Tribological studies of carbonitrided coatings synthesized on the 300 M steels by double-glow plasma alloying

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Abstract
The carbonitrided coatings were synthesized on the 300 M steel substrates using a new method named double-glow (DG) plasma alloying. The phase composition and microscopic morphology of the coatings were investigated by scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS) and x-ray diffraction (XRD). When the substrate is treated at 950 V source voltage and 500 V workpiece voltage for 5 h, the maximum diffusion thickness is about 100 μm, and the corresponding average microhardness is 10.1 GPa, which is significantly higher than that of substrate. The phase composition of the carbonitrided layer was composed of γ′-Fe₄(C, N), ε-Fe₃(C, N) and a small amount of CrN phases. Moreover, the wear tests showed that the wear resistance of the carbonitrided specimen was remarkably improved as compared with the substrate.

Introduction
300 M steel is a kind of low-alloy and ultrahigh-strength steel [1]. It is a standard material for the landing gear of the aerospace industry due to its extra strength, high fracture toughness and excellent corrosion resistance [2–4]. Unfortunately, 300 M steel workpieces cannot fully guarantee their service life, especially under reciprocating friction or heavy load [5]. Many researches have involved surface treatments to build up hardness and wear resistance, in which the most common process is carburizing or nitriding [6–9]. The carbonitriding process is one of the most widely used surface hardening techniques in the industry, mainly because of its simple operation and good performance. [10–12]. There are many techniques that can be used for carbonitriding, such as gas carbonitriding, chemical vapor deposition, and electrolytic plasma carbonitriding [13–16]. Gas carbonitriding is widely used in industrial production and has the advantages of high infiltration rate and being unrestricted by shape. However, since the NH₃ and CH₄ are used as reaction gases, the introduction of H element easily leads to hydrogen embrittlement cracking in the workpieces. Kusmanov et al [17] reported that they synthesized nitrocarburised coating on Q235 steel using anode plasma electrolytic and obtained a hardened surface layer microhardness up to 1060 HV during 5 min at 750 °C, and the total thickness of the effective hardening layer was about 120 μm. Nevertheless, this process would inevitably form an oxide layer with certain thickness on the surface and reduce its service life. Despite the flexibility of these methods, these processes or products have certain drawbacks [18]. Therefore, it has become critical to choose a new method to improve process and product quality.

Double glow (DG) discharge plasma surface metallurgy process was a new surface modification technology invented by Professor Zhong Xu based on the principle of ion nitriding in 1985, which was called Xu-Tec Process in international [19, 20]. The unique advantage of this technology was that it can penetrate solid alloying elements into the surface of metal materials thus forming on the surface of the substrate not only a deposit layer but also a diffusion layer, which was difficult for other processes to achieve. This process has successfully produced various coatings, some of which have even been industrialized, such as Zr, Cr–Ni and NiCoCrAlY
Qiu et al. [24] reported that they deposited on gear steel a dense W-Mo coating with excellent wear resistance and high adhesion strength. Zhang et al. [20] had applied this new technology to synthesize a hydrogen-free carbonitriding layer on the surface of titanium alloy, which had significantly improved mechanical and friction properties. Moreover, DG has the advantages of low energy consumption and non-pollution, especially the high strength of interface bonding between the coating and the matrix as a result of mutual diffusion, thus having wide development space and a broad scope in future application [25–27]. Therefore, the application of this novel technology could effectively synthesize carbonitrided coatings on the 300 M steel to enhance its hardness and wearability.

This present study aims to elucidate the wear resistance of the carbonitriding diffusion layer prepared by double-glow plasma alloying technique. Moreover, the mechanical properties, phase composition and microstructures of the coatings were also analyzed.

**Experimental details**

**Coating synthesis**

In this research, carbonitriding coatings were synthesized on 300 M steel substrates using the DG method. This experimental device is mainly composed of three electrodes showed in figure 1, the metal vacuum cover as anode, the graphite target as source electrode and the samples as cathode [27]. When the double controllable DC power supplies were switched on, the cathode and source electrode respectively generated a set of glow discharge that could excite active plasma ions (Ar⁺) and (N⁺) to bombard the source electrode and cathode. While the Ar⁺ and N⁺ bombarded the source electrode, it would heat the source electrode and sputter the alloy element of the target. At the same time, the sputtered alloy element moved through the glow discharge space toward the cathode and adsorbs on the surface of the workpiece. And the Ar⁺ and N⁺ would also bombard the cathode, rendering it heated and forming a large number of atomic vacancies on the surface, which accelerated the alloying of the workpiece surface. The deposition process began when the vacuum chamber was evacuated to 8.0 × 10⁻³ Pa by a turbo-molecular-pump system, and the working pressure was 35 Pa. High purity Ar (99.999%) with a flow rate of 30 sccm and N₂ (99.999%) of 60 sccm were used as the working gas and the reactive gas, respectively. In this experiment, the graphite target is a disc 100 mm in diameter, 4 mm in thickness and 99.99% in purity, which is prepared by powder metallurgic method. There are some round holes evenly distributed on the target (figure 2), namely a honeycomb structure. This structure can enhance the sputtering
intensity of the glow discharge to the target and form a hollow cathode effect between the two cathodes, thereby rapidly heating the workpieces [19]. Based on this special structure, the workpiece reaches its maximum temperature (850 °C~900 °C) within an hour and then continues to stabilize until the end of the experiment. Some processing parameters were displayed in table 1.

