



## High temperature tribological behaviors of plasma electrolytic borocarbided Q235 low-carbon steel

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### ABSTRACT

Plasma electrolytic boronizing is a novel technique to fabricate rapidly a hardening layer on steel. In this study, a boride layer on Q235 low-carbon steel was prepared by plasma electrolytic borocarbiding process (PEB/C) in the 30% borax electrolyte with carbon-containing organic additive at 330 V. The microstructure and phase constituent of the PEB/C steel were analyzed by SEM and XRD. Microhardness profile of the PEB/C steel was determined, and its tribological properties under dry sliding against ZrO<sub>2</sub> ceramic ball were evaluated using a ball-on-disk friction tester at ambient temperature and high-temperature (up to 500 °C) environments. Friction coefficient and wear rate before and after PEB/C treatment were measured, and the wear mechanism was also discussed. The results show that the boride layer mainly consists of the Fe<sub>2</sub>B phase, the hardness of which is close to 1800 HV. The PEB/C treatment could significantly decrease the friction coefficient and improve wear resistance of the low-carbon steel. Meanwhile, the friction coefficient and wear rate of the untreated and PEB/C treated steel samples also increase with increasing the environment temperature, but the wear rate of PEB/C treated steel is always much lower than that of the untreated steel at different environment temperatures. Plasma electrolytic borocarbided low-carbon steel can maintain higher wear resistance at high temperature environment, which ascribes to the formation of Fe<sub>2</sub>B phase with good thermal stability in the hardening layer. The wear mechanism of untreated low-carbon steel is mainly the fatigue wear at ambient temperature and the fatigue wear and adhesive wear at 200 °C. The PEB/C treated steel displays the adhesive wear at 200 °C. However both the untreated and PEB/C treated samples are transferred to the oxidation wear and adhesive wear at 500 °C.

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### 1. Introduction

The Q235 low-carbon steel is widely applied in some fields such as making repairs or building room frame, bridge construction, vehicle, ships, mechanical parts and high-voltage transmission tower. However, it is currently restricted to non-tribological applications due to its low hardness, poor wear resistance and large quenching distortion. Some surface hardening techniques such as carburization, nitriding and nitrocarburizing have been developed to overcome these problems [1–6].

Boronizing is a thermochemical surface treatment, in which boron atoms diffuse into the surface of steel to form a hardening layer. The boride layer has high hardness, wear and abrasion resistance, heat resistance or corrosion resistance [7,8]. However, the conventional

boronizing processes [9–12] such as gas boronizing and paste boronizing on steels take disadvantages due to long treating duration, environmental contamination, toxic and explosive nature. In general, the boronizing process results in a hard and thick boride layer and intermetallics with FeB and Fe<sub>2</sub>B needle-like microstructure on the surface of steel. The main disadvantage is a brittleness of boriding layer, especially of the FeB boride [9,10,12]. There was the danger of extensive crack formation and spallation of the top boride layer after boriding due to the generation of very high internal stresses upon cooling. It was found that a single-phase Fe<sub>2</sub>B layer [13,14] might provide better performance with low brittleness. In addition, the mutual penetration of boron, carbon and nitrogen atoms can also reduce the brittleness of boride layer [15,16].

Plasma electrolytic technique was recently developed for surface modification of steels in solution [17–25]. In the plasma electrolytic boronizing (PEB) or borocarbiding (PEB/C) process, the workpiece as a cathode in an electrolysis cell is connected to a direct-current power supply [24,25]. Plasma discharges around the workpiece heats the steel surface over 900 °C and enhances rapid diffusion of boron and carbon atoms into the steel.

