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Tribological characteristics of samples made from titanium alloy VT5 nitrided in plasma glow discharge

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Abstract
The effect of nitriding of VT5 alloy (3.7115) in the plasma of a glow discharge excited in a hollow cathode on its tribological characteristics in pairs with different materials in jet fuel TS-1 and under dry sliding conditions has been studied. For research and tribological tests, samples of VT5 alloy were nitrided in the plasma of a glow discharge excited in a hollow cathode at a temperature of 975 ± 15 °C and a pressure of 58 ± 2 Pa in a nitrogen-argon gas mixture with a nitrogen content of 78 vol. %. Tribological tests were performed on a friction machine 2070 SMT-1 according to the scheme "cube-roller". Tests under conditions of sliding with jet fuel TS-1 were performed at a load of 200 N. The sliding speed during the tests was 1.3 m/s, and the test time was 75 s. The test under dry sliding conditions was carried out at a load of 100 N and a sliding speed of 0.785 m/s. The test time was 30 minutes. It is shown that the nitrided VT5 alloy in friction pairs with several materials has rather low wear values ($\leq 2.26 \times 10^{-6}$ mm^3/Nm), as well as a coefficient of friction at the level of 0.08 to 0.12.

1. Introduction

Titanium alloys have a number of certain properties, such as high specific strength, fatigue properties, and corrosion resistance, which determine the expansion of their use in various fields of application, in particular, in the production of aerospace equipment [1,2]. The introduction of titanium and titanium-based alloys is one of the urgent tasks in aircraft engineering. However, the tendency to grip during sliding and other low tribological properties limit the possibility of their wider use in aerospace units and assemblies. The application of titanium alloys can be significantly expanded if their surface is modified by different methods of processing or coating, increasing their wear resistance, antifriction and other properties [3-6]. The demand for the application of titanium alloys and alloys on its base in friction units and equipment is caused both by the general tendency to decrease the weight of aerospace products and by the need to replace aluminium-based alloys if it is necessary to ensure the operation of corresponding units at temperatures above 130 °C [1]. Among the enterprises working in this direction is, in particular, FED JSC (Ukraine) [7]. When determining the possibility of using a particular titanium alloy, first of all, of course, its physical and mechanical properties are taken into account, but its price and availability are also important. In this respect, low-alloyed, with average strength level, single-phase α-alloy of titanium VT5, the close analogue of which is the titanium alloy 3.7115 (Grade 6), could be considered as quite suitable for use in friction pairs with relatively low load level and operating temperatures up to 400 °C after modifying its surface by methods used to improve the tribological properties of titanium-based alloys. In addition, it is a single-phase α-alloy and, unlike the stronger and higher-temperature two-phase α + β titanium alloys, it has such important properties as thermal ageing resistance, low creep, and stability of mechanical characteristics during long-term operation [8].

One of the modern methods to improve the performance characteristics of titanium and titanium-based alloys under sliding conditions is the method of ion-plasma nitriding [9-11]. Thus, work [9] shows that after the ionic nitriding of VT6 alloy, the microhardness of the surface increases by 3 to 4 times compared to the initial state, depending on the nitriding conditions. Measurements of wear of the samples nitrided in different modes carried out on the Nanovea tribometer according to the scheme "ball-on-disc" at a load of 4 N showed that its value can be improved four times, that is, it depends significantly on the conditions of nitriding the samples. In work [10] the tribological characteristics of the titanium alloy (Ti-6Al-4V), which is an analogue of the domestic alloy VT6, were studied. The wear value and coefficient of friction were measured with a CSM tribometer in the unidirectional sliding mode with a Si_3N_4 ball at a load of 3 N. The studies showed that the coefficient of friction could have a value from 0.6 to 1.0, depending on the mode of ion nitriding and the presence of texture of the sample surface layer, as well as the wear value, which was in the range from 0.025 to 0.3 mm^3. In [11] the tribological studies of the VT8 titanium alloy nitrided in different modes were conducted on a universal friction machine UMF 2168 under the "disc-finger" (pin-on-disc wear test) scheme in the conditions of dry sliding with a loading of about 20 N. The counter-body was hardened steel 45 (HRC 45). It was found that the coefficient of friction of nitrided titanium samples at such test conditions decreased by 2 to 3 times compared to the test of non-nitrided samples and could range from 0.2 to 0.4, depending on the nitriding modes, and the wear rate decreased by 70 and 40 times, respectively.

