



Study on the tribological properties of plasma nitrided bearing steel under lubrication with borate ester additive



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ABSTRACT

The nitrided layer on 52100 steel was prepared by plasma nitriding treatment. The tribological behaviors were examined under lubrication with borate ester containing nitrogen (B-N). The nitrided steel exhibited a lower friction coefficient and smaller wear scar diameter compared with the untreated steel. When 1.25 wt% B-N was used, the friction coefficient and wear scar diameter of nitrided steel were reduced by 34% and 45%, respectively. The main mechanism of this effect was attributed to the fact that the B-N additive can produce a tribofilm containing higher content of BN on the nitrided surface.

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1. Introduction

For wind energy equipments, severe working conditions, such as high loading, unsteady operation, system vibration, system misalignment and exposure to the extreme environment, lead to the reduced durability and efficiency of some key components [1,2]. Improving the reliability of the devices is important to lower the cost of wind energy. Plasma nitriding is a widely used chemical heat treatment in mechanical equipments. This method can produce a compact and high hardness nitrided layer on the steel surface with improved fatigue, wear and corrosion resistance of work pieces [3]. Plasma nitriding is an effective way of fulfilling the requirements of the materials used for key components in wind energy equipments. In addition, an appropriate selection of lubrication additives is also important to the wind energy devices facing very harsh working conditions [4].

However, the majority of the lubrication additives were studied for Fe based material without surface modification, which is inconsistent with the real application. Whether the additives for Fe based material are still effective for the modified surface is worth studying. Some previous works about the tribochemical interactions between nitrided steel surface and base oils, like alkyl naphthalene, ionic liquid and liquid paraffin, and some additives, have been conducted [5–7]. It showed that the nitriding treatment can make large contribution to improving the friction and wear behaviors effectively [8–10].

In order to accommodate the working condition of the wind energy equipment exposed in open-air, the lubricating oil should be environment-friendly. In other words, the additives should not contain S and P elements and other harmful components in the molecular formula. As a kind of potential eco-friendly lubrication additive, B and N containing additives are of significant interest because of the desired intrinsic properties, including environment-acceptability, absence of S and P in the molecular formula, good anti-wear and friction-reducing properties and good hydrolytic stability [11–13]. Zheng et al. [14] synthesized several kinds of N-containing borate esters as additives. They found that borate esters with a stable five- or six-member ring possessed good hydrolytic stability and friction-reducing ability, and exhibited good extreme pressure and anti-wear properties as well.

Greco et al. [15] found that the electrochemical boride surface and the nano-colloidal boron nitride lubricant additive showed an excellent synergistic effect in the wind turbine gearbox. Whether the nitrided surface lubricated with N-containing borate ester additives can also show such a good effect? In order to find the answer, a study on the tribological properties of plasma nitrided 52100 steel under lubrication with borate ester additives was carried out in this work. A new idea for improving the durability and efficiency of wind energy devices is proposed.

2. Experimental details

AISI 52100 steel balls (with a diameter of 12.7 mm, hardness of 770 HV, and surface roughness Ra 0.025 μm) were nitrided in a pulsed plasma nitriding furnace (LDM2-25). The treatment

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proceeded at a temperature of 520 °C with a voltage of 700 V for a total duration of 5 h. NH₃ is used as the source gas with a pressure of 670 Pa. Then the samples were cooled in vacuum to ambient temperature.

The friction and wear tests were carried out on an MS-10JR four-ball friction and a wear tester with a ball-to-ball contact configuration. The upper sample was a rotating ball fixed on the spindle, and the lower samples were three fixed balls. The four balls used in each test were selected as the same material. The friction and wear tests were conducted at a condition of load of 392 N (corresponding to the Hertz mean contact stress of 2.293 GPa), linear speed of 0.461 m/s, and test duration of 60 min. The minimum film thickness was determined using the Dowson and Hamrock minimum film thickness equation for an elastohydrodynamic point contact [16]. The lambda ratios (calculated minimum film thickness/initial composite surface root mean square roughness) for these tests were calculated as 0.812 and 0.13 for the untreated and nitrided surfaces, respectively. They are not the same but both are less than 1. Therefore, the lubrications on untreated and nitrided surfaces are in the boundary regime. The friction force and friction coefficient were measured by a force sensor and recorded by a computer. The wear scars were measured by a 15 J-type microscope. The tests were replicated at least three times. A good repeatability for the friction coefficients and wear scars in the whole process of the test was recorded and the results were averaged.

The base oil used was synthetic oil polyalphaolefin (PAO) whose kinematic viscosity was 16.7 mm² s⁻¹ at 40 °C. The main chemical composition of borate ester (B-N) is B 1.0 wt% and N 2.6 wt%. The density is 0.98 g cm⁻³ at room temperature. Two series of tests were carried out under lubrication of PAO with B-N additive. One was on the untreated surface, and another was on the nitrided surface. B-N's concentrations (mass fraction) in the tested oil were 0.0%, 0.25%, 0.5%, 0.75%, 1.0%, 1.5% and 2.0%.

