Tribological behavior of borocarburized layer on low-carbon steel treated by double glow plasma surface alloying

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Abstract

In this paper, the borocarburized layer was fabricated on the surface of low-carbon steel via double glow treatment to enhance the wear resistance. Benefited by the gradient structure of an outermost boride layer and inner carburized layer, the bonding strength of the protective coating was improved. The novel design improved the mechanical properties at the coating-substrate interface and extended the service life of the coating. The microstructure and composition of borocarburized layer were analyzed by SEM, XRD, XPS. The tribological properties of substrate and borocarburized layer under dry sliding against ZrO2 ball were investigated. The results indicated that borocarburized layer consisted of a 38 μm boride layer with main phase of Fe2B and a 75 μm carburized layer with main phase of Fe5C3. The microhardness of borocarburized layer emerged in gradient distribution attributed to the double glow plasma surface alloying technique, and the maximum value was around 1700 HV. In addition, the major wear mechanisms of low carbon steel were fatigue and abrasive wear, which transformed to abrasive and adhesive wear with the load increased. The wear mechanisms of borocarburized layer were abrasive and adhesive wear. During wear process, borocarburized layer with high hardness against ZrO2 ball due to the decreased real contact areas. Therefore, the borocarburized layer fabricated on low carbon steel enhanced the wear resistance effectively.

1. Introduction

Low carbon steel is widely used in building construction, vehicle, bridge structure, and mechanical part due to the favorable properties of weldability, plasticity, and toughness [1–4]. However, the low carbon steel is limited to the areas of non-tribological applications because of the poor wear resistance. In the past, many hard coatings with the different elemental composition (nitriding, carburization and nitrocarburizing) were carried out, which mainly concerned the tribological behaviors of low carbon steel [5–8]. Furthermore, the boriding is another surface modification method with the hardness around 2000 HV [9–11], the boride layer is much harder than nitriding, carburization or nitrocarburizing layers.

Generally, the boriding is an effective surface modification method with superior wear resistance. However, the single boride layer is much harder than substrate, which will cause the boride layer peeling off in the service environment [12]. In addition, the traditional boride layer with double-layer structure (FeB and Fe2B phase) has a serious drawback resulting from the cracks in boride layer due to the difference thermal expansion coefficient between FeB phase and Fe2B phase [13]. To resolve the problems, many scholars are carried out on multilayered structure of boron, carbon and nitrogen layers [14, 15]. Wang et al [16] had fabricated the borocarburized layer by plasma electrolytic saturation technique aiming at improving the wear resistance of steels. Kulka et al [17] designed the complex (B + C) diffusion layers by gas carburizing and gas boriding process, which might be concluded that the borocarburized coating exhibited great wear resistance. Shiwei Zuo et al [18] fabricated the borocarburized coating by pack cementation process to improve the wear resistance of substrate. However, the environmental contamination and the toxic nature cannot be avoided during these treating processes, the boride
layer was undesired due to the double-layer structure. Thus, it is meaningful to search an effective method to improve the hardness and wear resistance of substrate, which will be beneficial for the stable operation of the workpiece in a harsh environment.

Recently, double glow technique is a novel method called Xu-Tec by others, and has been applied successfully to fabricate many coatings, such as C, Nb, Cr, and Mo, etc. The plasma region produced by double glow discharge is the most remarkable characteristic. The surfaces of target and substrate were activated due to the bombardment by argon ions with a certain high kinetic energy, which is beneficial due to the diffusion of elements inside the substrate. The target stroke by argon ions with high ionization and beneficial elements is sputtered to the substrate [19–21]. The sputtering rate of beneficial elements can be controlled by double glow technique, which is advantageous for obtaining an ideal coating structure. The double glow technique has an obvious advantage which will not cause pollution to the environment. In addition, using double glow plasma surface technology can save precious metal resources and reduce energy consumption in industrial production, the double glow technique can also be applied to large surface areas [22–24].

In this study, specimens of Q235 were subjected to borocarburized coating via double glow technique. Firstly, the substrate is carburized to prevent the spalling of boride layer, and then boride layer fabricated on the carburized samples to enhance the property of hardness and wear resistance principally. The microstructure, hardness and wear resistance of borocarburized layer were investigated, and wear mechanism of borocarburized layer was discussed at 330 g, 530 g, and 730 g respectively.