The results of the effect of ion-plasma nitriding on the tribological characteristics of titanium and its alloys when working with those or other materials, which are given above, indicate the high efficiency of such treatment to improve the tribological characteristics of titanium-based alloys, although they may differ significantly even for identical materials of friction pairs due to the mismatch of conditions of their manufacture, methods and conditions of an investigation of their characteristics. Therefore, it is possible to correctly compare the tribological characteristics of friction pairs made of nitrided titanium-based alloys with different materials based on literature data only if the conditions of their manufacture, nitriding, methods and conditions of testing are the same. Finding information in the literature about the possibility of using the alloy VT5 in friction pairs with metallic materials and its tribological characteristics after modifying its surface by ion-plasma nitriding was not possible. These circumstances prompted the authors of this work to conduct a study to find out the effect of plasma nitriding of the VT5 alloy on its tribological characteristics in friction pairs with different materials.

The aim of this research is to study the effect of nitriding of the VT5 alloy in glow discharge plasma excited in a hollow cathode on its tribological characteristics in pairs with various materials under dry sliding conditions and under jet fuel TS-1 lubricated conditions.

2. Equipment, research methods and materials

For research and testing, samples were made of VT5 alloy (table 1) in the form of cubes with a side of 10 mm and a roughness of *Ra* = 0.63 µm. Before making the samples, the alloy was annealed at 850 °C for one hour in a vacuum furnace at residual gas pressure ≤ 1.3×10^{-2} Pa. Nitriding of the samples was carried out in glow discharge plasma excited in a hollow cathode in the form of a cube with dimensions of 100 × 100 × 100 mm at a temperature of 975 ± 15 °C and pressure of 58 ± 2 Pa for 4 hours in a nitrogen-argon gas mixture containing nitrogen. Figure 1 shows a photo of a glow discharge in a hollow cathode during sample nitriding.

The temperature of the samples during nitriding was controlled with a Promin pyrometer, the composition of the gas mixture was set using FG201 flowmeters from Bronkhorst, and the pressure was measured with a KPDR900 vacuum gauge from Kurt J. Lesker. The hardness of the nitrided surface of the samples was determined using a Qness 60M hardness tester manufactured by Chemika at a load of 5 kg. The roughness of the samples was determined using a Jenoptik nanoscan 855

Table 1. Chemical composition of VT5 alloy (wt. %)

C	Si	Fe	N	Al	V	Ti	Mo	O	Zr	H
≤ 0.1	≤ 0.12	≤ 0.3	≤ 0.05	4.5 – 6.2	≤ 1.2	90.63 – 95.2	≤ 0.8	≤ 0.2	≤ 0.3	≤ 0.01

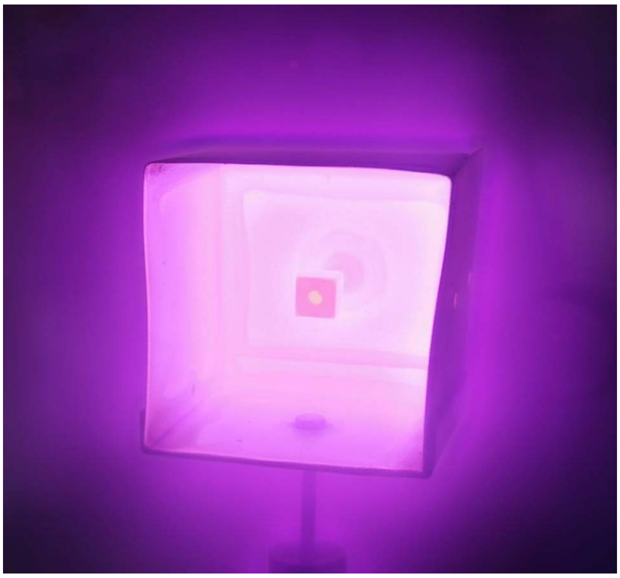

Figure 1. Photograph of a glow discharge in a hollow cathode during nitriding of a sample located in the centre of the cathode

profilometer. The surface topography before and after tribological tests was studied using an Altami MET-1C optical microscope with a digital camera.