The microhardness of the sample was measured by an MH-6 microhardness tester, and the applied load was 1.96 N with loading time of 5 s. The phase composition of the plasma nitrided layer was determined with a D-max/2550 X-ray diffractometer (XRD) in glancing angle geometry, using Cu K α radiation as the excitation source. After the wear test, the samples were ultrasonically washed with petroleum ether and alcohol (the volume ratio 3:1). A JSM-6460LV scanning electron microscope (SEM) was utilized to observe the wear scar morphologies of the balls. The chemical states of tribofilms were investigated using a PHI Quantera X-ray photoelectron spectroscopy (XPS). The instrument employed a high-power rotating anode and monochromatised Al K α X-ray source. The tested sample surface was sputtered about 5 nm in depth by Ar ion (sputtering depth on the standard SiO₂ sample). The binding energy of 284.6 eV for contaminated C was used as a reference for charge correction.

3. Results and discussion

3.1. Characterization

Fig. 1 shows the SEM images of the untreated surface and the nitrided surface. It can be clearly seen that the spherical particles were piled up closely with a lot of fine micro-pores distributed on the nitrided surface (Fig. 1b), which resulted in a higher roughness Ra 0.16 μ m. However, none was found on the untreated surface (Fig. 1a).

Fig. 2 shows the variation of the hardness of nitrided layer with the depth. The hardness of nitrided samples increased to 900–1000 HV with a distance of about 40 μ m from the surface, and then decreased gradually to 500–600 HV when the distance from

the surface reaches 40–140 μ m. When the distance from the surface is larger than 140 μ m, the hardness remained at a lower value.

From Fig. 3, it was found that the compositions of the nitrided sample were mainly γ' -Fe₄N, ϵ -Fe₂₋₃N and CrN, while those of the untreated sample were martensite and austenite.

3.2. Friction and wear behavior

Fig. 4 shows the variations of friction coefficients of the untreated and nitrided surfaces with different concentrations of B-N. It indicated that the friction coefficients of the nitrided surface are always lower than those of the untreated surface under the same lubrication condition. The variation of the friction coefficients along with the increase of additive contents is irregular. Only when lubricated with 1.0 wt% and 1.25 wt% B-N, the friction coefficients of nitrided surface presented a sharp decrease to about 0.07, much lower than other concentrations. It may attribute to a friction reducing tribofilm formed on the nitrided surface lubricated with the proper B-N content.

Fig. 5 shows the variation of wear scar diameters with different concentrations of B-N. It can be seen that wear scar diameters on both surfaces were almost the same on untreated and nitrided surfaces in the lubrication of PAO. This results from the fact that the hardness of mating pairs was the same in each test [17]. The wear scar diameters of the untreated surface were almost unchanged for all concentrations, while those of nitrided surface were gradually reduced with the increase of B-N percentage. The minimum wear scar diameter reached 0.4 mm at the concentration of 1.25%, 45% less than that on the untreated surface. When the concentrations of the B-N continue to rise, the wear scar diameter on the nitrided surface increased again, but was still smaller than that on the substrate surface. It can be considered that there existed certainly a beneficial synergistic effect between the nitrided 52100 steel surfaces and B-N additive, which led to the improvement of wear-resistance through forming a protective tribofilm.

3.3. SEM analysis

In order to understand the difference of the friction-reducing and anti-wear mechanism of the B-N additives on the nitrided and the untreated surfaces, the wear surfaces were analyzed by SEM. Fig. 6 shows the SEM morphologies of the worn surfaces lubricated with PAO and 1.25 wt% B-N. So many deep furrows can be found on the surfaces of untreated and nitrided samples under the lubrication of PAO. It is obvious that the nitrided surface shows a smoother surface than the untreated ones under the same lubrication conditions. The furrows become obviously shallower as the addition of the additives. Compared with some corrosive pitting on the untreated surface lubricated with 1.25% B-N, the nitrided surface lubricated with 1.25% B-N shows the smoothest wear surface. This might be attributed to the excellent synergistic effect of borate ester and a higher hardness of the nitrided surface.

3.4. XPS analysis

The wear surfaces of the nitrided 52100 steel lubricated with different lubricants were analyzed by XPS so as to acquire more information about the tribochemical reaction involved during the sliding process, as shown in Fig. 7. Table 1 exhibits the elemental composition, determined by XPS quantitative analysis, of the tribofilm on the wear surface lubricated with 1.25% B-N. The binding energy values and quantifications of all the elements on the surface and the main components of the reference compounds [18] are listed in Table 2. It can be found that 9.45% B and 1.80%

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