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The wear behavior of borided steels with the conventional boronizing processes at ambient and elevated temperatures was evaluated by some investigators [26–32]. Taktak [31] analyzed the sliding wear behavior of the powder-pack borided AISI 440C and 52100 steels at high temperature. It indicated that boriding process could greatly improve the wear resistance of steels at various temperatures. However, the microstructure of PEB or PEB/C treated steel has larger difference with conventional boronizing layer, and their wear properties at ambient and high temperatures are not reported. It is believed that the PEB/C treated steel should have a good wear resistance due to its high hardness, but it is not clear whether the PEB or PEB/C steel can keep its excellent wear resistance at high temperatures.

In this work, we prepared a hardening layer on Q235 low-carbon steel by plasma electrolytic borocarburing process (PEB/C). The microstructure, phase constituent and hardness of the PEB/C-treated sample were determined, and the high-temperature tribological behavior at different temperatures reaching up to 500 °C before and after PEB/C treatment was investigated. The wear mechanism of the hardening layer at different environment temperatures was discussed.

## 2. Experimental details

As-received Q235 low-carbon steel sheet has a nominal composition (wt.%): 0.14–0.22 C, 0.30–0.65 Mn,  $\leq 0.30$  Si,  $\leq 0.045$  P,  $\leq 0.055$  S, and Fe rest. The Q235 low carbon steel sheet was cut into small pieces of 55 mm  $\times$  16 mm  $\times$  1.5 mm and polished with a 1000-grit emery paper to achieve a surface roughness  $R_a$  of 0.108  $\mu\text{m}$ . Then these samples were cleaned with alcohol and dried. The plasma electrolytic borocarburing (PEB/C) treatment of steel samples was carried out in 30% borax aqueous solution with carbon-containing organic compound additive. In the plasma electrolytic saturation process, the Q235 steel sample and stainless bath were cathode and anode, respectively. The negative bias voltage on Q235 steel cathode was initially set in the range of 120–140 V. The aqueous solution was heated to produce a gas envelope around the sample, then the dielectric breakdown of gas envelope generated plasma discharge which heated the surface temperature of steel over 950 °C [17]. When the plasma envelope becomes stable, the voltage was adjusted to 330 V for stable discharge. The activated carbon and boron atoms in the plasma envelope diffused into the steel, forming a borocarburing layer on the steel surface. The whole PEB/C discharging duration was 30 min.

After completion of the borocarburing treatment, the Q235 steel sample was taken from the electrolyte and cleaned with running water. The hardness profile of the cross-sectioned borocarburing layer was measured using a HX-1 Vickers microhardness tester with 50 g load and 15 s dwell time. The phase composition of borocarburing layer was characterized on the surface of borided samples by X' PERT PRO MPD X-ray diffraction (XRD) analysis using Cu  $K_{\alpha}$  radiation. The polished cross-sectional sample was etched in 4% nitric alcohol solution. Then microstructure and composition of PEB/C layer were analyzed using S-4800 scanning electron microscope (SEM) with energy dispersive spectroscopy (EDS).

The surface loose layer of the PEB/C sample was grinded and a hard and dense borocarburing layer with a surface roughness of 0.352  $\mu\text{m}$  was left for wear test. The surface hardness of the polished samples is about 1700 HV. A HT-1000 ball-on-disk high-temperature friction and wear tester was used to evaluate tribological properties of bare Q235 steel and PEB/C sample at dry sliding with 5 N load and 300 rpm rotating rate against ZrO<sub>2</sub> ceramic ball of 4.763 mm diameter. The wear tests were performed at ambient temperature of about 25 °C, and high temperatures of 200 °C and 500 °C. The wear track radius is 5 mm and the sliding time is 10 min. The friction coefficient ( $\mu$ ) was automatically recorded during sliding wear test. The cross-sectional area ( $S$ ) of the wear tracks was measured by a surface profilometer, thus the wear volume ( $V$ ) and wear rate ( $k$ ,  $\text{mm}^3/\text{N}\cdot\text{m}$ ) were calculated from this cross-sectional area and stroke amplitude

according to the formula of  $k = V/FI = \pi dS/FI$ , where  $d$  is the wear track diameter,  $F$  is the normal load and  $I$  is the total sliding distance of ZrO<sub>2</sub> ball. Finally, the typical morphology and composition of wear tracks were analyzed by SEM equipped with energy-dispersive X-ray spectroscopy (EDS). The typical wear scars of ZrO<sub>2</sub> ball were observed by Carl Zeiss image A2m optic microscope.

