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Short Communication

Plasma paste boronizing of AISI 8620, 52100 and 440C steels

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ABSTRACT

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_2–30%Ar under a constant pressure of 4 mbar. The properties of boride layer were evaluated by optical microscopy, X-ray diffraction and Vickers micro-hardness tester. X-ray diffraction analysis of boride layers on the surface of the steels revealed FeB and Fe_2B phases for 52100 and 8620 steels and FeB, Fe_2B, CrB and Cr_2B borides for 440C steel. PPB process showed that since the plasma activated the chemical reaction more, a thicker boride layer was formed than conventional boronizing methods at similar temperatures. It was possible to establish boride layer with the same thickness at lower temperatures in plasma environment by using borax paste.

1. Introduction

Boronizing is a thermo-chemical diffusion process in which boron is diffused into the steel at high temperatures. Surfaces of boronized irons and steels have high hardness, excellent wear resistance, good corrosion resistance and strong chemical stability. Boronizing process can be applied in solid, liquid and gaseous environment [1–7]. The above three boronizing methods have certain disadvantages. In gaseous boronizing, boron sources such as BCl_3, TMB (trimethyl borate), TEB (triethyl boron) and BF_3 along with H_2 and Ar gases are used [8–10]. Traditional gaseous boronizing agents are very sensitive to even the slightest traces of moisture, very poisonous, more costly and subject to explosion. In the case of liquid boronizing, the sample is dipped into a melted salt bath which consists of borax, boric acid and ferro silica and held at that temperature for the required amount of time. But formation a firmly adhered salt layer on the workpieces constitutes its advantages and this can be quite costly to remove after boronizing has been completed. Pack boronizing is generally performed with patent protected agents that consist of approximately 5%B_4C, 5%KBF_4 and 90%SiC (commercial Ekabor®). In this method the powder mixture is filled into a leak proof box in which the sample is placed. The box is heated up to the required temperature. It is kept at this temperature as necessary and then cooled [11]. Although pack boronizing is used commonly for commercial purposes among these methods, higher treatment temperatures and longer periods of time constitutes its drawbacks [12,13].

Studies have been carried out in order to reduce the boronizing temperature and time for the last 40 years. The studies have demonstrated that ion implantation boronizing [14,15], and plasma enhanced boronizing processes [13,16–19] are effective for the reduction of the boronizing temperature and time. Lately, studies carried out on plasma boronizing (PB) have been increasing gradually [19–23]. PB process has a superior advantage when compared to conventional boronizing processes. For example, thanks to the high energy generated in PB process it is possible to operate at lower temperatures and distortion (deterioration of shape) can be minimized. In addition to this, in PB process it is possible to reduce FeB amount or establish a single layer Fe_2B layer by changing gas mixture ratios [11,19,20]. However, the gases (B_2H_6, BCl_3) used in plasma boronizing, which are expensive, poisonous and explosive characteristics, is a disadvantage. Moreover, in plasma boronizing process carried out in BCl_3 environment, the boride layer having pores poses a tremendous problem [8,21,22].

The disadvantages in PB process can be eliminated through PPB surface process. The paste used having environmentally friendly boron raw materials and gases generally being hydrogen, argon and nitrogen which have inert characteristics make this process advantageous. Yoon et al. [9] have boronized AISI 304 steel by plasma paste which consists of borax ($Na_2B_4O_7$) and amorphous boron, at different temperatures in Ar/H_2 gases and have examined the diffusion kinetics and morphology of the layer. They reported that using plasma paste process caused lower activation energy for the formation of the boride layer than that for the conventional boronizing processes.

The characterization of boronized steels by using various boronizing processes has been evaluated by a number of investigator

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[5–8,12–16,23]. However, there is little literature about characterization of paste boronized steels in plasma environment. The main objective of this study was to characterize plasma paste boronized AISI 8620, 52100 and 440C steels, which are popular bearing steels with various alloy elements content, by using 100% borax paste. For this reason each sample was studied by optical microscopy, micro-hardness, surface roughness and X-ray diffraction analysis.