The substrates material used is 300 M steel, and the chemical compositions of the specimen are (in wt%): 0.44% C, 1.8% Ni, 0.80% Cr, 1.64% Si, 0.78% Mn, 0.35% Mo, and 0.06% V, with the balanced Fe. Before coating, the specimens were cut into pieces of 15 mm × 15 mm × 5 mm, whose surfaces were polished by 80-1000 SiC waterproof sandpaper and then cleaned by ultrasonication in acetone solution.

Coating characterization
The chemical composition and microstructures of the carbonitrided layer were observed and analyzed by energy dispersive spectrum (EDS) and scanning electron microscope (SEM, JSM 6010LA). X-ray diffraction (XRD, D/Max 2500) with Cu Kα radiation in the range of 30 to 90° was used to characterize the phase composition of carbonitrided coatings. Microhardness and effective elastic modulus of 300 M steel specimen and the carbonitrided coating were evaluated by using nano-indentation system equipped with a Berkovich indenter. The load function involves loading to a maximum load of 150 mN within 15 s, then holding for 10 s under this load, and then unloading it within 15 s. The maximum indentation depth should be less than 10% of the thickness of the coating to eliminate any impact of the substrate on the coating. Moreover, the corresponding elastic modulus, and the microhardness were calculated by the Oliver-Pharr method [28, 29]. The tribological behaviours of the substrates and the carbonitrided coatings were investigated at 25 °C using a ball disc wear tester (HT-500, CAS). The counterpart was a ZrO2 ball 5 mm in diameter. The effects of different loads (5 and 15 N) on wear resistance were also investigated. And the friction coefficient were read directly from the computer connected to the tribometer. To further explore the wear profiles and wear scars depth, the 3D morphology of the wear tests specimens were examined using a super depth-of-field 3D system (CONTOUR GT-K).

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Table 1. The process parameters of carbonitriding layer prepared by double glow plasma surface metallurgy technique.

| Parameter                        | Value                  |
|----------------------------------|------------------------|
| Source voltage /V                 | 950 ~ 1000             |
| Cathode voltage /V                | 500 ~ 550              |
| Glowing atmosphere                | Ar/N₂                  |
| Workpiece temperature /°C         | 850 ~ 900              |
| Distance between source and cathode /mm | 12                    |
| Working time/h                    | 5                      |

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Figure 2. Schematic diagram of honeycomb graphite target structure.
Results and discussion

Microstructure

The surface SEM micrographs of the carbonitrided layer are shown in figure 3. Under the influence of glow discharge, a large amount of activated carbon and nitrogen atoms are formed between the double cathodes, and the surface of the sample is heavily bombarded [23]. Meanwhile, the iron atoms bombarded on the surface of the sample become carriers of activated carbon atoms and nitrogen atoms, and a large amount of iron-nitrogen and iron-carbon compounds are formed on the surface of the sample. The surface microstructure of the coating is shown in figure 3 (a), and the EDS element analysis for it is shown in table 2, which indicates that excess carbonitride particles are formed on the surface and stacked into a three-dimensional island shape about 2 to 5 μm in diameter. However, for this part of the deposited layer of carbonitride compound, we need to eliminate it in the later experiments, and the outer diffusion layer of the carbonitrided coating is shown in figure 3 (b).

Figures 3 (b1)∼(b3) is the distribution maps of C, N, Fe of carbonitrided coating. Although the surface still has some network structures formed by a three-dimensional island-like film formation mechanism, its quality is greatly enhanced [25]. The electronic image map shows a relatively uniform distribution of all detected elements, which will fully improve the performance of the coating.

