Effect of Hydrogen Heat Treatment on Antifriction Properties of Nitrided VT6 Titanium-Based Alloy

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Abstract—A comparative study of the antifriction characteristics of specimens of the VT6 titanium-based alloy subjected to nitriding without preliminary treatment and after hydrogen heat treatment has been carried out. Hydrogen heat treatment that precedes nitriding reduces the coefficient of friction of the nitrided specimens of the VT6 alloy in pair with the 12Kh18N10T stainless steel by 4–9% under dry friction and increases it by ~47% in a working fluid, which is a 0.9% NaCl solution. Hydrogen heat treatment combined with ion-plasma nitriding reduces the coefficient of friction in working fluid by ~41%.

INTRODUCTION

Titanium and titanium-based alloys have found widespread application as materials for implants [1, 2]. The use of these alloys ensures enhanced biochemical resistance and biomechanical properties of implants compared to the properties of other materials, including stainless steel and cobalt–chromium alloys. The alloys of the Ti–6Al–4V system (ISO 5832-3) are the most popular; one of these alloys is the VT6 alloy, which has better physicomechanical properties than commercial titanium [3–5]. The VT6 titanium-based alloy possesses higher mechanical and performance characteristics than commercial titanium; it is characterized by higher strength, hardness, endurance limit, and abrasive, cavitation, and erosion wear resistances, as well as a lower susceptibility to seizure and scoring. However, like titanium, this alloy has poor wear resistance. The surface treatment of titanium-based alloys is widely used to enhance their wear resistance, and nitriding attracts special attention of researchers for the surface hardening of these alloys [6–10]. The good biocompatibility of titanium nitride with both blood and bone tissue [11, 12], as well as its high mechanical, tribological, and corrosion-resistant characteristics, determine the use of titanium nitride for protecting surfaces of medical implants. In the course of production, medical articles made of titanium-based alloys are subjected to various kinds of treatment, including hydrogen heat treatment, in order to form a three-dimensional structure, which ensures the required functional properties of implants [13, 14].

The aim of this work was to study the effect of hydrogen heat treatment on the tribological behavior of the VT6 titanium-based alloy subjected to nitriding using various methods.

EXPERIMENTAL

The objects of the study were 20 × 20 × 2-mm specimens based on the VT6 ($\alpha + \beta$) titanium-based alloy (Ti–6Al–4V).

Hydrogen Heat Treatment

The specimens were hydrotreated to a concentration of hydrogen of 0.7 vol % using a Siwertz apparatus at a temperature of 850°C and then annealed using a VEGA-3M vacuum furnace at a temperature of 625°C. The duration of annealing was chosen so that the concentration of hydrogen in the course of degassing decreased to a safe concentration of ~0.006 vol %. The concentration of hydrogen after vacuum annealing was determined using an ISP-51 spectrograph equipped with a MOPC-1/2048/PCI attachment.

Thermal-diffusion nitriding was carried out in moleculat nitrogen at atmospheric pressure of ~10^5 Pa and in a dynamic flux of nitrogen rarefied to a pressure of 1 Pa at temperatures of 700 and 750°C. The duration of isothermal holding in saturation was 1 h. Nitriding was performed using commercial gaseous nitrogen (GOST 9293–74). Before being supplied to
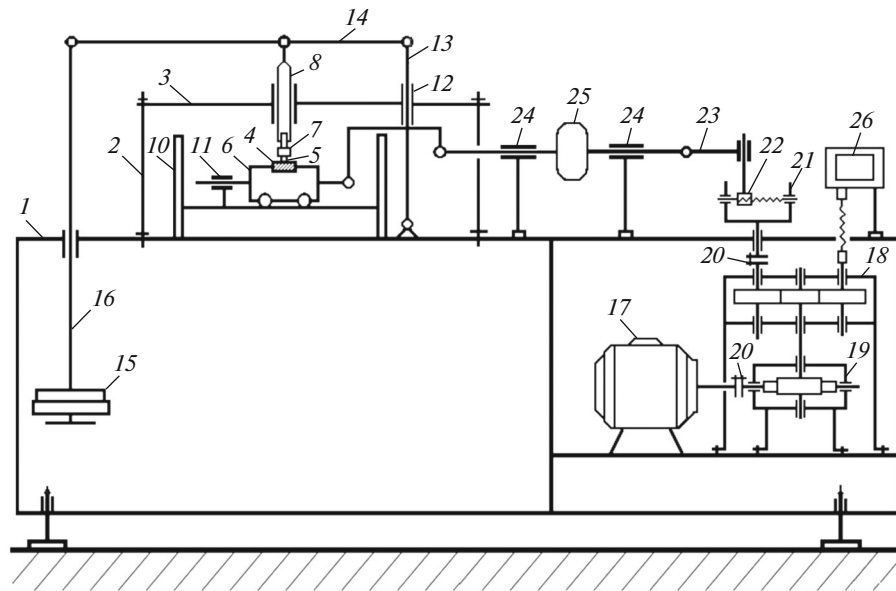
Fig. 1. Schematic of sliding friction machine. Positions are specified in the text.

