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Possibilities of increasing wear resistance of steel surface by plasma electrolytic treatment

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ABSTRACT

The paper considers the effect of the anode plasma electrolytic treatment (PET) on tribological properties of steels after their boriding (PEB), nitriding (PEN), and nitrocarburising (PEN/C). Electrolyte compositions and processing temperatures improved wear resistance of samples under dry friction condition using a ball-on-disk test with an Al_2O_3 ball as counter-body are determined. The anode PEB of medium carbon steel in solution of boric acid and ammonium chloride (850 °C, 5 min) followed by quenching in electrolyte enables to decrease friction coefficient, surface roughness 3 by times, and volume loss by 3.7 times. This treatment results in the change of wear mechanism from abrasive-adhesive wear to polishing mode. The PEN of medium carbon steel in a solution containing ammonia and ammonium chloride (650 °C, 5 min) followed by quenching in electrolyte leads to decrease in friction coefficient from 0.65 to 0.45, surface roughness by a factor of 1.74, and volume loss by 4.6 times. PEN/C of low-carbon steel in electrolyte containing glycerol, ammonium chloride and ammonium nitride at 950 °C also promotes surface roughness and wear rate reduction of by 9.5 times.

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1. Introduction

Case hardening of steel parts occupies an important place among the methods of increasing their wear resistance. Technologies of diffusion saturation of steel include PET which enables to decrease the processing time to several minutes and does not require expensive equipment or toxic components. Effective application of plasma electrolytic saturation of steels with nitrogen, carbon or boron for enhancement of their wear resistance is presented in many publications. Cathode PEN in a solution of carbamide at 400–600 °C for 3–10 minutes diminishes wear rate of cast iron G3500 by 2.5 times and steel S0050A by 3times under dry friction with WC balls as counter-bodies (5 N normal load, 0.1 m/s sliding speed, and 200 m sliding distance) [1]. However, electrical discharges inherent to cathode treatment lead to friction coefficient increase from 0.37 to 0.4 for steel S0050A and from 0.14 to 0.4 for cast iron G3500. Similar results were obtained for PEN of high speed steel R6M5 [2] or structural steel 34CrNi1Mo [3] where abrasive wear resistance decreased by a factor of 1.5.

The cathodic PEC of pure iron in glycerol-based electrolyte decreases wear rate by one order but the friction coefficient increases [4]. Pulse PEC of high-carbon steel T8 in glycerol-based

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http://dx.doi.org/10.1016/j.wear.2016.12.053 0043-1648/© 2017 Elsevier B.V. All rights reserved. electrolyte also results in the formation of diamond-like carbon layer which provides an increase in the wear rate by 5 times and reduces friction coefficient by 2 times approximately [5]. Maximal values of microhardness and wear resistance of low-allow steel H13 after its PEC in glycerol electrolyte were obtained at frequency of 10 kHz [6]. The wear resistance of steels can be enhanced by means of PEN/C, as well. In particular, the steel treatment in carbamide-based electrolytes reduces the weight wear of low carbon steel 1020 by an order of magnitude approximately [7], Q235 [8] or stainless steel 316 L [9]. Moreover, PEN/C of steel 316 L enhances its fatigue strength [10]. Wear resistance of cast iron can also be increased using PEN/C in electrolyte containing acetamide and glycerol [11]. Additional increase in wear resistance of AISI 304 stainless steel can be obtained using a diamond-like carbon coating on the PEN/C pre-treated substrate [12]. This treatment results in a simultaneous reduction of the friction coefficient and wear rate due to changes in the wear mechanism from adhesion/ abrasion to asperity deformation and polishing. It is established that this coating does not get damaged under 10-25 N loads against different counter-bodies [13].

Positive results were obtained for PEB of steels. Weight wear rate of steel H13 after its treatment in borax-based electrolyte (969 °C, 10 min) decreased by 13 times in comparison with untreated samples [14]. Anode PEB of medium carbon steel in electrolyte containing borax also results in diminishment of friction coefficient from 0.26 to 0.16 and weight wear rate by a factor of 7 during dry wear testing against hardened steel disk (HRC 45–50)





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[15]. Some better results are obtained with anodic PEB of medium carbon steel in a solution of boric acid and ammonium chloride (900 °C, 5 min) where surface roughness decreased by 3 times, friction coefficient from 0.85 to 0.15 and wear rate was reduced by a factor of 15 [16].

Simultaneous saturation of steels with boron and other elements is investigated in some studies. Borocarburising of lowcarbon steel Q235 in a solution of borax and glycerol provides weight wear decrease by 12 times in comparison with raw sample after dry wear testing against ZrO₂ ball [17]. In this case, friction coefficient reduces from 0.6 to 0.17 in spite of a rise of surface roughness by an order. Replacing glycerol with other organic substance can further improve wear resistance of steel Q235 by a factor of 19 under the same test conditions [18]. Simultaneous saturation of H13 steel with boron and nitrogen in electrolyte containing borax and sodium nitrite provides growth of wear resistance by more than 17 times in comparison with untreated steel [19].

