

High Temperature Tribological Behavior of Borocarbured Layer on Q235 Steel

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A borocarbured layer was successfully fabricated on the surface of Q235 low-carbon steel via double glow treatment to improve the wear resistance at elevated temperature. The phase composition and microstructure of borocarbured layer were investigated by XRD and SEM. The microhardness of borocarbured layer from the surface to the substrate was studied. The tribological behaviors of borocarbured layer and substrate were investigated under the dry-sliding against ZrO₂ ball at three temperatures. The results indicate that the borocarbured layer consists of an outermost boride layer and a transition layer of carburized layer. The boride layer with main phase of Fe₂B has a high hardness around 1700 HV, and the hardness of transition layer with main phase of Fe₅C₃ is around 600 HV. The novel gradient structure of an outermost boride layer and inner carburized layer is design in this research decreases the hardness mismatch of coating to prevent the boride layer peeling off. The friction coefficient and specific wear rate of borocarbured layer are much lower than that of substrate at the same temperature. In addition, for the samples after wear test at 20 °C and 200 °C, it can be concluded that the wear mechanism of borocarbured layer is main abrasive wear while the wear mechanism of substrate is main fatigue wear and slight adhesive wear. As the temperature rises to 500 °C, the tribological behaviour of both borocarbured layer and substrate are main oxidation wear and adhesive wear. The borocarbured layer effectively improves the wear resistance of low carbon steel due to the higher hardness and great thermal stability at high temperature. *Keywords:* borocarburing, double glow, surface hardness, tribological behaviour, surface modification.

1. INTRODUCTION

A majority of low carbon steel components are often used under the severe conditions such as corrosion, oxidation and abrasive wear, which can drastically degrade their mechanical properties [1–3]. Attributing to the inexpensive of low carbon steel, many researchers have not given up on improving its mechanical properties. In recent years, a lot of surface techniques such as induction hardening, surface shot blasting and surface heat treatment are applied to improve the surface performance of steel [4–6]. However, the thermal diffusion technologies [7–10] (nitriding, carburization and nitrocarburizing) are the most favorite option for researchers to treat large numbers of workpieces at a reasonable cost.

Boriding is an effective surface modification technique. The boride layer with great wear resistance of FeB and Fe₂B phase [11], which could substantially improve the wear resistance and corrosion resistance of steels. Nonetheless, traditional boride layer has a serious drawback that is the crack propagation in it due to the difference of thermal expansion coefficient between FeB and Fe₂B phase [12]. Kusmanov et al. [13] pointed out that the boride layer with single-phase Fe₂B was better than that with FeB and Fe₂B phase. In addition, boride layer is much harder than steel, which easy to peel off due to the non-uniform distribution of stress in the service of environment [14]. B. Wang et al.

[15] have fabricated the borocarbured layer by plasma electrolytic borocarburing process which produced the waste and pollution of the electrolyte, the boride layer was undesired due to the FeB and Fe₂B phase. So, it is required to find an effective method to improve the mechanical properties of steels that will be beneficial for the stable operation of the workpiece in a harsh environment.

Recently, double glow plasma surface technology that is a novel method called Xu-Tec by others, deserved great attention. Many practical achievements have confirmed that double glow treatment has unique advantages in preparing coatings [16, 17]. Using double glow plasma surface technology can save precious metal resources and reduce energy consumption in industrial production. Double glow treatment employs plasma region, which produced by double glow discharges. Beneficial elements are sputtered by argon ions and then go to deposit on substrate surface [18–20]. The coatings manufactured by double glow process have gradient structure due to the diffusion of beneficial elements, and the adhesion between coating and substrate is strong because of the metallurgical bonding effects [21–23].

In this paper, the borocarbured layer fabricated on Q235 low-carbon steel via double glow plasma surface technology. The microstructure and phase constituent were determined. Tribological behavior at different temperatures was investigated. Finally, wear mechanism of

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borocarbonized layer at different temperatures was discussed.