Tribological tests were performed on a modernised 2070 SMT-1 friction machine according to the "cube-roller" scheme (Fig. 2) under conditions of dry sliding and sliding with lubrication.

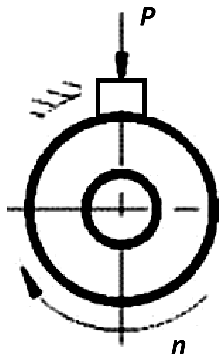

Figure 2. Tribological test scheme of "cube-roller" configuration

Lubrication was carried out by immersing the movable counter-body (roller) in a bath with jet fuel TS-1 (GOST 10227-86). Cubic specimens were used as a fixed specimen "shoe". The "shoe" specimen during the tests was fixed in a special mandrel with the possibility of "self-installation" on prismatic supports. This made misalignments during loading impossible and allowed (taking into account the shape of the specimens) to minimise the scatter of data during testing. The movable specimen "roller" was used as a standard sample with a diameter of 50 mm and a height of 12 mm, made of various materials that can be used in various branches of mechanical engineering and aircraft building (steel 1.4878, steel 1.3505, cemented steel 20Cr3MoWV, nitrided steel 20Cr3MoWV, cast iron GGG80, bronze Br.O10C2N3 (wt. %) (Sn 9-11; Pb 2-3.25; Ni 3-4), VB-23NTS (wt. %) (Pb 18-22; Ni 3-4; Zn 3-4; Sb 3-4; P 0.15-0.3), VB-24 (wt. %) (Sb 4.7-6.2; P 0.4-0.9). The conditions of tribological tests are given in Table 2.

Table 2. Conditions for conducting tribological tests

Load, N	Lubricant	Sliding speed, m/s	Test time, s
200	with	1.3	75
100	without	0.785	1800

The test time was determined under the conditions that the depth of wear tracks did not exceed the thickness of the hardened layer (≤ 30 μm) of specimens made of VT5 alloy. The value of the coefficient of friction was determined continuously throughout the test. The amount of wear was determined by the weight method. Samples were weighed before and after tests (after thorough washing) on analytical scales with an accuracy of 1×10^{-4} g. Average arithmetic values of the test results of each mating material were calculated from the data of 2 parallel experiments. Instrumental errors in the determination of coefficients of friction at tests with 100 N overloading were 2 – 10 %, while at tests with 200 N overloading the errors were 1.85 – 12 %. The smallest values of percent errors refer to large values of coefficients of friction. When mass wear was converted to volumetric wear, the density data for the corresponding materials were used.

3. Results and discussion

Studies of specimens made from the VT5 alloy after nitriding showed that the surface roughness *Ra* was in the range of 0.58 – 0.64 μm, and the hardness of the surface (HV5) was 525 ± 20 kgf/mm² with a base hardness of 325 ± 15 kgf/mm². Figure 3 shows the structure of the cross-section of the VT5 alloy sample nitrided layer, etched in a mixture of hydrofluoric and nitric acids. In the structure of the nitrided layer, there is a thin, 3 – 4 μm thick, dark outer layer, then a light uniform continuous layer 25 – 30 μm thick, behind which there is an intermediate layer between the base and the solid light layer. The intermediate layer has a mixed structure of light-coloured fragments, similar to a continuous layer,

