Change in PPB temperature does not either cause any significant change in case of unlubrication friction. In case of unlubrication friction, while the friction coefficient changes between 0.39-0.68 under 5 N loads, it changes between 0.55-0.78 in 20 N load. In both temperatures, the lowest friction coefficient has been observed in PPB 52100 for 5 h (0.39). Besides the highest friction coefficient has been recorded as 0.78 for 440C steel.

In Figs 3 and 4, in lubrication and unlubricated mediums, the wear rate values are observed under 5 and 20 N loads for PPB samples in 700°C and 800°C. In the wear experiments performed in lubrication medium, the wear rates changes from  $3.1 \times 10^{-9}$  to  $7.2 \times 10^{-9}$  mm<sup>3</sup>/Nm. Less than 20 N loads, the wear rates are higher as expected. While the lowest wear rate is observed on 52100 steel boronized with plasma paste in 700°C ( $\sim 3 \times 10^{-9}$  mm<sup>3</sup>/Nm), the highest wear rates have been observed in 440C steel boronized with plasma paste in 700°C ( $\sim 7.2 \times 10^{-9}$  mm<sup>3</sup>/Nm).

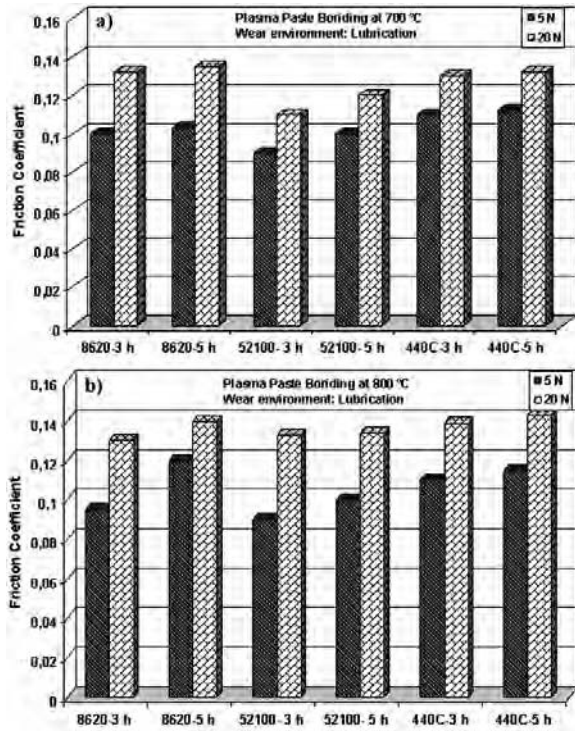


Fig. 1—Change of friction coefficients of PPB AISI 8620, 52100 and 440C steels in lubrication medium against WC-Co ball in various temperatures (a) 700°C and (b) 800°C

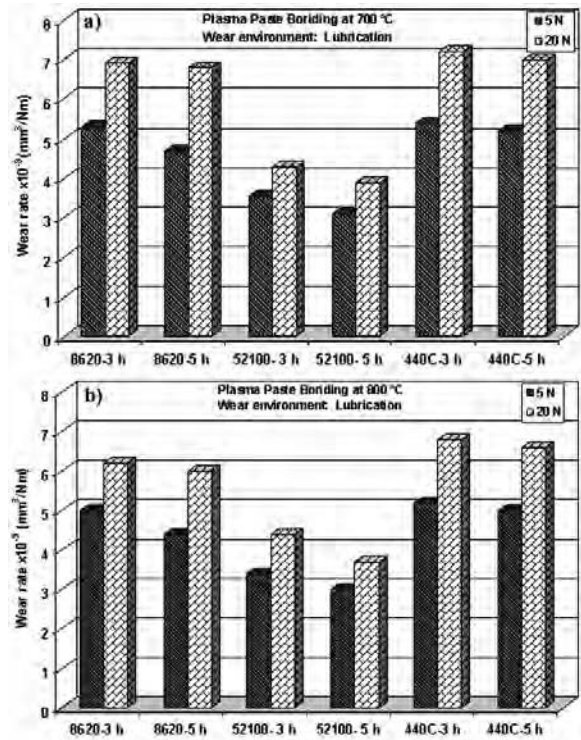


Fig. 3—Change of wear rates of PPB AISI 8620, 52100 and 440C steels in lubricated medium against WC-Co ball in various temperatures (a) 700°C (b) 800°C