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

2. Experimental

The chemical composition of AISI 8620, 52100 and 440C steels with the dimensions of Ø20 × 6 mm used in this study are shown in Table 1. PPB process was performed in a dc plasma system shown in Fig. 1. It consists of a stainless steel container in which

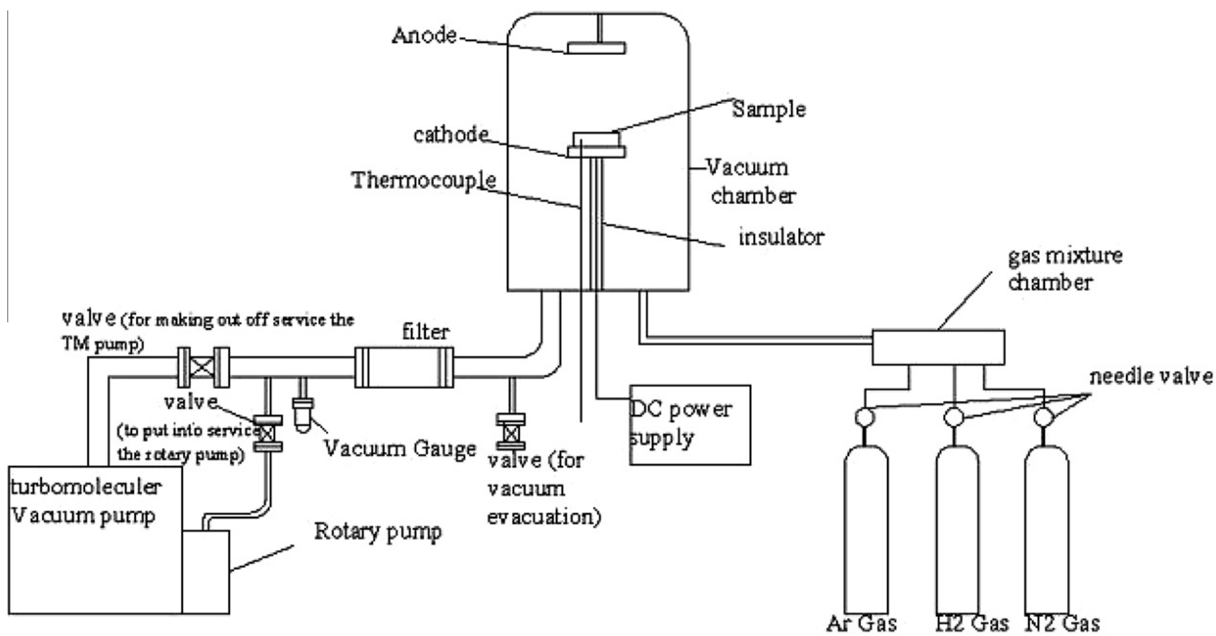

Fig. 1. Schematic illustration of plasma paste boronizing device.