2. EXPERIMENT

The base material was Q235 low-carbon steel (purchased from Beijing Iron and Steel Research Institute of China), and chemical composed (wt.%) of Q235 steel was 0.35 Mn, 0.3 Si, 0.18 C, ≤ 0.04 S, ≤ 0.04 P and Fe rest. The samples were cut by wire cutting, and the size of each sample was 10 mm \times 10 mm \times 8 mm. During pretreatment process, the sample was polished by 1200 meshes water-proof abrasive paper, and then cleaned in acetone to dislodge any surface contamination.

The machine of double glow plasma alloying technology consisted of two parts: the cathode and anode (Fig. 1). During the double glow treatment, argon was used as inert gas, which was stimulated as Ar⁺ because of double glow discharge [24]. The surfaces of target and workpieces were activated due to the bombardment by Ar⁺ with a certain high kinetic energy [19]. The beneficial elements were sputtered from target to workpiece and diffused into the surface of sample under the high temperature device chamber environment [25]. The borocarbonized layer was fabricated by double glow plasma alloying technology in the following steps. Firstly, carburized layer as transition layer was prepared on surface of substrate by pure carbon target, and then boride layer was prepared on the carburized sample by B₄C target. The detailed experimental parameters are showed in Table 1.

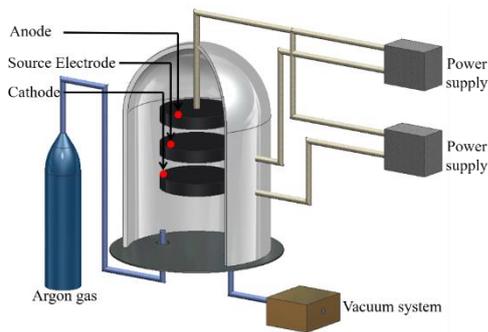


Fig. 1. Sketch of double glow plasma surface alloying machine

Table 1. The experimental parameters of double glow process

Parameter	Values (C Deposition)	Values (B Deposition)
Voltage of the source electrode, V	950	950
Voltage of the cathode, V	450	550
Working pressure, Pa	35	38
Distance between the source electrode and cathode, mm	15	10
Treatment time, h	3.5	4.5
Argon flow rate, sccm	70	50

The Scanning electron microscopy (FEI, Quanta450, USA) was used to determine the surface and the cross-sectional microstructure. The phase composition of borocarbonized layer was examined by X-ray diffraction (Rigaku, DMAX-RB12KW, Japan) with copper K α radiation ($\lambda = 1.5418$ Å, $20^\circ \leq 2\theta \leq 80^\circ$). The microhardness of borocarbonized layer was examined by a

Vickers Indenter (DianYing, DHV-1000/Z, China) with the applied load of 50 g and 15 s dwell time. The ball-on-disk friction and wear tester at high temperature (KaiHua, HT-500, China) was used to detect the tribological behaviors of substrate and borocarbonized layer in air with relative humidity of (45 ± 5) % under dry sliding against ZrO₂ ball, the normal load was 730 g and 560 rpm rotating rate with a turning radius of 2 mm at 20 °C, 200 °C, and 500 °C respectively. The depth-of-field system (Keyence, VHX-1000, Japan) was used to measure the morphology of wear tracks. To evaluate friction and wear performances, the wear volumes and wear rate of the samples were calculated by following equations [26].

$$V = \frac{2\pi hr}{6b} \cdot (3h^2 + 4b^2), \quad (1)$$

where, V is volume of wear area, mm³; h is the depth of wear area, mm; b is the width of wear area, mm; r is radius of wear area, mm.

$$v = \frac{V}{S}, \quad (2)$$

$$K = \frac{V}{SP}, \quad (3)$$

where, v is wear rate, mm³m⁻¹; S represents sliding distance, m; K is specific wear rate, mm³N⁻¹m⁻¹; P is the load, N.