A number of authors note that cathode treatment increases surface roughness of steels [7–9 and etc.]. The anode processes are characterized by a decrease in surface roughness of steel due to anode dissolution of samples which enables to reduce friction coefficient and wear rate. The anode PEN of steel 40Cr in a solution of ammonia and ammonium chloride (750 °C, 5 min) leads to diminishment of friction coefficient from 0.42 to 0.34 and wear rate by 15 times for dry wear testing with pin of sintered TiC as a counter-body [20]. Anode PEN/C in carbamide-based electrolyte provides decrease in surface roughness of low-carbon steel by 9 times and wear rate by 6.7 times during lubricant wear testing against hardened steel disk (HRC 50) as a counter-body with normal load of 315 N, sliding speed of 0.47 m/s, and 1000 m sliding distance [21]. Finally, we present an example of anode saturation of mild steel with boron, nitrogen and carbon in electrolyte containing boric acid, carbamide and ammonium chloride [22]. In this case, surface roughness diminishes from 1.0 µm to 0.8 µm, dry friction coefficient from 0.16 to 0.11, and weight loss during testing from 6.8 mg to 3.8 mg at sliding distance of 1000 m.

Analysis of publications shows the prospects for plasma electrolytic processes to improve wear resistance of steel parts. However, these positive results were obtained under various and fixed conditions of wear testing. Morphology of friction tracks and the mechanism of wear have not been the focus of most studies. The aim of this work is to study the effect of temperature saturation of steels with interstitial elements on the coefficient of friction and wear rate of the steel samples treated in different ways. Results of the anode processes of PEB, PEN, and PEN/C of low-carbon and medium carbon steels including the structure of the modified layers, their phase composition, microhardness, surface roughness and morphology of the worn surface will be considered.

2. Materials and methods

2.1. Materials and characteristics of treatment

Cylindrical samples (\emptyset 10 × 15 mm) of low-carbon 20 and medium carbon 45 steels (Table 1) were ground with SiC abrasive

Table 1Chemical composition of the samples (wt. %).

| Grade | С | Mn | Si | Р | S | Cr | Ni | Cu | As |
|----------|--------------|--------------|----|---|----------------|----|----|----|--------------|
| 20 45 | 0.20 0.44 | 0.38 0.59 | | | 0.013 0.017 | | | | 0.01 0.01 |

paper grit size P320 to Ra ~1.0 µm and ultrasonically cleaned with acetone. Plasma electrolytic saturation was carried out in a cylindrical electrolyzer (volume of 1.5 l) with an axially symmetric electrolyte flow supplied through a nozzle located at the bottom of the electrolyzer [23]. In the upper part of the electrolyzer, the electrolyte was overflowing into the sump and was further pumped through a heat exchanger at a rate of 2.6 l/min which was measured with a RMF-0.16 GUZ flow meter (accuracy of \pm 2.5%). This scheme provides stabilization of processing conditions. The solution temperature was measured using thermocouple placed at the bottom of the chamber. The electrolyte temperature was maintained at 30 \pm 2 °C. The samples were connected as positive output and the electrolyzer was connected as negative output of the 15 kW DC power supply. Voltage and current were measured using DP6-DV voltmeter and DP6-DA ampermeter (\pm 0.5%).

After switching the voltage of 200 V, the samples were immersed in the electrolyte at speed of 1–2 mm/s. At slow immersion vapour-gaseous envelope is easily formed in the samples initial area adjoining electrolyte, and further extends across the sample as it submerges. As far as a sample is immersed at a depth equal to its height, voltage is corrected for reaching prescribed sample's temperature (850–950 °C).

The sample temperature was measured with MY K-type thermocouple and multimeter APPA109N (accuracy to 3% over a temperature range of 400–1000 °C). Thermocouple fixed in a hole made in the samples at a distance of 2 mm from the sample bottom. The treatment time was 5 min. The treatment temperature varied from 650 to 950 °C. After PET samples were quenched in the electrolyte (hardening).

Aqueous solutions of boric acid H_3BO_3 (3 wt.%) with ammonium chloride NH_4Cl (10 wt.%), ammonia NH_3 (5 wt.%) with ammonium chloride NH_4Cl (10 wt.%), and glycerol (8 wt.%) with ammonium chloride NH_4Cl (15 wt.%) and ammonium nitrate NH_4NO_3 (5 wt.%) were used as the working electrolytes. This is a cost-effective, ecologically friendly and non-hazardous components compared to the organic, highly flammable and toxic electrolytes.

2.2. Surface characterization

Microstructural studies of the surface layers were performed using conventional techniques. Quanta 3D 200i scanning electron microscopy (SEM) (FEI Company) with backscattering electron detector (working distance was 15.2 mm and acceleration voltage was 10 kV) was used to observe the structure of the surface layer of the samples after polishing and etching with the use of a 4 wt. % nitric acid solution in ethanol for 5–10 s. The phase composition of the surface layers after PET was investigated with the use of an ARL X'tra x-ray diffractometer (Thermo Fisher Scientific) with Cu K α radiation at a simple scanning in the theta-2theta-mode and scanning rate of 2°/min.

Microhardness of the samples' surface layer after PET was measured on a PMT-3M apparatus at a loading of 50 g (4 measurements).

Surface roughness before and after PET was investigated using a roughness tester TR200 (5 measurements).

A ball-on-disk tribometer was applied to evaluate wear resistance of the untreated and treated samples under dry testing conditions with 10 N normal load, 0.2 m/s sliding speed, and 240 m sliding distance (diameter of track is 9 mm) with an Al_2O_3 ball (6.35 mm in diameter) as counter-body. The typical morphologies of wear tracks were analyzed with an optical microscope. The profiles of wear tracks were investigated using a roughness tester TR200. The volume loss was tested using 5 measurements. To enhance reproducibility of experimental data (before wear tests) the friable part of the oxide layer was removed

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