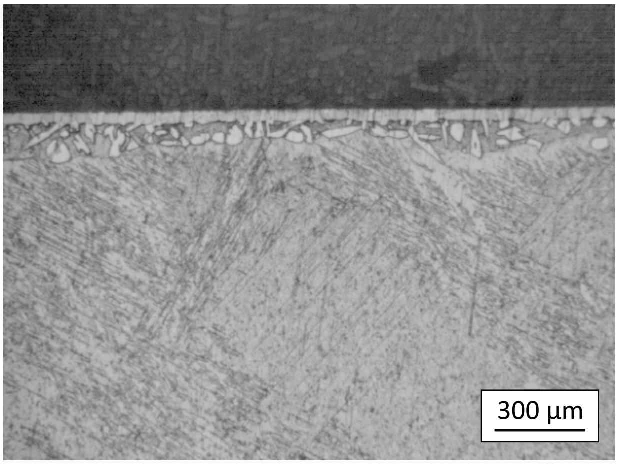

Figure 3. Structure of the cross-section of the VT5 alloy sample nitrided layer etched in a mixture of hydrofluoric and nitric acid

interspersed in a field with a base structure. Such a three-layer structure of the surface nitrided layer is formed during the nitriding of titanium and α-alloys in a certain temperature range above the temperature of the α → β phase transition, which for the VT5 alloy, according to various literature data [12,13], is in the temperature range of 940 – 990 °C. The outer layer is titanium nitride TiN_x, then the layer in which the transition from the β phase to the α-phase occurred due to its saturation with nitrogen to a concentration sufficient for such a transition at the nitriding temperature. In the intermediate layer, only in some fragments of its structure, a nitrogen concentration was reached that was sufficient for the formation of the α-phase at the nitriding temperature in the β-phase layer. On cooling, these fragments, having the same colour as the overlying layer, did not experience a polymorphic transformation, while the base and the rest of the intermediate layer experienced a β → α transformation on cooling. The core structure of the nitrided sample did not change compared to the alloy structure it had after annealing at 850 °C.

The results of tests of nitrided samples in contact with different materials under lubricated sliding conditions are presented in Table 3. They can be conditionally divided into two groups. One group of materials is friction pairs that have shown satisfactory results in terms of wear and coefficient of friction for these test conditions, which include all types of tested bronzes, cast iron GGG80, cemented steel 20Cr3MoWV and steel 1.3505. Another group of tested materials in friction pairs, which included nitrided steel 20Cr3MoWV and steel 1.4878, had high values of both the coefficient of friction and wear of the contacting materials.

The first group of materials had a low coefficient of friction and induced minimum wear of the nitrided titanium. Nitrided titanium sample in contact with cemented steel 20Cr3MoWV even shows an increase in mass after testing. Wear tracks with this group of materials are hardly noticeable on the surface of nitrided samples during the visual inspection and practically did not change the surface structure compared to the original one (Fig. 4).

The second group of materials tested in friction pairs, which included nitrided steel 20Cr3MoWV and steel 1.4878, had high values of both the coefficient of friction and wear of the contacting materials. A nitrided steel 20Cr3MoWV showed higher wear and also caused higher wear of nitrided titanium. The coefficient of friction of this group of materials turned out to be the same and had a value of 0.54. Figure 5 shows a view of the wear track on a nitrided titanium sample paired with nitrided steel 20Cr3MoWV, after a tribological test. The track has contours with torn edges, and its surface is a rough