3. RESULTS AND DISCUSSION

3.1. Characterization of borocarbonized layer

Fig. 2 shows the surface microstructure of borocarbonized layer on low-carbon steel, which is dense and homogeneous. There are a lot of compact submicron-sized particles arranged in stacked structure, and this is the typical feature of double glow techniques. The surface roughness of borocarbonized layer (around 1.175 μ m) is much higher than that of substrate (around 0.217 μ m). The results indicated that the beneficial elements were sputtered by active Ar ions, which got a certain high kinetic energy from target to sample due to the difference of the voltage. The beneficial elements with super power unremittingly struck the sample surface caused the increase in the surface roughness.

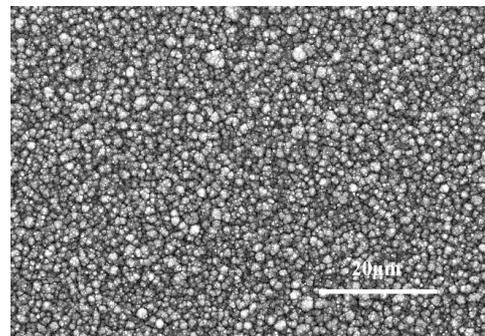


Fig. 2. SEM image of borocarbonized layer

The cross-sectional microstructure and EDS results of borocarbonized layer are shown in Fig. 3. As shown in figure, the borocarbonized layer displays a multilayered structure, and the special structure consists of an outermost boride layer and inner transition carburized layer. The thickness of boride layer and carburized layer are 38 μ m and

75 μm , respectively. From Fig. 3 b, the EDS results indicated that the distribution of beneficial elements are gradient at the interfaces because of the metallurgical bonding effects, which is a typical feature of double glow plasma alloying technology [19]. Furthermore, the microstructures of boride layer differ from that of the conventional boriding process. The needle-like microstructure cannot be discovered [27]. Salim Sahin et al. [28] indicated that the microstructure of boride layer depends on the temperature, treating time, and the ratio of alloying elements.

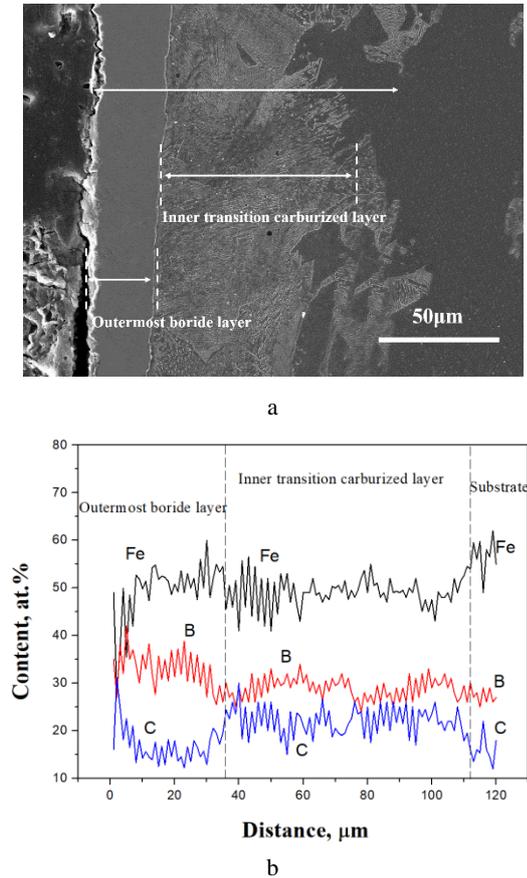


Fig. 3. a – cross-section microstructure; b – EDS results of borocarburized layer

The XRD pattern of substrate and borocarburized layer is shown in Fig. 4. After carburizing process, the main phase of sample is Fe_5C_3 phase, and Fe_7C_3 and $\text{C}_{0.09}\text{Fe}_{1.91}$ are discovered at the same time. The results indicated that carbon atoms diffuse into substrate participated in chemical reaction with iron atoms to form iron-carbon compounds. In addition, only Fe_2B phase is detected in Fig. 4 c, and the FeB phase in conventional boriding process is absent. The results are consistent with the cross-section microstructure (see Fig. 3 a). And the content of FeB and Fe_2B phase in boride layer principally depends on the ratio of alloying elements and temperature. In general, the XRD technology cannot detect the phase with very lower content (less than 5 %). Furthermore, the phases of iron-carbon compounds in transition layer cannot be detected in Fig. 4 c because of the high depth from the surface of boride layer to cauburized layer.