## 3. Results and discussion

### 3.1. Microstructure observation and phase components

Fig. 1 shows the surface morphology of the plasma electrolytic borocarburing sample with 30 min discharge at 330 V in borax solution. Its surface roughness is 1.687  $\mu\text{m}$ , which is higher than that of Q235 low-carbon steel substrate of 0.168  $\mu\text{m}$ . Many particles appear on the surface of PEB/C sample and some holes caused by strong plasma discharge can also be observed. The surface morphology of PEB/C sample implies that the moving discharge spots should appear during plasma electrolytic borocarburing, though it looks like a continuous discharge envelope around the Q235 steel sample and we could not observe some moving sparks. A high applied voltage causes an increase in the kinetic energy of the discharge spaces and a rise in pressure in the plasma envelope, which results in an intensification of plasma etching and consequent surface roughening.

Fig. 2 depicts the microstructure and composition profiles of the cross-sectional sample of Q235 steel after 30 min borocarburing treatment at 330 V in borax solution. As shown in Fig. 2a and b, the PEB/C sample etched in 4% nitric alcohol solution displays a multi-layer structure: one loose top layer of about 6  $\mu\text{m}$ , one boride layer of about 20  $\mu\text{m}$  and one diffusion layer of about 40  $\mu\text{m}$ . As shown in Fig. 2b, the Fe concentration from the steel substrate to the surface gradually decreases, but the B concentration in the boride layer is higher than that in other layers. Meanwhile the C and O concentrations in the top surface layer suddenly increase. In addition, the microstructure in Fig. 2a is different from the morphology of the conventional boronizing process, where the FeB and Fe<sub>2</sub>B needle-like microstructure are usually observed at the boronizing zone [12–14]. The morphology of needle-like microstructure depends on the ratio of alloying elements as well as the treatment temperature and time [33,34].

After the top layer of PEB/C sample is polished, the hard boride layer is left. Fig. 3 displays the XRD patterns of bare Q235 steel substrate, and unpolished and polished PEB/C samples. The PEB/C sample mainly consists of Fe<sub>2</sub>B and FeB phases and little Fe<sub>2</sub>O<sub>3</sub>, Fe<sub>3</sub>O<sub>4</sub> and Fe<sub>3</sub>C phases, which identifies that the boron and carbon elements of electrolyte certainly diffuse into the steel during plasma discharge.

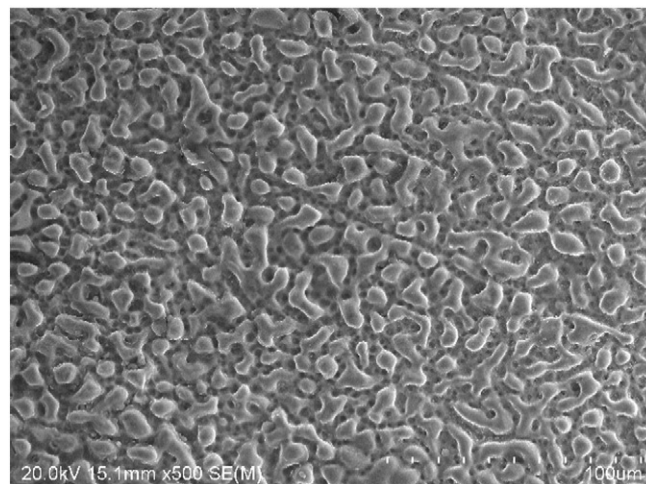


Fig. 1. SEM surface topography of PEB/C sample treated at 330 V with 30 min.

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