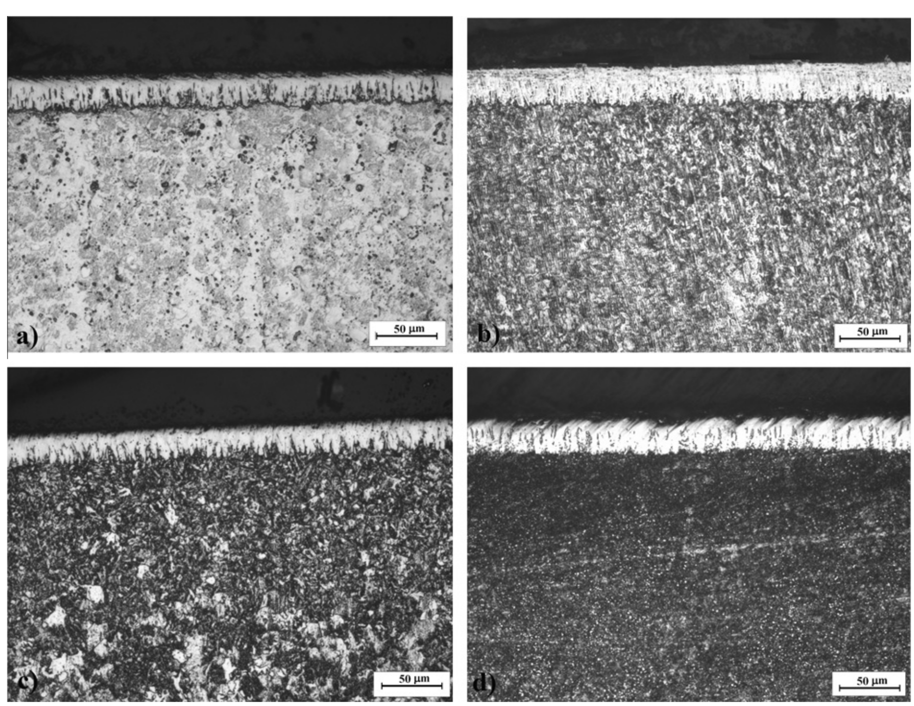

Fig. 2. The cross section micrographs of the plasma paste boronized AISI 8620 and 52100 steels at temperature of 700 °C (a) AISI 8620 for 3 h, (b) AISI 8620 for 5 h, (c) AISI 52100 for 3 h, (d) AISI 52100 for 5 h.