Fig. 5 shows the microhardness distributions of borocarburized layer. The results indicated that the

microhardness profiles of borocarburized layer from the surface of coating to the substrate is glaxis, which could improve the service life of coating in a harsh environment.

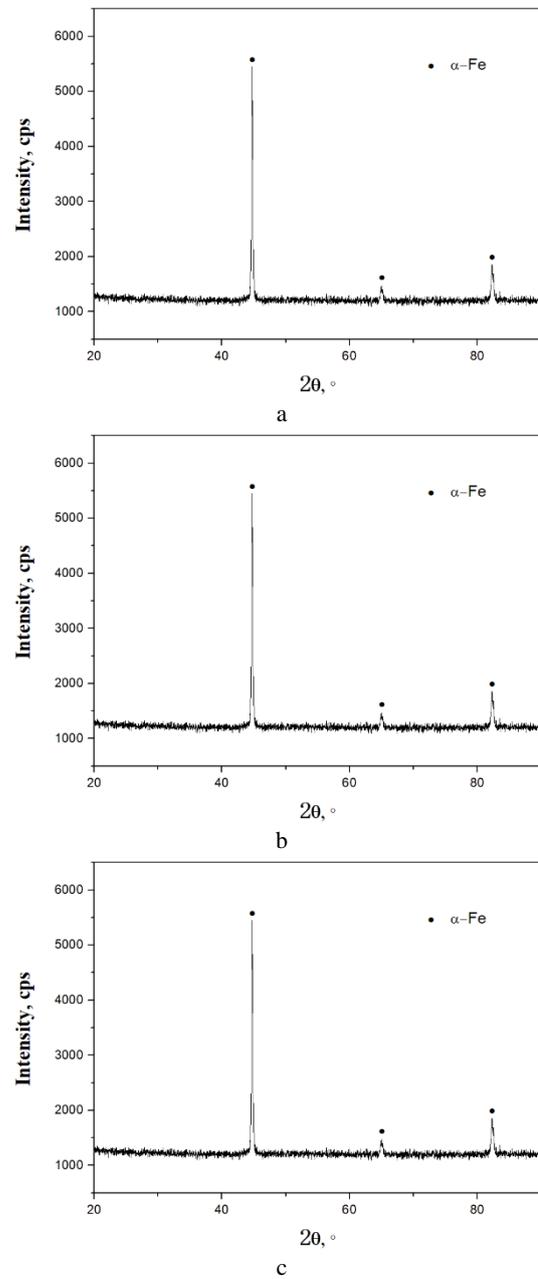


Fig. 4. XRD patterns: a – substrate; b – after the carburizing process; c – after boriding process

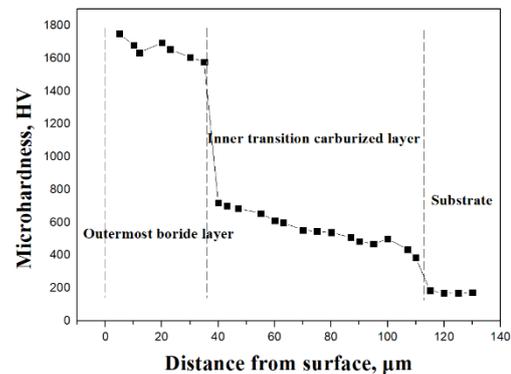


Fig. 5. The microhardness distributions of borocarburized layer

The effective hard-layer of borocarbured layer is boride layer with the hardness around 1700 HV. And the transition layer (carbured layer) has a hardness around 600 HV, which delays the hardness gradient between coating and substrate. Above all, the borocarbured layer has an optimized structure, which enhances the hardness of substrate effectively and prevents the outermost layer (boride layer) peeling off.