2. Experiment

The samples of Q235 is a kind of low-carbon steel, and the demission of the sample is \(10 \times 10 \times 8\) mm by using wire cutting. During the pretreatment, all samples were polished by the water-proof abrasive paper of 1200 meshes, and cleaned in acetone by ultrasound equipment to dislodge any surface contamination.

The borocarburized layer was fabricated via double glow equipment which was composed of two parts (figure 1): the cathode (workpiece) and anode (target and device chamber). The inert gas of argon was used during the deposited process, which was excited to active \(\text{Ar}^+\) due to the double glow discharge. The target and workpiece were overlaid by glow discharge when the current supply was switched on, which were activated because of the bombardment by \(\text{Ar}^+\). The double glow discharge could transfers heat and ions from target to workpiece because the active ions (\(\text{Ar}^+\)) hit target incessantly with a certain high kinetic energy [20]. Borocarburized layer was fabricated by double glow treatment can be divided into two steps. Firstly, carbon deposition on the substrate by pure carbon target, and then boron deposition on the carburized samples by \(\text{B}_4\text{C}\) target. The experimental parameters are listed in table 1.

The surface and cross-section microstructures of borocarburized layer were examined by scan electron microscopy (FEI, Quanta450, USA). Main phase compositions of borocarburized layer were calculated by x-ray diffraction (Rigaku, DMAX-RB12KW, Japan) with copper Kα radiation (\(\lambda = 1.5418\ \text{Å}, 20^\circ \leq 2\theta \leq 80^\circ\)). The x-ray photoelectron spectroscopy (Thermo, Escalab-250, USA) was used to detect the chemical states and the compositions of borocarburized layer. The micro-hardness of borocarburized layer was characterized by Vickers Indenter (DianYing, DHV-1000/2, China) with the applied load of 50 g and 15 s dwell time. The scratch tester (BangYi, WS-2005, China) was used to calculate the bonding strength between protective coating and
substrate, which has the Rockwell diamond indenter with radius of 200 μm, the maximum load was 100 N, the 25 N min⁻¹ was designed for rate of load, the indenter was drawn on the coating with a speed of 1 mm min⁻¹.

The tribological behaviors of substrate and borocarburized layer were tested by the ball-on-disk friction and wear tester (KaiHua, HT-500, China) in air with relative humidity of (45 ± 5)% under dry sliding against ZrO₂, the normal temperature was 20 °C and 560 rpm rotating rate with a turning radius of 2 mm at 330 g, 530 g, and 730 g respectively. The morphologies of wear tracks were determined by the depth-of-field system (Keyence, VHX-1000, Japan).

3. Results and discussion

3.1. Borocarburized Layer characterization

Figure 2 shows the microstructure of borocarburized layer on low-carbon steel. After plasma carburization treatment, the surface microstructure of the sample is exhibited in figure 2(a). The high carbon martensite has the characters of homogeneous and dense. Figure 2(b) indicates that the surface microstructure of borocarburized layer is consist of dense submicron-sized particles arranged in a stacked structure. This is the typical feature of double glow techniques. The results suggested that the beneficial elements of carbon and boron atoms were sputtered by active Ar ions, which got a certain high kinetic energy from target to sample due to the

| Parameter                                | Values (C Deposition) | Values (B Deposition) |
|------------------------------------------|-----------------------|-----------------------|
| The voltage of the source electrode (V)  | 950                   | 950                   |
| The 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                    |

![Figure 2. SEM images of borocarburized layer (a) the carburized layer, (b) borocarburized layer, (c) cross-section image (d) EDS result.](image-url)
difference of the voltage. The beneficial elements diffused into sample and had a chemical reaction with the sample.

In addition, the cross-section microstructure of borocarburized layer and the corresponding EDS results manifested that borocarburized layer consists of an outermost boride layer and inner transition carburized layer (figures 2(c) and (d)), which displays a multilayered structure. The boride layer exhibits superior quality without holes or cracks, and the high hardness will enhance the wear resistance. The carburized layer between substrate and boride layer is a transition layer, which can effectively reduce the hardness mismatch of the coating and prevent boride layer peeling. The EDS results of cross-section are along the depth direction (see figure 2(d)) which indicates that the thickness of boride layer and carburized layer are 38 μm and 75 μm, respectively. The distribution of beneficial elements is gradient at the interfaces due to the metallurgical bonding effects, which is a typical feature of double glow plasma alloying technology. Furthermore, the needle-like microstructure of boride layer cannot be discovered, which is different from that of the conventional boriding process [9, 13]. Kusmanov et al [11] indicated that the microstructure of boride layer depends on the temperature, treating time, and the ratio of alloying elements. Comparing with other borocarburized layer process [15–17], the borocarburized layer fabricated by double glow treatment is homogeneous and dense due to the uniform sputtering with a certain high kinetic energy. There are no loose oxides formed on the top of the coating because of the pure B4C target and the vacuum environment during double glow treatment. During glow discharge processing, the beneficial ions or atoms sputtered from the target and penetrated into the substrate due to the sufficient energy.