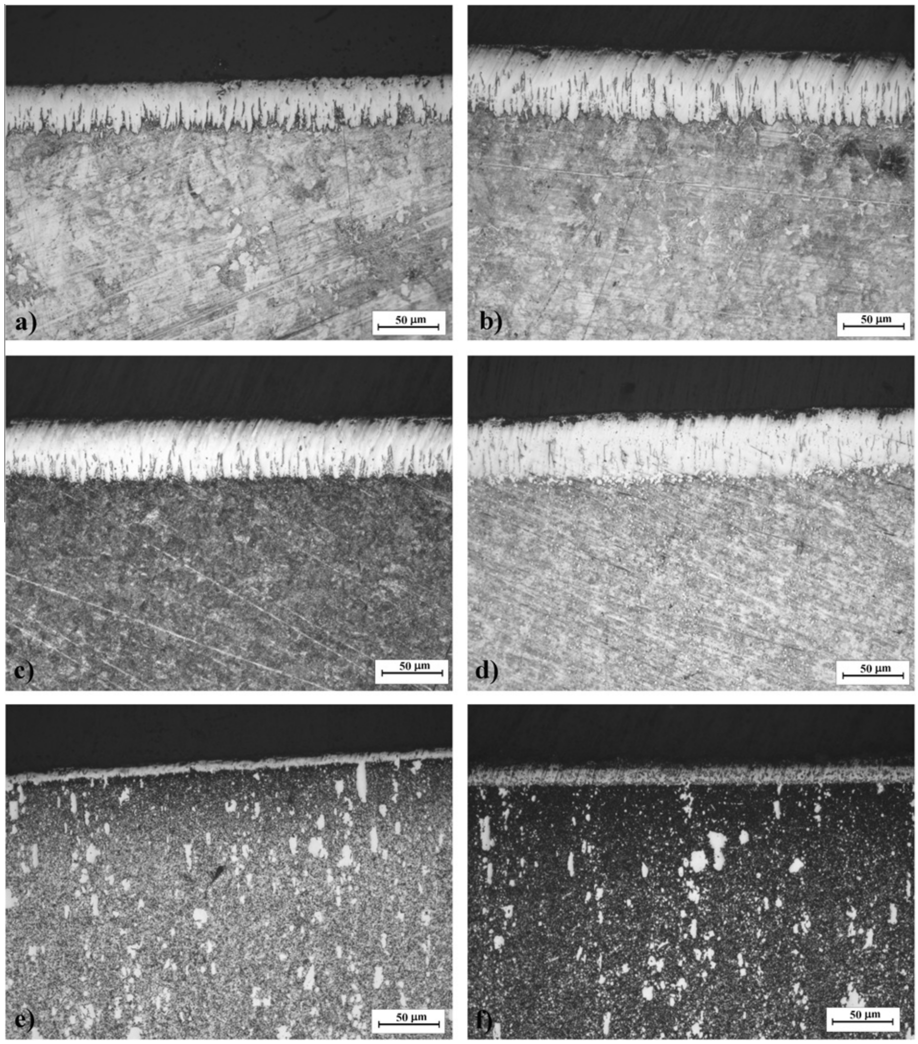

Fig. 3. The cross section micrographs of the plasma paste boronized AISI 8620, 52100 and 440C steels at temperatures of 800 °C (a) AISI 8620 for 3 h, (b) AISI 8620 for 5 h, (c) AISI 52100 for 3 h, (d) AISI 52100 for 5 h, (e) AISI 440C for 3 h, (f) AISI 440C for 5 h.

an insulated central electrode is situated, which holds samples. The anode at earth potential surrounds this. The cathode has a nest on which the sample is located. The prepared samples were placed in the vacuum container and container pressure was set to 2.5×10^{-2} mbar of vacuum. The samples were plasma paste boronized at 700 and 800 °C for 3 and 5 h in a gas mixture of 70%H_2–30%Ar under a constant pressure of 4 mbar at 5 kHz pulse frequency and duty rate of 80%. Temperature of the samples was measured using a chromel–alumel thermocouple, placed at the bottom of the treated samples.

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

Boronizing procedure	Steels	Boride layer thickness, μm			
		700 °C		800 °C	
		3 h	5 h	3 h	5 h
Plazma paste boronizing	AISI 8620	27	30	36	44
	AISI 52100	22	29	38	46
	AISI 440C	–	–	8	18
Pack boronizing	AISI 8620	–	–	23	35
	AISI 52100	–	–	20	40
	AISI 440C	–	–	6	12

Cross-sections of PPB steels were prepared metallographically to observe morphological details using BX60 Olympus microscope. The X-ray diffractograms were obtained using a copper tube source in the conventional Bragg–Brentano (θ–2θ) technique having symmetric geometry with monochromatized radiation (Cu Kα, λ = 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 layers was measured on the cross-sections using Micro-Vickers indenter (Shimadzu HMV-2) with 50 g loads. After the process the surface roughness values were obtained by a Perthometer M2 roughness device.

3. Results and discussions

3.1. Surface characterization

The cross section optical micrographs of the PBB AISI 8620, 52100 and 440C steels at temperatures of 700 and 800 °C for 3 and 5 h are shown in Figs. 2 and 3. While the boride layers were observed on the surfaces of 8620 and 52100 steels at 700 °C, no layer formed on 440C at the same temperature. At 800 °C a boride layer formed on all three steels, but the boride layer thickness formed on the martensitic stainless steel (440C) having high Cr content was much more less when compared to other steels. This