Cross-sectional microstructure

Figure 4 displays the cross-sectional SEM of the carbonitriding layer, and the EDS element analysis of the four point positions of the a, b, c and d is shown in table 3. It can be seen that the nitrogen element indicates a decreasing trend, while the carbon element suggests a form of wave distribution with wide fluctuation. The reason for this variation is that during the formation of the compound layer, the nitrogen element is enriched outward while the carbon element diffuses inward. After etching with a 4% nitric acid solution, some clear boundaries of the carbonitrided layer can be found. The deposition layer, outer diffusion layer, interdiffusion
layer, and the matrix correspond to regions I to IV, respectively. The total thickness of the effective hardening layer is approximately 110 μm, while the compound layer and the interdiffusion layer is 40 μm and 60 μm, respectively [30]. To compare with other carbonitride processes, the samples prepared by DG technique have thicker compound layer, which will be conducive to improve the effective hardening layer and carrying capacity. Careful observation showed that the compound layer was homogeneous and dense without obvious holes, as the result of uniform sputtering by DG technique. In addition, the furnace atmosphere only comprised argon and nitrogen, which can avoid the generation of hydrogen embrittlement and oxide. However, we must admit that compared with other processes, the production efficiency of DG technique is relatively low, which is also an aspect to be improved in our future work.

X-ray microanalysis
Figures 5(a), (b) shows the x-ray diffraction patterns for the 300 M steel substrate and carbonitrided coating, respectively. It is evident that the phase composition of the carbonitrided layer is composed of γ′-Fe4(C, N) and ε-Fe3(C, N) in figure 5(b). It is well known that the ε phase is a solid solution based on Fe3(C, N) with close-packed hexagonal crystal lattice structure, and γ′ phase is a solid solution based on Fe4(C, N) with face-centered cubic lattice structure [12]. In this experiment, both γ′-Fe4(C, N) and ε-Fe3(C, N) phases exhibit the (111) preferred orientation. Tan et al [31] reported that the γ′-Fe4N composite layer will lead to higher hardness and bearing capacity. Careful observation reveals that there are some small peaks in figure 5(b) corresponding to CrN, as Cr in the matrix forms CrN in the plasma carbonitriding process. Meanwhile, the CrN phase has a high hardness and load carrying capacity, so a tiny amount of CrN contributes to the wear resistance of the carbonitrided layer [32]. Compared with the untreated 300 M steel pattern in figure 5(a), no α-Fe was found, and all the phases were new phases, which indicates that the nitrogen and carbon elements had diffused into the matrix and the reaction is quite complete. Moreover, the formation of these new phases is vital to enhance the mechanical properties, especially the hardness of carbonitrided layers. In addition, although the process took place at 1000 °C, since it was in a vacuum low pressure state, there was no oxide generation in the new phase, such as Fe2O3 or Fe3O4.

Scratch scar and nanoindentation
Figure 6 shows the scratched morphology and corresponding acoustic signal of the carbonitrided sample after polishing. Figure 6(a) illustrates that the movement of the indenter causes plastic deformation of the coating,
and the scratch gradually widens as the load increases. Moreover, figure 6(b) and the acoustic curve together suggest that since the carbonitrided layer is metallurgically bonded to the substrate, only a small acoustic wave signal appears at the end of the scratch. The entire scratch has no devastating and through cracks, indicating that the carbonitrided coating exhibits excellent plasticity and toughness and is well bonded to the substrate. In fact, the super adhesion strength is closely related to the density of the coating. However, the compound layer prepared by some heat treatment processes is uneven and brittle, which is one of the reasons why the compound layer of some aviation parts should be removed.

Nanoindentation experiments were used to research the elastic modulus and microhardness of the 300 M steel substrate and the carbonitrided diffusion layer. Figure 7 presents the load-displacement curves in the nanoindentation test of 300 M steel and carbonitrided samples. Comparing the test results of the two types of samples, we can see that the displacement offset of carbonitrided sample is smaller and the elastic modulus is larger, which indicates that its hardness and toughness are significantly improved. Moreover, the corresponding microhardness and elastic modulus were calculated by the Oliver-Pharr method. The microhardness of 300 M steel was 5.3 ± 1.2 Gpa, and that of the carbonitrided sample increased to 10.1 ± 1.6 Gpa; The elastic modulus of 300 M steel was 178 ± 17 Gpa, and that of the carbonitrided sample increased to 242 ± 21 Gpa.

**Tribological behaviour**

The 300 M steel substrates and carbonitrided coatings were tested at equal sliding distances to investigate the influence of loads on wear resistance. Figure 8 presents the specific coefficient of friction under 5 N and 15 N loads. The friction coefficient of 300 M steel is about 0.5 ~ 0.55, and the carbonitrided coating is about 0.1 ~ 0.15. It indicates that after carbonitriding the friction coefficient is significantly reduced, due to the improvement in the hardness of the diffusion layer, especially under the lubricating effect of graphite [33]. Generally, as the load increases, the contact area between the friction pair and the samples will increase, while the
friction coefficient will decrease. The carbonitriding coating has a relatively stable friction coefficient under both test conditions, and suggests well wear resistance due to high hardness and carburization.