The XRD pattern of borocarburized layer is shown in figure 3. After carburizing process, the Fe5C3 phase is the main phase at the coating surface. The phase of Fe7C3 and C0.09Fe1.91 are also detected because carbon atoms diffuse into substrate and have chemical reaction with iron atoms. However, the XRD result of borocarburized layer is only Fe2B phase. The other studies [15, 16] prepared borocarburized layer with many phases (Fe2B, Fe2C, Fe2O3 and FeB phase) because of the electrolyte with complex composition. The samples prepared by double glow technique have single Fe2B phase due to the vacuum environment and the boron atoms were sputtered from target to substrate and diffused into the surface under the high temperature during the treating process, which will be conducive to improve the stable operation of the workpiece in a harsh environment. The XRD results suggested that parts of carbon atoms and boron atoms diffused into the substrate during double glow treatment. The FeB phase [25] in conventional boriding process cannot be discovered, which might attribute to the very low content of FeB phase. In general, the content of the phase is above 5%, which could be detected accurately via XRD technology. Furthermore, the iron-carbon compounds in transition layer are not detected by XRD technology due to the depth from the surface to substrate.

In order to verify the accurate phases in the boride layer, XPS determination was executed on borocarburized layer, and the results are shown in figure 4. The B-1s spectra is decomposed into two peaks located at 188.23 eV and 187.22 eV, corresponding to Fe2B phase and FeB phase [26, 27]. Peak areas of Fe2B and FeB are calculated, which are 368.17 cnts·eV and 15.26 cnts·eV, respectively. The relative content of Fe2B could reach 95.86 At.%, which indicated that the main phase is Fe2B. The results of B-1s spectra is consistent with XRD patterns (see figure 3(b)), which admirably explained the reason that FeB phase disappears in XRD result of borocarburized layer. In addition, the Fe (2p½,3/2) spectra is shown in figure 4(b). The result indicated that main phase is Fe2B and the weak peaks of Fe2+ and Fe3+ are existent due to the small number of iron-compounds were generated during DG process. Wang et al [15] indicated that borocarburized layer fabricated by plasma electrolytic saturation technique which consists of B2O3, FeB and Fe2B phase, a mass of B2O3 phase is
detrimental to the application of the coating. However, the borocarburized layer prepared by DG process has no oxides due to the vacuum environment. During double glow treatment, the surfaces of substrate were activated due to the bombardment by Ar\(^+\) with a certain high kinetic energy, which is beneficial for the diffusion of boron atoms. The content of FeB phase is less to 4.14 At.% which suggested the boride layer is almost single Fe\(_2\)B phase. This is advantageous for the stable operation of the vorocarburized layer in a harsh environment.

Microhardness profiles of borocarburized layer are exhibited in figure 5. The results indicated that microhardness values of borocarburized layer gradually decrease from the coating surface to the substrate. The main hard-layer of coating is boride layer, which has a maximum hardness around 1700 HV. The hardness of substrate is about 200 HV, which is much lower than boride layer. The carburized layer (hardness around 600–400 HV) acts as a transition layer, which plays a role in reducing the hardness mismatch between the boride layer and substrate. The microhardness result indicates that borocarburized layer could enhance the hardness of the sample effectively, and the transition carburized layer is beneficial to prevent the boride layer peeling off.