the reaction chamber of the furnace, nitrogen was purified from moisture and oxygen.

Ion Nitriding

Vacuum ion-plasma nitriding was carried out using a modernized Bulat 6T setup for 40 min in an argon–nitrogen (80% : 20%) mixture under a total pressure of the gas in the chamber of 3×10^{-3} Pa at a temperature of 580°C; the arc current was 100–150 A and the base voltage was 40–60 V. The working mixture of the gases was prepared using a gas producer.

The hardening of surfaces was determined from changes in their microhardness, which was measured using a PMT-3M microhardness tester, and the roughness of the surfaces was measured using a 170621 proflometer. The composition of surface films was determined by X-ray phase analysis implemented in a DRON-3.0 diffractometer using the Bragg–Brentano focusing of monichromatic CuK_α radiation. The tribological characteristics of the coatings were studied using a linear tribometer developed at the Karpenko Physical-Mechanical Institute of the National Academy of Sciences of Ukraine (Lvov).

Reciprocal tribotests were carried out using the linear tribometer. The tribometer is designed for the laboratory assessment of the antifriction and antiwear characteristics of lubricants, structural materials, and coatings under the reciprocal movement of the rubbing solids; the schematic of the tribometer is shown in Fig. 1. This tribometer makes it possible to perform tests under a load applied to the friction unit of 0–250 N at a frequency of oscillations of 30 min^{-1} in a temperature range of 20–25°C. The length of the

stroke can be gradually varied in a range of 0–25 mm. The rubbing surfaces are lubricated by either their immersion into a lubricant or a dosed supply of the lubricant into the zone of friction. Both point (the stationary ball–oscillating plate arrangement) and surface (the stationary cylinder end–oscillating plate arrangement) contacts can be implemented in the tribometer. These arrangements of tribotests are widely used to study the tribological behavior of antifriction and antiwear coatings, which are deposited on the bottom and/or the top specimens, depending on a specific task. Test specimens can be shaped as cylinders 10 mm in diameter and 15 mm high the end surfaces of which play the role of the friction surface (top specimens) or (1–6) × 20 × 45–mm plates (bottom specimens).

The tribometer (Fig. 1) is installed on a metal table *1* that is 500 × 1000 mm in area and 800 mm high. Plate *3*, which serves as the base for mounting the loading mechanism of the pair of specimens *4* and *5*, is fastened to plate *1* using four columns *2*.