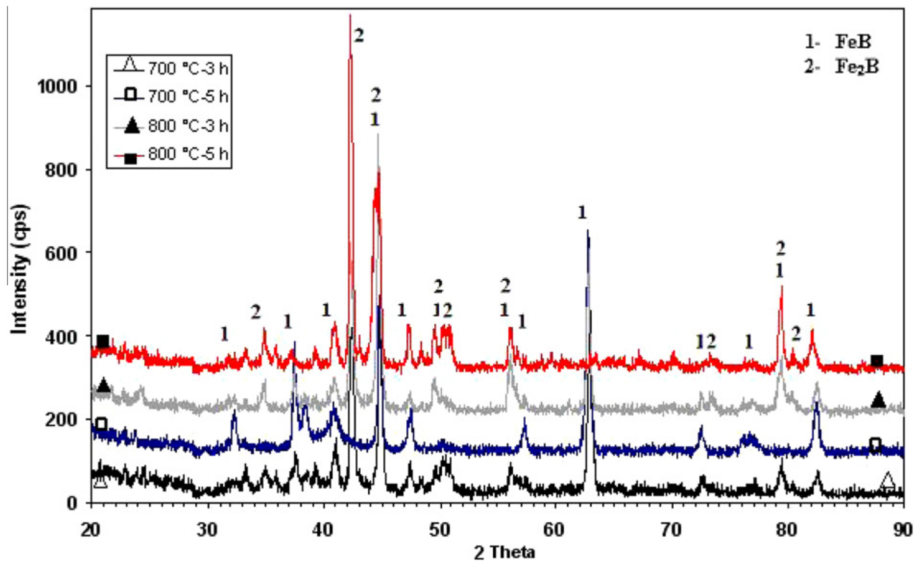

Fig. 4. The X-ray diffraction (XRD) patterns of plasma paste boronized AISI 8620 steel at various temperatures and times.

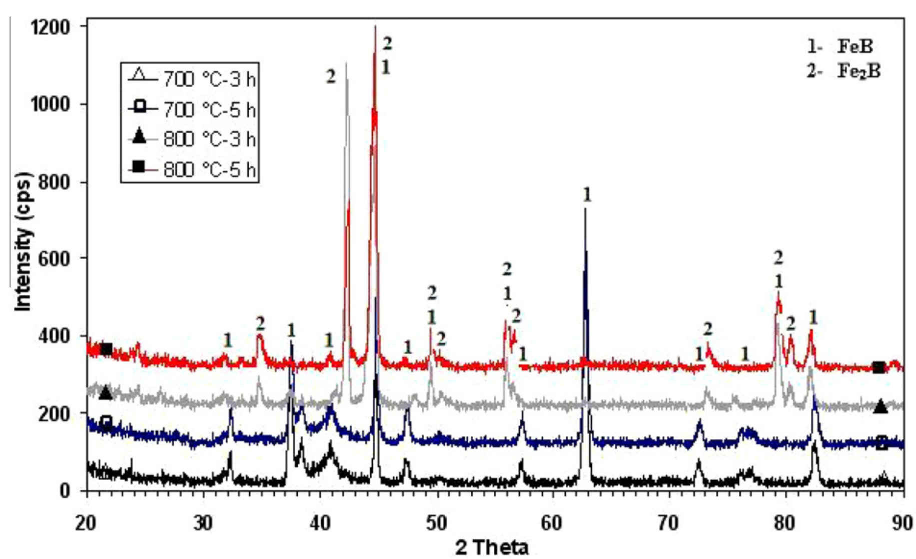

Fig. 5. The X-ray diffraction (XRD) patterns of plasma paste boronized AISI 52100 steel at various temperatures and times.

could be attributed to that chromium borides formed in the grain boundary prevented the diffusion of boron [24–26]. When the layer morphologies were examined, it was possible to see a dominant saw tooth formation on the 8620 steel. By the increase of the alloy element and carbon amount in the 52100 and 440C steels, the saw tooth morphology deteriorates and the layer thickness decreases [13,27].

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 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 and pack boronized three steels. It was seen that boride layer thickness values of PPB steels were higher when compared with pack boronized steels. This suggested that boronizing process at lower temperatures and times (700 and 800 °C) can be performed by PPB that provides the energy and time saving. Yoon et al. [9] 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 [28,29].

The X-ray diffraction (XRD) patterns of paste boronized three steels by plasma at various temperatures and times are given in Figs. 4–6. As seen in Figs. 4 and 5, for plasma paste boronized AISI 8620 and 52100 steels, FeB and Fe_2B phases, where FeB was located near the surface and Fe_2B in the vicinity of steel matrix, were found. For plasma paste boronized AISI 440C steel (Fig. 6), CrB and Cr_2B borides were observed as well as the formation of FeB and Fe_2B borides. The strongest peak from the CrB was detected, suggesting that a large amount of CrB formed in the