The bonding strength between coating and substrate was detected by scratch tester. The results were shown in figure 6. When the load reaches 71.2 N, the acoustical signal began to wave, indicating that the microcracks appeared in coating and the load is called the critical load. When the load exceeded 90 N, the strong acoustical signal appeared continuously, which indicated that the protective coating fractured completely and be worn out by diamond indenter. Figure 6(b) shows the scratch image, which could be conclude that the borocarburized layer was slowly worn out and the scratch became wider and deeper gradually. Figure 6(c) shows the scratch

**Figure 4.** XPS spectra of borocarburized layer (a) B (1 s), (b) Fe (2p\(_{3/2}\)).

**Figure 5.** Microhardness distributions of borocarburized layer.
image of 80–100 N stage, which indicated that the furrows are very deep and wide. However, the peeling was not taken place at the end of scratch test, the grate bonding strength provided a pledge of good wear resistance.

3.2. Tribological behavior
The ball-on-disk friction and wear tests were directed to evaluate the tribological properties of substrate and borocarburized layer. Figure 7 shows friction coefficients of borocarburized layer and substrate under dry-sliding against ZrO₂ ball with 330 g, 530 g, and 730 g load at room temperature. The results indicated that friction coefficients of the substrate (figure 7(a)) increased rapidly at the initial stage in wear process. With the increment of testing time, values of friction coefficients remain stable between 0.8 and 1.0. And the average friction coefficients of substrate had slightly affected by the increasing load. The friction coefficients curve of substrate fluctuated greatly due to the surface roughness, resulting from the abrasive dust between ZrO₂ ball and convex parts of the substrate surface.
Figure 7(b) shows friction coefficients of borocarburized layer, and the results indicated that values of friction coefficients were lower than substrate obviously, which were basically stable between 0.18 and 0.29. And the average friction coefficients also increase slightly with increasing load. Comparing with results of figures 7(a) and (b), the change of friction coefficients between borocarburized layer and substrate was related to the difference of surface hardness (see figure 5). The previous studies pointed out that [28, 29] the sample with higher hardness lead to lower friction coefficient of it. During wear process, Fe2B phase with high hardness reduced the real contact parts against the friction pair.

Figure 8 shows surface profilometry of wear tracks of the substrate and borocarburized layer at the load of 330 g, 530 g, and 730 g, respectively. For substrate, the width and depth of wear track became larger with the increased applied load. The wear track of substrate with 730 g was largest obviously because of the heaviest load condition. As for borocarburized layer, the width and depth of wear track were also increased with the applied load from 330 g to 730 g. However, the areas of wear tracks were smaller than those of substrates obviously.

The wear results of the substrate and borocarburized layer were calculated from surface profilometry, which was listed in table 2. To evaluate friction and wear performances, wear volume of the sample was calculated by the following equation [30].

\[ V = \frac{2\pi hr}{6b} \cdot (3h^2 + 4b^2) \]  

(1)

Where \( V \) is the volume of wear area, \( h \) is the depth of wear track, \( b \) is the width of wear track, \( r \) is radius of wear track.

The wear rate is the relationship between wear volume and sliding distance. The specific wear rate is the real measure of friction and wears properties, which is by equations (2) and (3) respectively.

\[ v = \frac{V}{S} \]  

(2)

\[ K = \frac{V}{SP} \]  

(3)

Where \( v \) is wear rate, \( S \) represents sliding distance, \( K \) is specific wear rate, \( P \) is the load.

The values of wear rate and specific wear rate gradually increased as the load increased for substrate and borocarburized layer respectively. However, the Q235 steel had a larger wear rate and specific wear rate than that of borocarburized layer under the same load conditions. The wear results indicated that borocarburized layer was fabricated on Q235 low carbon steel can improve the wear resistance. The wear rate and specific wear rate heavily depend on the surface hardness (see figure 5). The hardness of borocarburized layer was larger than that of substrate due to the formation of boride layer and low hardness of Q235 (around 200 HV). During wear process, the substrate suffered considerable wear and friction heat was generated at the interface between ZrO2 ball and sample surface would be much higher than borocarburized layer. Finally, wear rates and specific wear rates of substrate were larger than that of borocarburized layer obviously.