3.2. Tribological behavior

Fig. 6 shows the friction coefficients of substrate and borocarbured layer under dry-sliding against ZrO_2 ball with 730 g load at 20 °C, 200 °C, and 500 °C respectively, and the test was repeated three times for every sample.

The results indicated that the fluctuations of friction coefficients curves of both substrate and borocarbured layer become larger with the increase of temperature. The friction coefficient of substrate at ambient temperature is 0.9 and increases slightly with the rise of environment temperature. However, the friction coefficient of borocarbured layer is only 0.25 at ambient temperature, but it suddenly increases to 0.67 at 200 °C. The friction coefficient of borocarbured layer changes larger with the temperature increase. The friction coefficient of borocarbured layer is much lower than that of substrate at the same conditions, which indicate that the borocarbured layer effectively improves the wear resistance of low carbon steel. Comparing with the friction coefficients of substrate and borocarbured layer, which indicated that the change of that relate to the difference of surface hardness. Krelling A P et al. [29, 30] pointed that the higher hardness of sample led to the lower friction coefficients. During the wear tests, boride layer with Fe_2B phase is much harder than substrate, which reduces the real contact areas against the friction pair of ZrO_2 ball. With the increase of temperature, the fluctuations of friction coefficients curves of both substrate and borocarbured layer become larger due to the surface roughness of samples. At high temperature, the oxidation of substrate and borocarbured layer occurs easily. The oxide film was broken by friction pair during wear process, and then a lot of abrasive dust was produced immediately, which induced the crash between friction pair and convex parts of substrate surface.

Fig. 7 illustrates the cross-section profiles of wear tracks of Q235 low carbon steel and borocarbured layer at the temperature of 20 °C, 200 °C, and 500 °C, respectively. As to substrate, the width and depth of wear tracks increase sharply as the temperature increases because of the oxidation occurs during the wear process. However, the width and depth of borocarbured layer are much smaller than substrate due to the higher hardness of coating at the same conditions.

The results are consistent with the friction coefficients in Fig. 6. In addition, the cross-section profiles of borocarbured layer greatly changes from 200 °C to 500 °C due to the oxide film broken and abrasive dust produced during the wear process.