Figure 9 represents the three-dimensional view of the abrasive traces of the substrates and the carbonitrided samples under different loading conditions. It observes that there is a 'furrow phenomenon' in the wear area of the substrates [34], whether it is 5 N or 15 N load. However, the wear marks of the treated samples are very blurry, even if the load is increased to 15 N. Figure 10 shows the two-dimensional contour curve corresponding to figure 9, which more directly describes the depth and width of the wear scars. During the tribological test, the depth and width of the wear scars of both kinds of samples grew as the load increased. In the same test, the depth and width of the wear marks of the carbonitrided samples were lower than those of the untreated ones. Under 15 N load conditions, the wear track width and depth of the untreated sample were about 650 μm and 10 μm,
respectively. However, the parameters of the corresponding carbonitrided samples were only 450 μm and 3 μm, respectively. It means that the 300 M steel carbonitrided by DG technique can maintain stable wear performance under high load.

**Wear mechanism**

Figure 11 depicts the SEM images of the wear tracks of 300 M steel substrate against by the ZrO₂ ball under different loads. Figures 11(b), (d) are enlarged microscopic images of the corresponding rectangular frames in figures 11(a), (c), respectively. In figures 11(a), (c), as the applied load increases, the wear track becomes wider and the wear debris increases, which accumulates in the furrows and the outer edge of the track. Under the test condition of 5 N load, large fatigue cracking and severe peeling can be observed on the worn surface in figure 11(b), which means that the wear mechanism is mainly abrasive wear [35]. When the load is increased to
15 N, although the wear profile is similar to 5 N, there are many sharp furrows, and some large pieces of wear debris can be seen in figure 11(d). Hence, the wear mechanism of 300 M steel is fatigue wear and adhesive wear.

Figure 12 displays the photomicrograph of the wear surface of 300 M steel and carbonitrided samples tested at 5 N and 15 N loads. As shown in figures 12(a), (c), with the test load increases, the depth and width of the wear scars increase significantly. The carbonitriding treatment improves the resistance against plastic deformation and fatigue wear by comparison with the 300 M steel substrate (see figure 11(a)). After 30 min of wear test, under the 5 N load condition (figure 12(a)), the wear scar boundary is blurred, only a slight wear scar is observed, and there is almost no damage. Figure 12(b) depicts that the 300 M steel had a white network structure on the surface after carbonitriding, and the wear test only smoothed part of the network structure, which indicates that slight abrasive wear occurred at 5 N load. Nevertheless, when the load was increased to 15 N, a little wear debris, large fatigue cracking and severe peeling were observed in the middle of the relatively smooth wear surface (see figure 12(d)). Therefore, the wear mechanism at this time was mainly fatigue wear. In addition, compared to figure 11(c), the wear scars boundary of carbonitrided sample under 15 N load was still very blurry and there is no deep furrow. In summary, thereby the carbonitriding treatment significantly improved the wear resistance of 300 M steel.

Figure 13 shows the SEM micrographs of typical wear profiles of ZrO2 balls on 300 M steel matrix and carbonitrided sample under 15 N load test conditions. Due to the interaction of forces, the wear of the grinding balls in figure 13 are consistent with the wear of the respective grinding samples. For the ZrO2 ball worn on 300 M steel substrates, the wear profile is clear and the diameter is about 700 μm, which is consistent with the wear profile of the 300 M steel substrate. Moreover, as the 300 M steel had low hardness, the ball surface left a lot of debris, some of which had been embedded into the ball. However, due to the high hardness of the carbonitrided layer and the lubrication of carbon atoms, the wear profile of the ZrO2 ball on the carbonitrided sample was more indistinct, with only a small amount of debris left on the profile edge [36]. The cause lies in the fact that fatigue wear and adhesive wear occurred in the 300 M steel under the 15 N load, while the carbonitrided sample only withstood fatigue wear.

Conclusions

(1) The carbonitrided diffusion layer was prepared on the 300 M steel substrate by DG alloying technique. The diffusion layer consisted of the compound layer (about 40 μm) and the interdiffusion layer (about 60 μm),
whose constitution was very compact and homogeneous. In addition, there is excellent cohesion property between the diffusion layer and the substrate, because the connection is metallurgical bonding.

(2) A large number of Fe4(C, N) and Fe3(C, N) phases in the compound layer significantly improve the hardness of the diffusion layer compared to the matrix. Moreover, the wear resistance of 300 M steel is also significantly improved, and the increase carbon content contributes to reducing the friction coefficient of the material.

(3) In all case, since the 300 M steel has a lower hardness and a larger coefficient of friction, it shows fatigue wear and adhesion wear in terms of wear mechanism. However, the carbonitrided coating exhibits excellent wear resistance, and its wear marks are blurred under both 5 N and 15 N load conditions. Apart from the lubrication of graphite, the wear mechanism is only fatigue wear.

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