3.3. Tribological mechanism

The high magnification scanning images of wear tracks (figure 9) shows tribological morphology of substrate and borocarburized layer at the load of 330 g, 530 g, and 730 g, respectively. As for substrate, the severe peelings and large fatigue cracking could be observed on the wear track under 330 g load condition. This phenomenon suggested a feature of fatigue wear and slightly abrasive wear. With the load increased to 530 g, a lot of particles
Table 2. Wear results of the substrate and borocarburized layer.

| Specimens               | Width b/mm | Depth h/μm | Volume loss V/10^{-4} mm³ | Wear rate v/10^{-6} mm³ m⁻¹ | Specific wear rate K/10^{-7} mm³ N⁻¹ m⁻¹ |
|-------------------------|------------|------------|---------------------------|------------------------------|------------------------------------------|
| Q235 Substrate-330 g    | 0.18       | 0.3        | 4.52                      | 3.02                         | 9.32                                     |
| Q235 Substrate-530 g    | 0.22       | 0.38       | 7                         | 4.67                         | 8.98                                     |
| Q235 Substrate-730 g    | 0.36       | 1.2        | 36                        | 24.11                        | 33.71                                    |
| Borocarburized Layer-330 g | 0.04       | 0.12       | 0.4                       | 0.27                         | 0.82                                     |
| Borocarburized Layer-530 g | 0.08       | 0.19       | 1.27                      | 0.85                         | 1.63                                     |
| Borocarburized Layer-730 g | 0.1       | 0.28       | 2.34                      | 1.56                         | 2.18                                     |
appeared randomly on the wear surface, which produced deep furrows. The wear mechanism was dominated by abrasive wear simultaneously accompanied by adhesive wear. As the load increased to 730 g, the serious delamination appeared on the wear track, and plastic flow could be found easily from the wear morphology. The wear mechanism of substrate transformed to adhesive wear. From the wear morphology of the substrate, the phenomenon of plastic deformation, peeling, and huge scratches indicated that wear resistance of low substrate was poor due to the low hardness. However, no fatigue crack and peeling occurred on borocarburized layer surface. With the load increased, slight wear scratches and deformation disappeared gradually. Therefore, wear morphology results suggested that borocarburized layer was much harder than the substrate, which decreased the real contact areas during the wear process. In addition, high hardness of borocarburized layer consisted of boride layer (outermost layer) and carburized layer (inner transition layer), which made it difficult to deform and peel off. Above all, low carbon steel was destroyed seriously after wear treatment. Major wear mechanisms of substrate were fatigue and abrasive wear, which transformed to abrasive and adhesive wear with the load increased. However, wear morphology of borocarburized layer manifested that only slight wear scratches and deformation were observed on wear track, and major wear mechanisms were abrasive and adhesive wear. The

Figure 9. wear morphology of wear track at 330 g, 530 g and 730 g load, respectively (a), (c) and (e) substrate, (b), (d) and (f) borocarburized layer.
borocarburized layer fabricated on low carbon steel enhances wear resistance effectively. Comparing with other borocarburizing processes [16, 17], the surface profilometry of wear tracks and the wear rate of borocarburized layer fabricated by double glow technique were smaller and lower than others. No holes or cracks appeared on the wear tracks of double glow samples. This could be attribute to the homogeneous and dense of the coating, resulting from the uniform sputtering by double glow technique. In addition, the furnace atmosphere was vacuum, which could avoid the generation of oxide and hydrogen embrittlement. The borocarburized layer prepared by double glow technique can guarantee a better purity. However, the efficiency of double glow technique was lower than others, which is the major aspect to be improved in our future work.

4. Conclusion

The fabrication of protective borocarburized layer on the surface of Q235 steel via double glow treatment was to improve the property of wear resistance. The protective coating had desirable bonding strength because of the gradient structure of an outermost boride layer and inner carburized layer, which decreased the hardness mismatch at the coating-substrate interface and improved the service life of the coating.

(1) The borocarburized layer had no obvious defects, and the thickness of boride layer and carburized layer are 38 μm and 75 μm, respectively. The phase of boride layer was main Fe₂B, which had high hardness around 1700 HV. The carburized layer as a transition layer with hardness around 600–400 HV was main Fe₃C₃ phase. The bonding strength between the coating and substrate was about 71.2 N. At the end of scratch test, no peeling taken place, representing a grate bonding strength provided a pledge of good wear resistance.

(2) The friction coefficients of borocarburized layer were much lower than low carbon steel under the same load conditions. Furthermore, the wear rates and specific wear rates of low carbon steel were larger than that of borocarburized layer during the wear process. The major wear mechanisms of low carbon steel were fatigue and abrasive wear, which transformed to abrasive and adhesive wear with the load increased. The wear mechanisms of borocarburized layer were abrasive and adhesive wear. Borocarburized layer was much harder than the substrate, which decreased the real contact areas during the wear process. Therefore, the borocarburized layer fabricated on low carbon steel enhances wear resistance effectively.

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