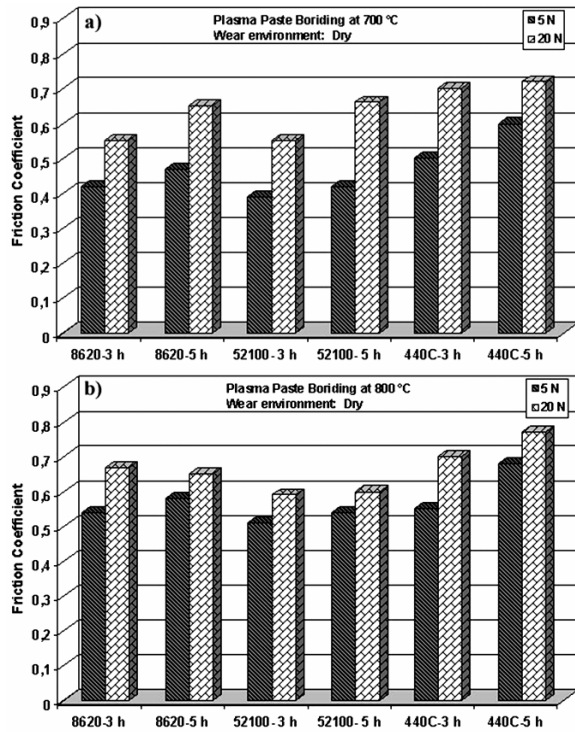


Fig. 2—Change of friction coefficients of PPB AISI 8620, 52100 and 440C steels in unlubricated medium against WC-Co ball in various temperatures (a) 700°C and (b) 800°C

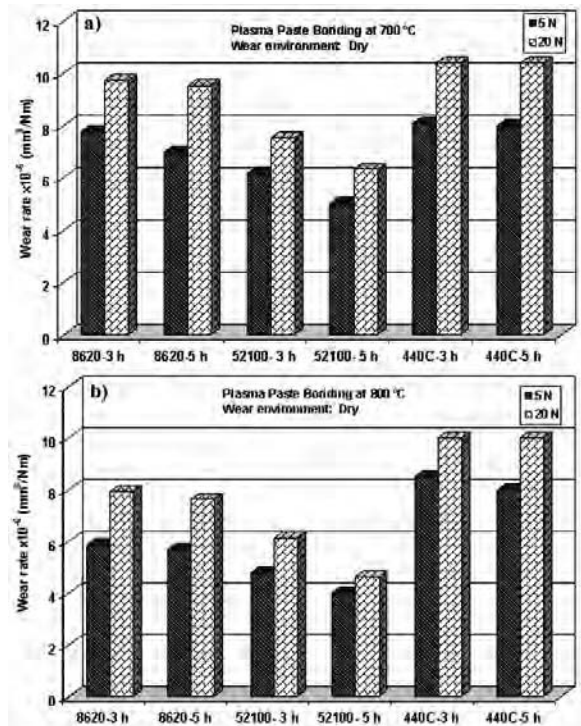


Fig. 4—Change of wear rates of PPB AISI 8620, 52100 and 440C steels in unlubricated medium against WC-Co ball in various temperatures (a) 700°C and (b) 800°C

In the wear experiments performed in unlubrication media, the wear rates are higher under 20 N loads. Under 700°C, PPB 52100 and 8620 steel demonstrates almost similar wear behaviors. While the highest wear rate is observed for 440C steel ( $8.1 \times 10^{-6} \text{ mm}^3/\text{Nm}$  in 5 N load,  $10.4 \times 10^{-6} \text{ mm}^3/\text{Nm}$  in 20 N load), the lowest wear rate has been found for 52100 steel boronized for 5 h ( $5 \times 10^{-6} \text{ mm}^3/\text{Nm}$  in 5 N load,  $7.54 \times 10^{-6} \text{ mm}^3/\text{Nm}$  in 20 N load).

In wear experiments performed in unlubrication medium in PPB steel in 800°C, the highest abrasion speed for 440C steel is ( $\sim 8.5 \times 10^{-6} \text{ mm}^3/\text{Nm}$  in 5 N load,  $\sim 10 \times 10^{-6} \text{ mm}^3/\text{Nm}$  in 20 N load) and the lowest wear rate is found for 52100 steel ( $5 \times 10^{-6} \text{ mm}^3/\text{Nm}$  in 5 N load,  $6.4 \times 10^{-6} \text{ mm}^3/\text{Nm}$  in 20 N load). When the wear rates in both operation temperatures are compared, similar conclusions are observed.

In characterization of the wear traces in lubrication and unlubrication media, only the wear surface morphology of PPB steels in 800°C for 3 h have been examined. In Fig. 5, SEM morphologies of the wear regions in lubrication media against WC-Co ball under 20 N load of each three steel PPB for 3 h at 800°C are seen. It is observed that in each three boronized steel types, a rather low level of wear has occurred. Furthermore, it is observed that the burrs on the surface have been reamed and many small pores have been created. With the increase of the amount of the applied load, the wear rate has increased<sup>5</sup>. From this, we conclude that an increase in process time increases the thickness of the boride layer, which results in a decrease in wear<sup>7, 29, 30</sup>.