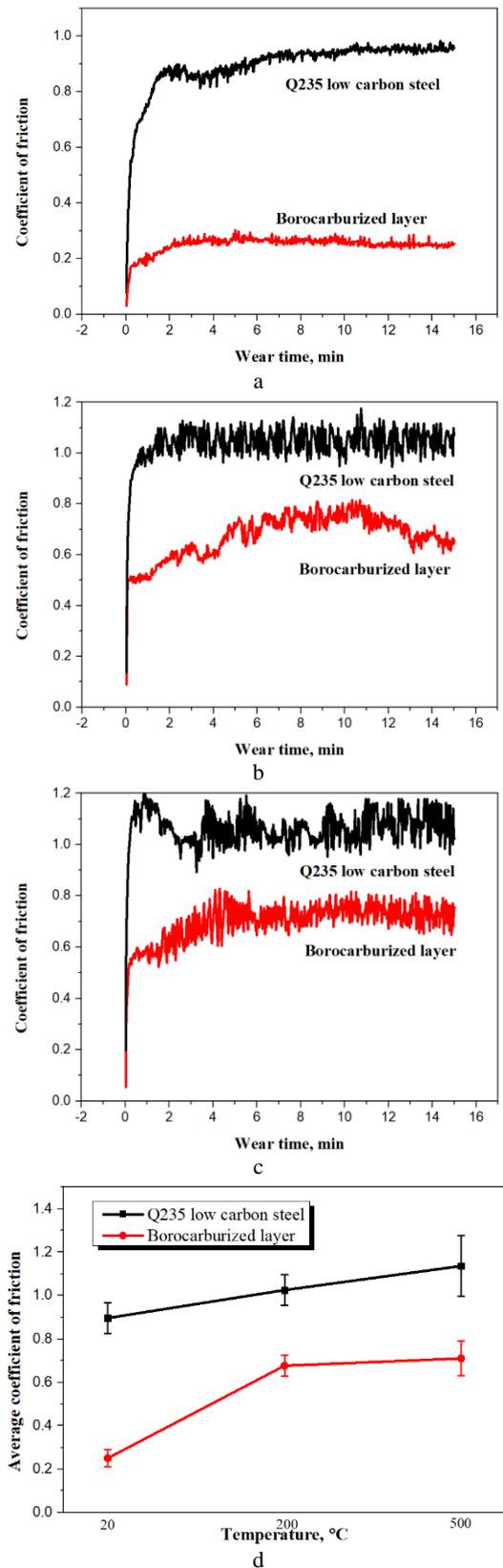


Fig. 6. Friction coefficients of substrate and borocarbured layer under dry-sliding against ZrO_2 ball at different temperature: a – ambient temperature; b – 200 °C; c – 500 °C; d – average coefficient of friction

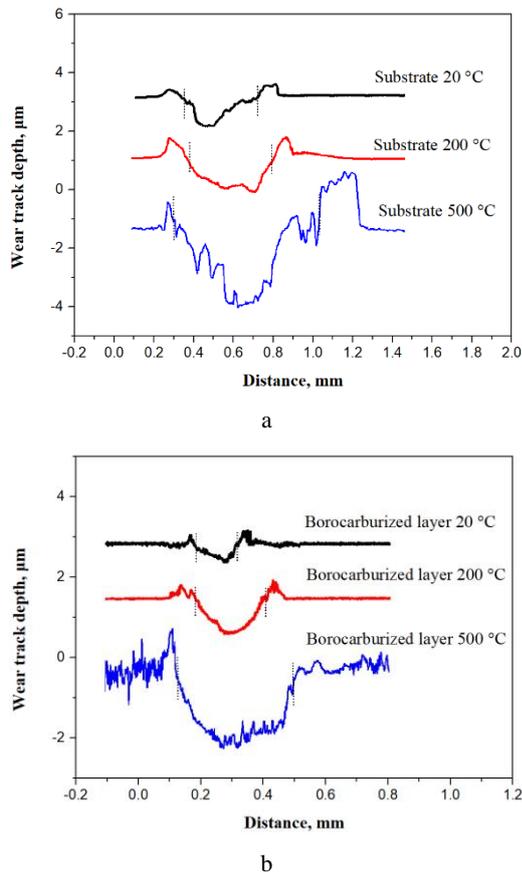


Fig. 7. The cross-section profiles of wear tracks: a–substrate; b–borocarburized layer

Above all, the areas of wear track of borocarburized layer is much smaller than that of substrate at the same condition, which means the borocarburized layer can improve the wear performance at high temperature.

The wear results of O235 low carbon steel and borocarburized layer were calculated from the cross-section profiles of wear tracks, which were displayed in Table 2.

Table 2. Wear results of substrate and borocarburized layer

Specimens	width b , mm	depth h , μm	volume loss V , 10^{-4}mm^3	wear rate v , $10^{-6}\text{mm}^3\text{m}^{-1}$	specific wear rate K , $10^{-7}\text{mm}^3\text{N}^{-1}\text{m}^{-1}$
Substrate 20°C	0.36	1.2	36.17	24.11	33.71
Substrate 200°C	0.43	1.3	46.81	31.20	43.61
Substrate 500°C	0.76	3.1	197.28	131.52	183.84
Borocarburized layer 20°C	0.1	0.28	2.34	1.56	2.18
Borocarburized layer 200°C	0.21	0.67	11.78	7.85	10.98
Borocarburized layer 500°C	0.32	1.2	32.15	21.44	29.96

As for substrate, the wear rate of substrate at 20 °C is $24.11 \times 10^{-6} \text{mm}^3\text{m}^{-1}$, which increase rapidly to $131.52 \times 10^{-6} \text{mm}^3\text{m}^{-1}$ with the temperature rise to 500 °C. And the specific wear rate of it increase rapidly from $33.71 \times 10^{-7} \text{mm}^3\text{N}^{-1}\text{m}^{-1}$ to $183.84 \times 10^{-7} \text{mm}^3\text{N}^{-1}\text{m}^{-1}$. The results indicate that the substrate is broken badly because of the oxidation at high temperature during the wear process.