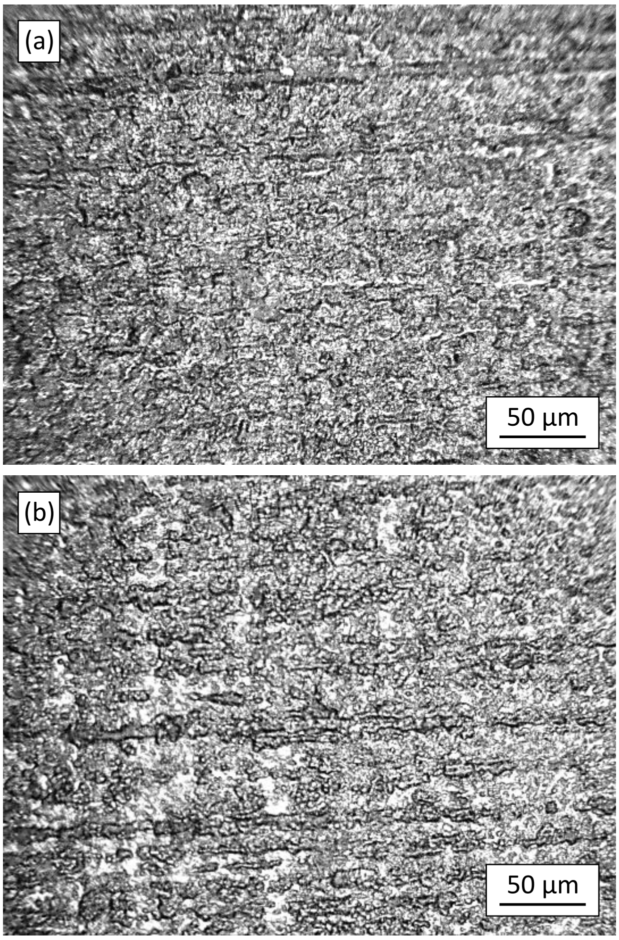

Figure 4. Structure of the surface of a nitrided sample in a pair with cemented steel 20Cr3MoWV lubricated with jet fuel TS-1: (a) before the tribological test and (b) after the tribological test

Table 3. Test results of friction and wear for pairs lubricated with jet fuel TS-1

No.	Roller material and hardness (HV), kgf/mm^2	Cube wear, g	Roller wear, g	Coefficient of friction	Cube wear rate, mm^3/Nm	Roller wear rate, mm^3/Nm
1	bronze VB-23NTS	0.0000	0.0030	0.08	$< 1.13 \times 10^{-6}$	1.81×10^{-5}
2	bronze Br.O10C2N3	0.0001	0.0010	0.10	1.13×10^{-6}	6.25×10^{-6}
3	bronze VB-24	0.0000	0.0002	0.10	$< 1.13 \times 10^{-6}$	1.21×10^{-6}
4	cast iron GGG80; 335 HV	0.0000	0.0016	0.11	$< 1.13 \times 10^{-6}$	1.14×10^{-5}
5	steel 1.3505; 675 HV	0.0002	0.0011	0.10	2.26×10^{-6}	7.22×10^{-6}
6	cemented steel 20Cr3MoWV; 740 HV	– 0.0002	0.0003	0.12	-2.26×10^{-6}	1.97×10^{-6}
7	nitrided steel 20Cr3MoWV; 830 HV	0.0020	0.0090	0.54	2.26×10^{-5}	5.92×10^{-5}
8	steel 1.4878	0.0014	0.0031	0.54	1.58×10^{-5}	2.42×10^{-5}

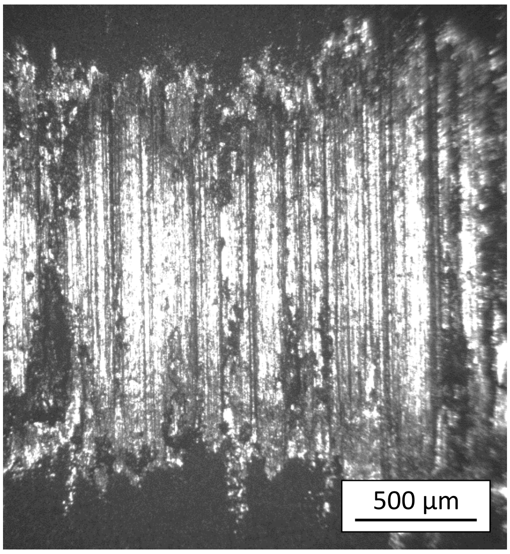

Figure 5. Wear track on a nitrided titanium sample paired with nitrided steel 20Cr3MoWV lubricated with jet fuel TS-1 after tribological test

striped relief with differences in the depth of valleys of tens of microns (Fig. 6) and a maximum average track depth of approximately 40 µm.