SEM morphologies of the wear regions in unlubrication media against WC-Co ball under 5 N load of each three PPB steel types for 3 h in 800°C are seen in Fig. 6. A strong smearing is observed on the edges of the wear trace of boronized 440C steel (Fig. 6a). When the wear trace is magnified, it can be seen that intensive abrasive particles are available in smearing regions (Fig. 6b). In the wear region of the boronized 8620 steel, abrasive particles and cracks on layer are observed (Figs 6c and 6d). In the wear region of boronized 52100 steel, there are cavities formed probably as a result of layer fatigue (Fig. 6e) and cracks concluded in delaminating wear (Fig. 6f) in the shape of network beginning from the edges of these cavities are observed<sup>10</sup>.

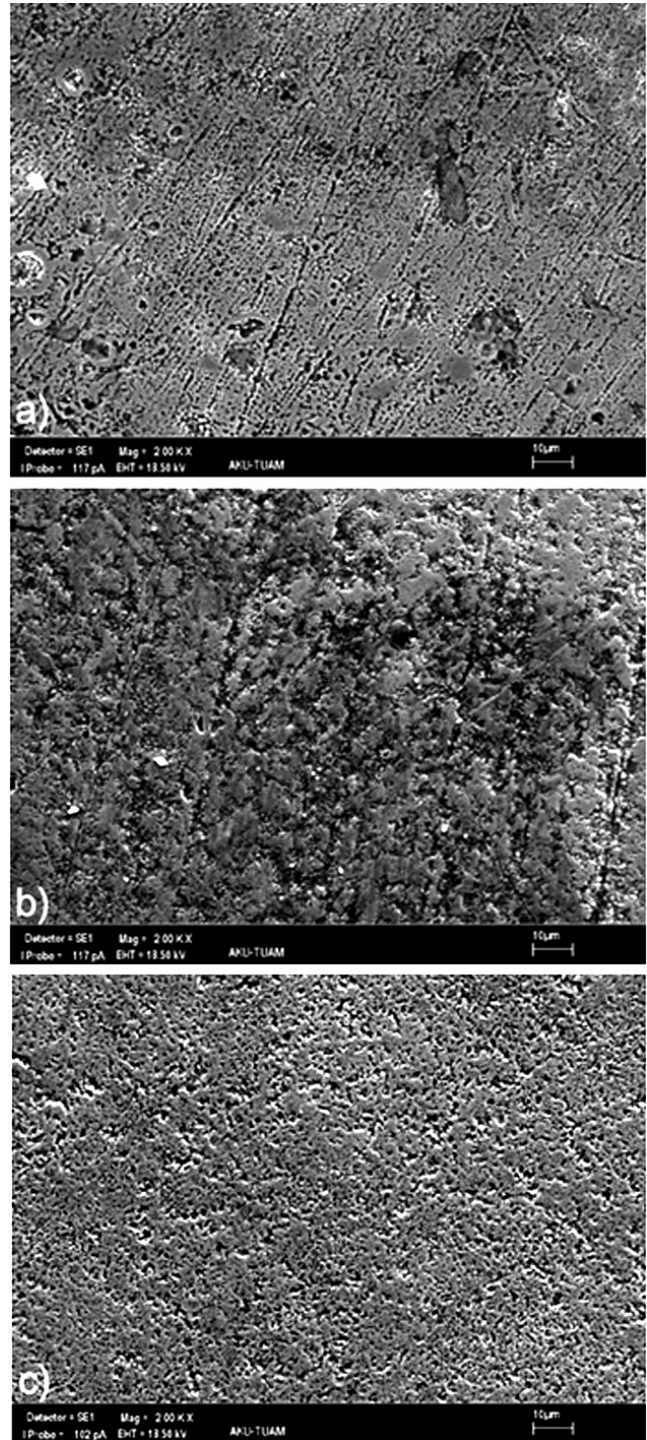


Fig. 5—SEM morphologies of the corrosion regions in greasy media against WC-Co ball in 20N load of each three PPB steel for 3 h in 800°C (a) 440C, (b) 8620 and (c) 52100