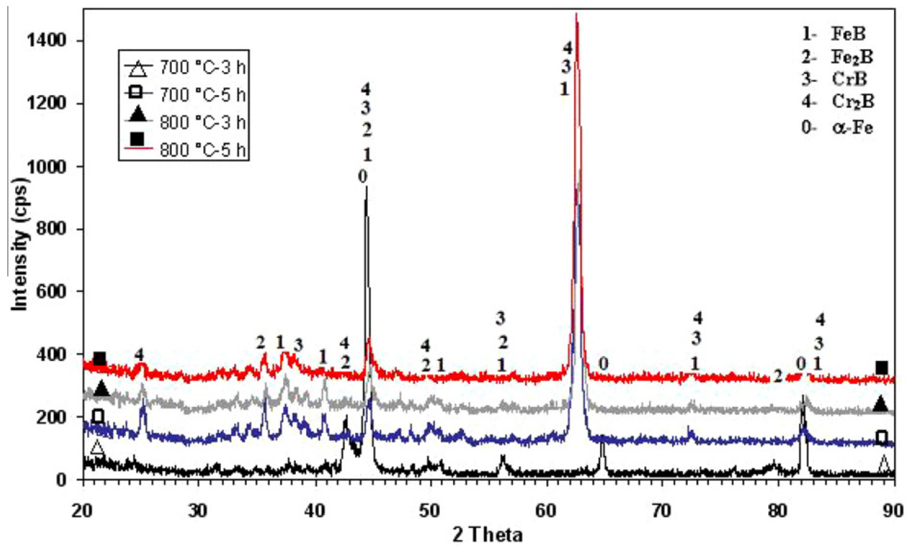

Fig. 6. The X-ray diffraction (XRD) patterns of plasma paste boronized AISI 440C steel at various temperatures and times.