The wear track paired with steel 1.4878 has a similar appearance after tribological tests, at a somewhat shallower depth. With such a depth of the wear track, wear in the area of contact of the nitrided sample with the counter-body must be completely subjected to the upper layer of titanium nitride, the continuous nitrided layer and partially the intermediate layer between the nitrided layer and the base material of the titanium alloy, as shown in Figure 1.

Figure 7 shows the structure of cross-sections of titanium samples with a nitrided layer in the area of wear tracks in contact with nitrided steel 20Cr3MoWV and steel 1.4878. It can be seen from Figure 7 that wear of the titanium sample material occurs in the region of the intermediate nitrided layer, not reaching the base material. Deformation distortions in the structure of the intermediate layer, caused by the influence of the friction force between the rotating roller and the surface of the test sample, are also clearly visible.

Sufficiently good tribological properties of cast irons in friction pairs with various materials are primarily associated with a high content of carbon in them, which, upon contact with rubbing surfaces, participates in the formation of surface films that provide sliding with a low coefficient of friction. From these positions, it is possible to explain why the results of tribological tests with nitrided and cemented steel 20Cr3MoWV differ so much. According to [14] the surface concentration of carbon, depending on the carburising modes, can range from 2.14 to 5.7 wt. %, but at a distance of 0.2 mm from the surface the carbon concentration can be from 0.95 to 1.45 wt. %.

At the operation of different friction pairs lubricated with grease in conditions of limiting friction, the coefficient of friction has a value at level 0.08 – 0.15, occupying intermediate values between values, characteristic for liquid sliding conditions and conditions of dry sliding. Exactly in this range, there is a value of the coefficient of friction for the first group of friction pairs. In the second group of friction pairs, the obtained value for the coefficient of friction indicates a significant contribution of adhesive interaction between materials, despite the presence of a lubricating liquid, which leads to the seizure and formation of scuffs in some parts of the contacting surfaces. This is evidenced by the type of wear tracks of these groups of materials.

In order to predict the behaviour of units and mechanisms under conditions of insufficient lubrication or interruptions in its supply, it is necessary to know the characteristics of the selected tribo-pair also in dry sliding conditions.

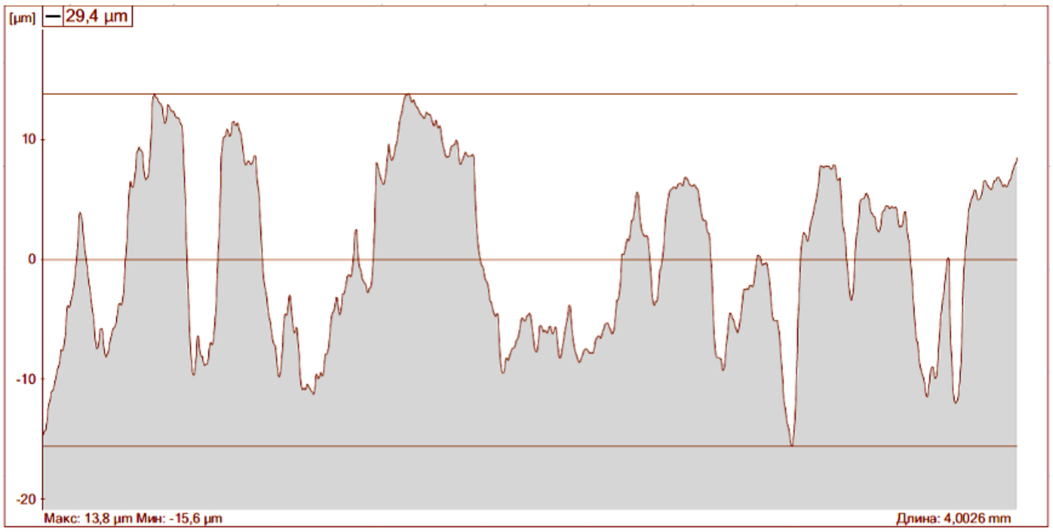

Figure 6. Profilogram in the region of the maximum depth of the wear track of a nitrided titanium sample in a pair with nitrided steel 20Cr3MoWV lubricated with jet fuel TS-1 after tribological test