However, the wear rate and specific wear rate of borocarburized layer are much lower than that of substrate at the same condition. The wear rate and specific wear rate relate to surface hardness closely, and hardness of the outermost layer (boride layer) is around 1700 HV, which is much higher than that of substrate (around 200 HV). Above all, the borocarburized layer with high hardness improve the wear resistance of low carbon steel at high temperature environment.

The high magnification scanning images of wear tracks shows in Fig. 8 to understand the wear behaviors of Q235 low carbon steel and borocarburized layer under the dry-sliding against ZrO_2 ball with 730 g load at 20 °C, 200 °C, and 500 °C respectively. Fig. 8 a, c, and e shows the wear surface of substrate at different temperature. The results indicate that a lot of fatigue cracks appear on the wear track at 20 °C, and a large of peeling could be observed at the same time. The wear mechanism of substrate at 20 °C presents a feature of fatigue wear and slightly adhesive wear. At 200 °C, the adhesion transfer can be observed easily and a lot of abrasive dusts appeared randomly on wear surface, the wear mechanism is mainly adhesive wear and fatigue wear. With the temperature rise to 500 °C, the much severe wear appears on wear surface. Many abrasive dusts are scattered on the wear surface, and some melting materials can be observed on it, which attributes to the local high temperature generated at the interface between ZrO_2 ball and sample surface during the wear process. The higher temperature can cause the oxidation, sintering, and the filling of abrasive dusts which is adverse for the wear resistance. Wear mechanism of substrate at 500 °C is mainly adhesive wear and oxidation wear. Fig. 8 b, d, and f shows wear surface of borocarburized layer at 20 °C, 200 °C, and 500 °C, respectively. Comparing with the substrate, the deformation of borocarburized layer is much lower which attribute to the higher hardness of the outermost layer (boride layer) in the coating. At 20 °C, there was no fatigue crack and peeling occurred on the wear track during wear process. When the is increased to 200 °C, slight wear scratches and fine cracks can be observed on wear surface of borocarburized layer, but no extensive spalling or large cracks on it, which indicates that the wear mechanism is slight abrasive wear. At 500 °C, the parallel grooves appear on the wear surface, and relatively smooth wear surface appears evidence of the plastic flow. The results indicate the wear mechanism of borocarburized layer at 500 °C is main oxidation wear and adhesive wear, but the coating has not been damaged seriously and the wear of it is much weaker than that of substrate due to the great thermal stability of the outermost layer (boride layer) [31]. These suggest that the borocarburized layer effectively improves the wear resistance of low carbon steel at high temperature.

The EDS results of areas I and II in the wear morphology of wear tracks of substrate and borocarburized layer are listed in Table 3. The concentration of Zr and O in area I are much higher than that in area II due to the longer contact between friction pairs of ZrO_2 and area I during the wear proves. As for both substrate and borocarburized layer, the concentrations of Zr and O increase with the temperature rise, which attributes to the oxidation and wear are serious at higher temperature.

Table 3. The EDS results of areas I and II in the wear morphology of wear tracks of substrate and borocarbured layer

Weight percentage, %	Fe		Zr		O	
	Area I	Area II	Area I	Area II	Area I	Area II
Q235 Substrate 20 °C	69.25	71.96	17.95	16.12	12.80	11.92
Q235 Substrate 200 °C	62.17	65.82	19.26	18.73	18.57	15.45
Q235 Substrate 500 °C	56.13	58.46	24.15	21.06	19.72	20.48
Borocarbured Layer 20 °C	77.74	81.02	10.28	9.11	11.98	9.87
Borocarbured Layer 200 °C	67.64	70.08	13.37	12.57	18.99	17.35
Borocarbured Layer 500 °C	65.11	66.75	15.22	14.17	19.67	19.08

Comparing with the substrate, the concentration of Zr and O of borocarbured layer are much lower at the same conditions. The results suggest that the borocarbured layer with high hardness which leads to the abrasive dusts from friction pairs are transferred to the edge of wear tracks [32].