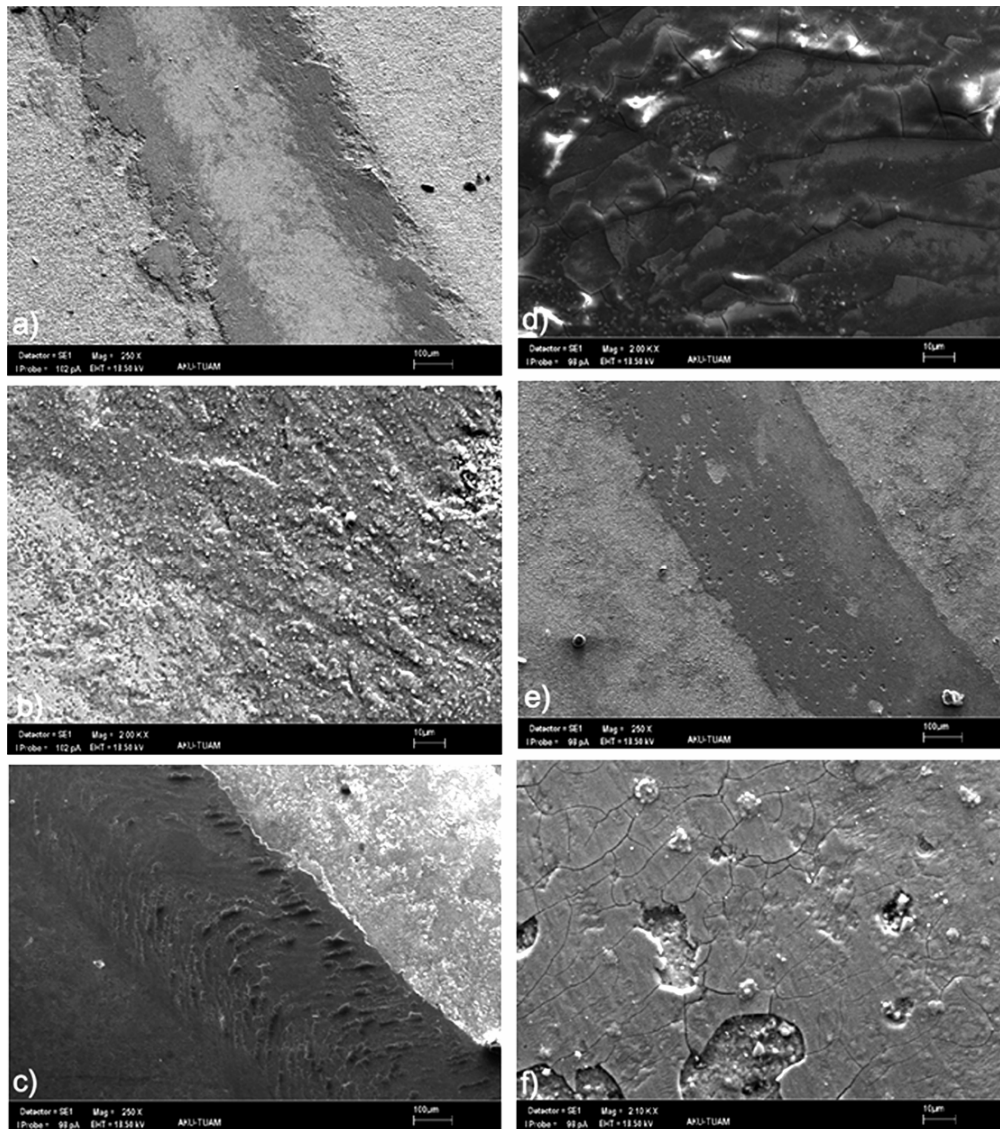


Fig. 6—SEM morphologies of the corrosion regions in unlubricated media against WC-Co ball in 5 N load of each three PPB steel for 3 h in 800°C (a) 440C, (b) 440C high-magnification, (c) 8620, (d) 8620 high-magnification, (e) 52100 and (f) 52100 high-magnification

## Conclusions

In this study, plasma paste boronizing process which consists of 100% borax was successfully performed on AISI 8620, 52100 and 440C steels and the following conclusions can be made:

- (i) While the lowest wear rate is observed in 52100 steel boronized with plasma paste in 700°C, the highest wear rates are observed in 440C steel boronized with plasma paste in 800°C.
- (ii) Together with the increase in the amount of load applied for wear, the wear rate and friction coefficients have also increased in each three steel types.

- (iii) While the lowest friction coefficient is observed in PPB 52100 steel in 700°C for 3 h, the highest friction coefficient has been obtained in 440C steel in 800°C for 5 h.
- (iv) Increase of plasma paste boronizing temperature and time in each three steels leads to increase in surface roughness values.
- (v) Increase in plasma paste boronizing time increases the thickness of the boride layer, which results in a decrease in wear.

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