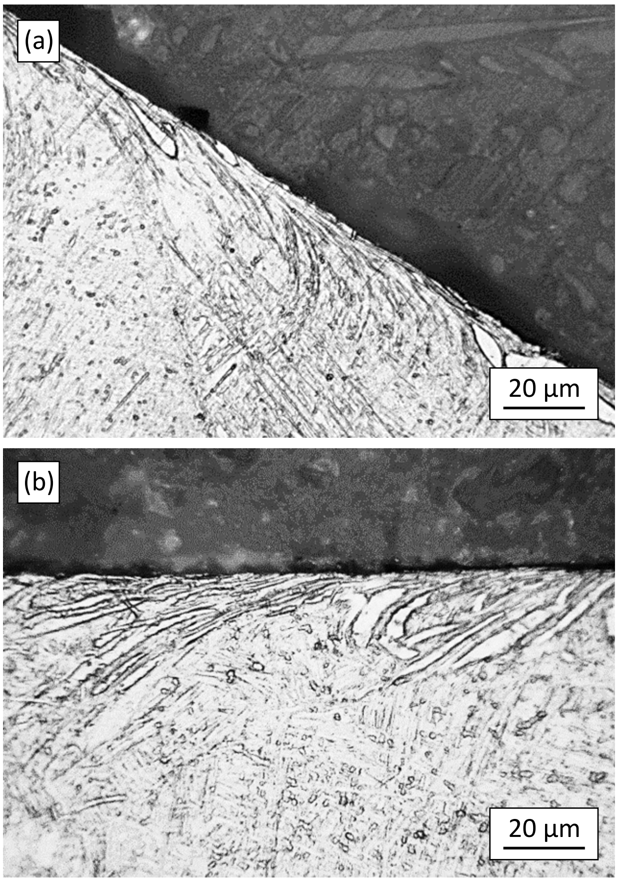

Figure 7. Structure of the cross-section of the nitrided titanium sample in the region of the maximum depth of the wear track after tribological tests paired with: (a) nitrided steel 20Cr3MoWV and (b) steel 1.4878

Besides, for many friction units, the use of lubricating oils according to the conditions of their operation is simply unacceptable. Cemented steel 20Cr3MoWV is widely used in the production of various units. Therefore, it was interesting to determine the tribological characteristics of nitrided alloy VT5 when working under dry sliding conditions, which showed an increase in cube weight rather than wear, when tested with lubrication in pair with this material. Given the non-lubricated friction pair operating conditions used in the units produced, the tests were conducted at a load of 100 N. The tests showed that during the first 75 seconds of testing, the coefficient of friction had an almost constant value of 0.34. After this period the coefficient of friction became inconsistent and lower, with values fluctuating first from 0.32 to 0.2 and then increasing until the end of the test to values from 0.26 to 0.52. Table 4 shows the results of measuring the wear of the studied samples in this experiment.

After testing the friction pair nitrided alloy VT5-cemented steel 20Cr3MoWV without lubricant, the shoe (cubic specimens) gain weight, as well as after testing this pair with lubricant. After testing this pair with lubrication the surface of the nitrided titanium alloy in the area of contact with the counter-body did not undergo significant changes, but in this case, a wear track was formed, which as measurements showed had a depth of about 13 µm. With such a track depth from the counter-body with a diameter of 50 mm, the calculated weight loss should have been at least 0.0006 g. The discrepancy between the calculated change in the block weight and the measured one indicates that during the sliding of this pair, the material is transferred from the roller. Figure 8 shows a photo of the wear track on a nitrided titanium sample, with material enveloping on the edge of the track and numerous areas with the products of interaction of contact materials.