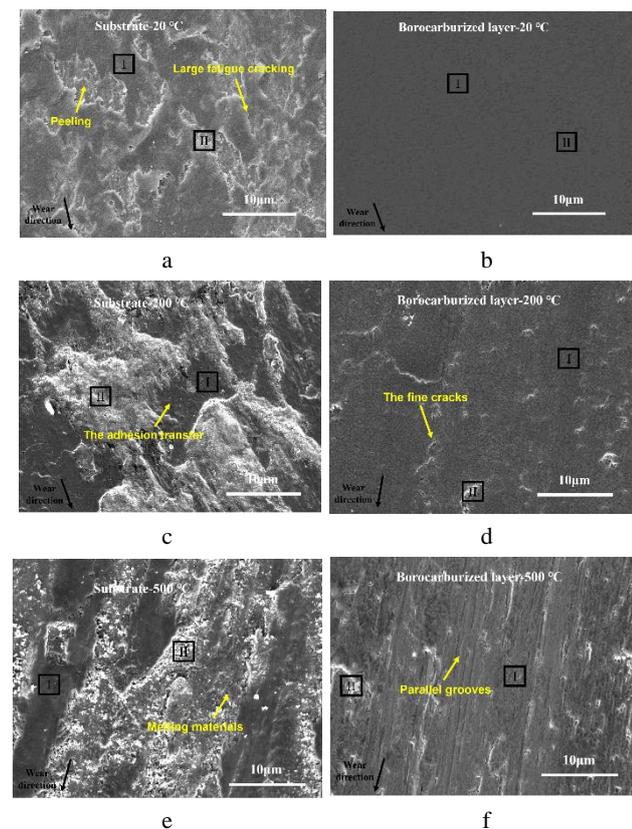


Fig. 8. Wear morphology of wear track at ambient temperature, 200 °C, and 500 °C, respectively: a, c and e—substrate; b, d and f—borocarbured layer

4. CONCLUSIONS

A borocarbured layer with high hardness was fabricated on the surface of Q235 low carbon steel via double glow plasma alloying technology. The protective coating contains an outermost boride layer and an inner carburized layer, which decreased the hardness mismatch to improve the service life of the coating. The borocarbured layer manufactured by double glow plasma alloying treatment has a gradient structure, and the adhesion between coating and substrate is strong because of the metallurgical bonding effects.

The borocarbured layer contains a boride layer with main phase of Fe_2B and a carburized layer with main phase

of Fe_3C_3 , which has no obvious defects. The outermost layer (boride layer) has the high hardness around 1700 HV, and the hardness of transition layer (carburized layer) is around 600 HV.

The friction coefficients and specific wear rates of substrate and borocarbured layer under the dry-sliding against ZrO_2 ball increase with rising temperature. But the friction coefficient and specific wear rate of borocarbured layer are much lower than that of substrate at the same temperature. The results suggest that borocarbured layer with high hardness can effectively improve the wear resistance of substrate at high temperature environment.

The wear mechanism of substrate is mainly fatigue wear and slightly adhesive wear at 20 °C. When the wear test performs at 200 °C, the substrate wear mechanism is adhesive wear and fatigue wear. As for borocarbured layer, the main wear mechanism is abrasive wear at 20 °C and 200 °C. When the temperature increases to 500 °C, the wear mechanism of both substrate and borocarbured layer are main oxidation wear and adhesive wear. The broken of wear tracks of borocarbured layer is lower than that of substrate, which indicates the borocarbured layer effectively improves the wear resistance of low carbon steel at high temperature.

Acknowledgments

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