Specimen *4* is fixed in a special seat in the top part of slider *6*. Specimen *5* is fastened to the bottom end of rod *8* through insert *7*; the rod can be easily moved in the vertical direction toward specimen *4*. Slider *6* installed on four ball bearings can be reciprocally moved in the horizontal direction over plate *9*, which is placed in bath *10* filled with the lubricant. The displacement of the slider is restructed by guide supports *11*. The static loading of the specimens is implemented using a lever mechanism with a lever ratio of 1 : 5. The lever mechanism consists of bar *13*, which is pin-connected to rocking arm *14* and the movement of which is controlled by sleeve *12*; the rocking arm is pin-connected to rod *8* and loaded using weight *15* through bar *16*. A
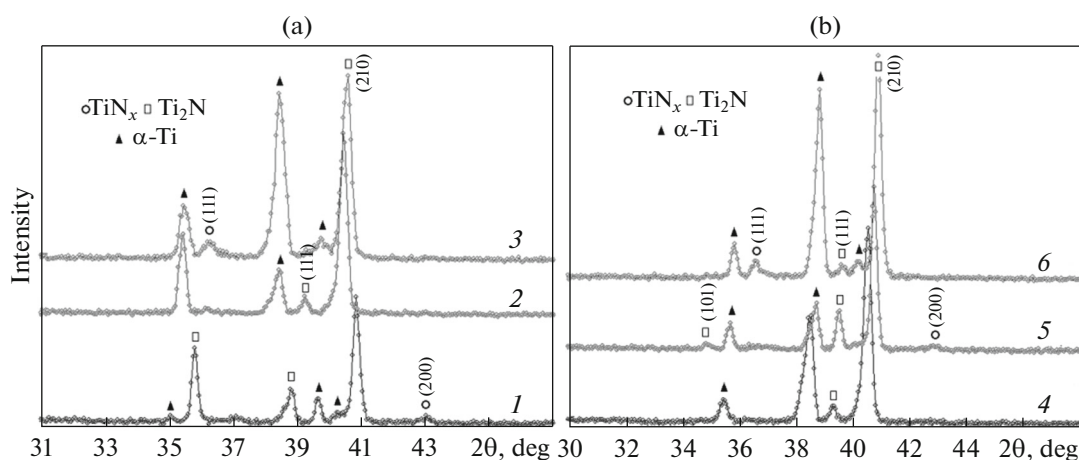
Fig. 2. Fragments of XRD patterns taken from surface of titanium-based VT6 alloy subjected to 1-h nitriding at temperatures of (*1*), (*2*), and (*4*) 750°C and (*5*) 700°C: (*1*) and (*5*) under atmospheric pressure of nitrogen, (*2*) and (*4*) in rarefied dynamic flux of nitrogen; as well as (*3*) and (*6*) after ion nitriding: (a) without and (b) with preliminary hydrogen heat treatment.

required static load applied to the specimens is assigned using a calibration curve. Cyclic reciprocal movement is transmitted to the slider from electric motor *17* using reducers *18* and *19*. Rotational movement is transmitted to the crank mechanism through couplings *20*; the crank mechanism consists of rotating head *21* and screw pair *22*. Nut *22* is pin-connected to crank rod *23*. The movement of the nut over the screw ensures a required length of the stroke during the rotation of the head and produces the reciprocal movement of the crank rod, which transmits the movement to slider *6* with specimen *4* through bars and dynamometer *25*. Thus, the relative movement of the contact surfaces of the pair of specimens *4* and *5* occurs. The force of friction of the specimens is measured using a strain-gage system, which consists of dynamometer *25* connected to a computer to record experimental data. The number of friction cycles is recorded by counter *26*. The test results were processed using mathematical statistics methods.

RESULTS AND DISCUSSION

The structure of the alloy hydrotreated to a concentration of hydrogen of 0.7 vol % at a temperature of 850°C changes in the following sequence: $(\alpha + \beta) \rightarrow [\alpha'(\alpha'') + \beta] \rightarrow \beta$. After the hydrotreating annealing of the VT6 alloy, the primary $\alpha'(\alpha_2)$ phase remains in its structure, while in the course of the low-temperature vacuum annealing of this alloy a structure is formed that is not typical of this alloy in the equilibrium state and consists of primary α'-phase particles, noncoherent α_2-phase particles enriched in aluminum, a small amount of the α phase, and the α_{deg} phase, which is formed from the β phase depleted of aluminum in the course of degassing. During subsequent low-temperature vacuum annealing, an $(\alpha + \beta)$ structure, which contains α-phase nanoparticles, is formed [15]. In

order to produce the original structural and phase state, which implied stress relaxaton and the removal of hydrogen, as well as the homogenization and stabilization of the structure of the alloy, the prepolished specimens were annealed in vacuum for 2 h at pressure $P = 0.5 \times 10^{-4}$ Pa at the temperature $T = 800°C$ and the rate of intrinsic leakage into the vacuum system $I_1 = 0.1 \times 10^{-3}$ Pa dm^3 s^{-1}. The use of hydrogen heat treatment (HHT) followed by vacuum annealing has led to a decrease in the size of structure units of the α phase from 2–5 to 0.1–0.6 μm and an increase in the hardness of the alloy from 32–34 to 38–42 *HRC* [16]. The roughness parameters of the surfaces of the as-prepared specimens corresponded to the 10th roughness class (GOST 2789–73) at the arithmetic average roughness $R_a = 0.08$–0.09 μm.

The HHT influences the structure of the alloy [13, 14]; therefore, during subsequent nitriding it will govern the kinetics of the physicochemical processes that will occur on the surface of the alloy, as well as the structural and phase state of the produced hardened surface layers.