Table 4. The results of the test without oil for the friction pair of nitrided alloy VT5-cemented steel 20Cr3MoWV

Distance, m	Cube wear, g	Roller wear, g	Coefficient of friction	Cube wear rate, mm^3/Nm	Roller wear rate, mm^3/Nm
1413	– 0.0005	0.0046	0.2 – 0.52	-6.51×10^{-7}	4.46×10^{-6}

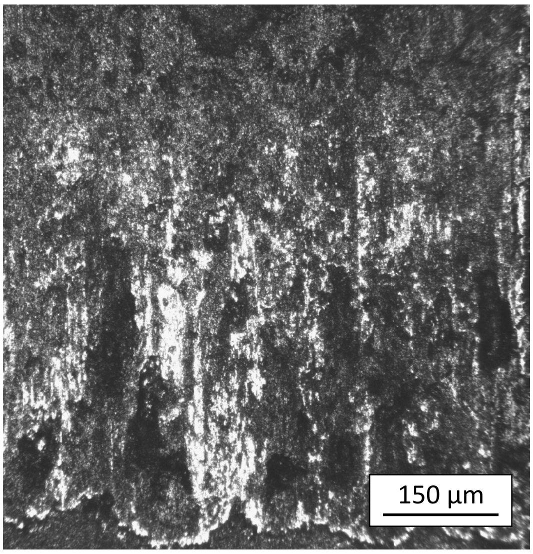

Figure 8. Wear track on a nitrided titanium sample paired with cemented steel 20Cr3MoWV without lubrication after the tribological test

The change in the value of the coefficient of friction during testing can be explained by a change in the composition of the contacting material of the cube during its wear. The initial period corresponds to the time during which the sliding was carried out over the titanium nitride layer, during which the adhesive interaction with the roller material was constant and, therefore, the coefficient of friction did not change. With a further increase in the depth of the wear track, the adhesive interaction of the roller material is enhanced due to the appearance of contact with the titanium alloy; the sliding is accompanied by the process of material transfer from the roller which affects the local setting of the contacting materials. This leads to instability of the coefficient of friction and an increase in the amplitude of its fluctuations as a result of an increase in the contact area of the counter-body with the titanium alloy. A similar character of the change in the coefficient of friction was also observed in [9] when nitrided VT6 alloy was tested on a "ball-on-disc" scheme in pair with a ceramic ball without lubrication.

The studies carried out in this work have shown that, depending on the material in contact with the nitrided alloy VT5, it is possible to provide sliding with sufficiently low values of the coefficient of friction and wear under the chosen test conditions. The presence of a titanium nitride layer on the surface of titanium nitrided alloys ensures their high wear resistance. The reduction of adhesion interaction and tendency to adhesion with contacting materials during sliding is also associated with the presence of a nitride layer on the surface of nitrided alloys. The processes of transferring the counter-body material to the nitrided surface of titanium alloys may play an important role. Based on these positions, an explanation of the results in this work, obtained by testing the friction pair nitrided alloy VT5-cemented steel 20Cr3MoWV without lubrication, is proposed.

4. Conclusions

Tribological tests of various materials (steel 1.4878, steel 1.3505, nitrided steel 20Cr3MoWV, cemented steel 20Cr3MoWV, cast iron GGG80 and bronzes Br.O10C2N3, VB-23NTS and VB-24) have established the following.

Low wear of nitrided titanium ($\leq 2.26 \times 10^{-6}$ mm^3/Nm) and coefficient of friction at the level from 0.08 to 0.12 were obtained in the test with jet fuel TS-1 lubrication in contact with bronzes VB-23NTS, Br.O10C2N3 and VB-24, steel 1.3505, cast iron GGG80 and cemented steel 20Cr3MoWV.

Low values of wear and values of coefficient of friction of steel 1.3505, cast iron GGG80 and cemented steel 20Cr3MoWV in the pair with nitrided alloy VT5 can be explained by the high content of carbon in them, which plays the role of solid lubricant during sliding.

The results of the conducted tests of the nitrided alloy VT5 should be considered as preliminary when considering the possibility of their use in the friction pairs of units in aerospace and other equipment, which may serve as an impetus for further research in this direction.

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