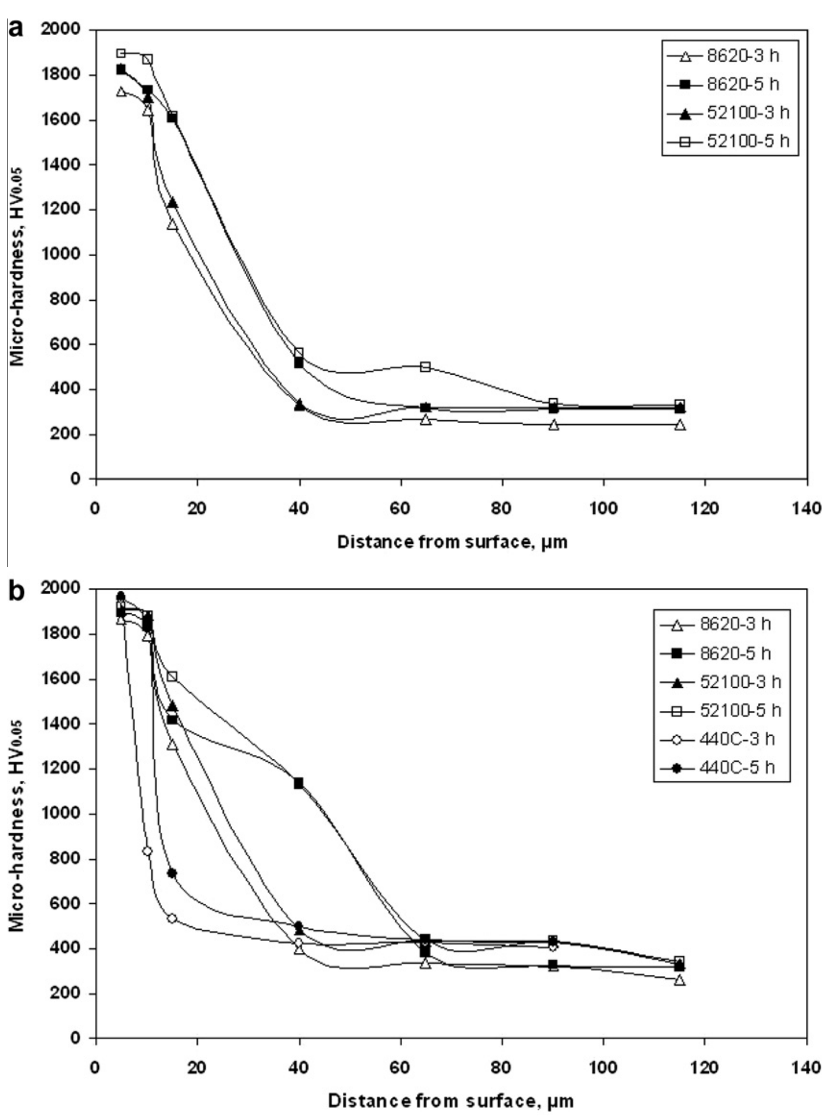

Fig. 7. The micro-hardness distribution from the surface to interior along a line on the cross-sections of the steels plasma paste boronized at (a) 700 °C (b) 800 °C.

boride layer. In the case of boriding of chromium steel, Cr atoms diffusing from the base alloy within the boride coating could react with active boron to give CrB which, in turn, enter the FeB lattice, substituting for iron [30]. Furthermore, it is reported that the FeB and Fe_2B phases dissolve considerable amounts of chromium and the solubility limit of chromium in Fe_2B is much lower than FeB [31]. While no boride layer was seen on steel boronized at 700 °C in optic microscope examinations, the XRD results showed the formation of boride phases even if they were low. While α-Fe was seen on surface of the 440C boronized at 700 °C for 3 h, it was not seen on the steel boronized for 5 h.

During plasma paste boronizing, borax reacts with active hydrogen (H^+) in glow discharge and the reaction occurs as follows;

$$Na_2B_4O_7 + 12H^+ \rightarrow Na_2O + 6H_2O + 4B \quad (1)$$

$$xB + yH^+ + e^- \rightarrow BxHy \text{ (Boron hydride)} \quad (2)$$

$$BxHy \rightarrow xB + y/2H_2 \quad (3)$$

$$B^{+1} + 2Fe \rightarrow Fe_2B \quad (4)$$

$$Fe_2B + B^{+1} \rightarrow 2FeB \quad (5)$$

Atomic boron was produced through the decomposition of the boron hydride (BxHy) from the paste, and this atomic boron became the active boron, B^{+1} within the molten borax or in the glow discharge. Finally, this active boron, B^{+1}, diffused and reacted with Fe to form the boride layer.

The micro-hardness variation from the surface into the matrix on the cross-sections of the steels plasma paste boronized at 700 and 800 °C is shown in Fig. 7a and b. Since the boride layer could not be observed, micro-hardness measurements were not possible for the 440C steel boronized at 700 °C. 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 [32,33]. 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 [23,34].

The results of micro-hardness measurement pointed out that boride layer enhanced the surface hardness of metal surfaces. The high hardness of boride layer on 440C steel could be associated to the CrB phase. Badini et al. [35] have reported that chromium increased the hardness of the boride layer. In thermo-chemical boronizing treatments, high hardness is attained directly through formation of borides during boronizing and does not require quenching.

4. Conclusions

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

- At 700 °C plasma temperature, while the boride layers were observed on the surface 8620 and 52100 steels no boride layer formed on 440C. The boride layer thickness for the 440C was much more less when compared to other steels at 800 °C.
- Since the plasma activates the chemical reaction more, a thicker boride layer is formed than conventional boronizing methods at similar temperatures. It is possible to establish boride layer with the same thickness at lower temperatures in plasma environment.
- The boride layer on 52100 and 8620 steels consists of FeB and Fe_2B phases. CrB and Cr_2B borides have been found on 440C steel as well as FeB and Fe_2B borides.
- While the highest hardness value (1968 $HV_{0.05}$) was determined for the boride layer on 440C steel plasma paste boronized at 800 °C, the lowest hardness value (1730 $HV_{0.05}$) was found for the boride layer on 8620 steel plasma paste boronized at 700 °C.

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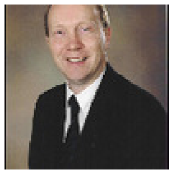

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