The nitriding of the alloy in molecular nitrogen under atmospheric pressure reduces the quality of its surface within the 10th roughness class to $R_a = 0.095$–0.107 μm. These changes in the microgeometry of the surface are due to intensive nitride formation (Fig. 2, spectrum *1*), which is accompanied by the formation of a surface relief typical of diffusion-assisted nitriding [9]. The nitride surface film primarily consists of titanium nitride with the lowest valence of titanium; this is confirmed by the presence of reflections (200), (111), and (210), which correspond to the Ti$_2$N phas, in the XRD spectrum. The titanium mononitride TiN$_x$ phase is only presented by the reflection (200) with a lower relative intensity. The HHT, which precedes the nitriding at the chosen parameters (at tem-

Table 1. Microhardness of surfaces of specimens of VT6 alloy subjected to nitriding under various conditions without and with preliminary hydrogen heat treatment as function of load applied to indenter

Conditions of nitriding		Microhardness of surface H_μ^s, GPa		
		load applied to indenter, N	preliminary treatment	
			without HHT	with HHT
1	750°C, 1 h, 10^5 Pa N_2	0.196	9.7 ± 0.9	10.1 ± 0.5
		0.49	8.7 ± 0.8	6.8 ± 0.4
		0.96	7.3 ± 0.6	6.1 ± 0.4
2	700°C, 1 h, 10^5 Pa N_2	0.196	–	9.0 ± 0.4
		0.49	–	6.5 ± 0.3
		0.96	–	5.1 ± 0.5
3	750°C, 1 h, 1 Pa N_2	0.196	5.8 ± 0.5	9.2 ± 1.4
		0.49	5.7 ± 1.1	7.4 ± 0.5
		0.96	5.1 ± 0.3	6.2 ± 0.3
Ion nitriding, 580°C, 40 min, 0.6×10^{-3} Pa N_2		0.196	7.4 ± 1.1	6.9 ± 0.6
		0.49	5.7 ± 0.6	6.8 ± 0.2
		0.96	5.0 ± 0.3	5.4 ± 0.5

peratures of 700 and 750°C and the duration of the nitriding of 1 h), intensifies nitride formation due to the refinement of the structure of the alloy, which results in a rougher relief of the surfaces of the specimens.

Nitriding in rarefied molecular nitrogen leads to the retardation of the process of nitride formation on the surface of the alloy [9], which is confirmed by a decrease in the intensity of the reflections of the Ti_2N phase and the absence of reflections of the TiN_x phase in the XRD pattern (Fig. 2, spectrum 2). In this case, the quality of the surface of the alloy subjected to nitriding is retained; i.e., the roughness of the surfaces of the specimens decreases to $R_a = 0.067–0.069$ μm. The HHT, which precedes this nitriding, favors the intensification of the formation of nitrides on the surface of the alloy; this is confirmed by an increase in the intensity of the reflections of the Ti_2N titanium nitride phase with the lowest valence of titanium, which are present in the XRD spectrum recorded from the surface (Fig. 2, spectrum 4). Thus, the relative intensity of the reflection (210) increases from 872 to 994 arb. units. However, in this case, this hardly deteriorates the quality of the hardened surface.

Like the thermal-diffusion saturation in molecular nitrogen at atmospheric pressure, the ion nitriding deteriorates the quality of the original surface to $R_a = 0.128–0.139$ μm, while the HHT, which precedes the nitriding, increases the roughness of the surface. The surface film also consists of TiN_x and Ti_2N nitrides the relative intensity of the reflections of which increases if the preliminary HTT has been carried out (Fig. 2, spectra 3 and 6).

An analysis of the surface hardening of the nitrided specimens using the measurements of the microhardness has shown that, with an increase in the load applied to the indenter from 0.196 to 0.981 N, the microhardness of the surface decreases (Table 1). This is related to the fact that the microhardness is an integral characteristic of the material and depends on the depth of indentation. The changes in the microhardness of the surface due to varying load applied to the indenter show the gradient-like character of surface hardening, which is typical of diffusion-assisted processes. With an increase in the depth of indentation, the microhardness decreases since the concentration of the diffusant impurity, in particular, nitrogen and, therefore, the hardening decrease. This approach makes it possible to determine the gradient of the characteristics in a thin (up to 2 μm thick) surface layer using the difference in the values of the microhardness of the surface measured under various loads applied to the indenter in accordance with GOST 9450–76.

It has been found that the microhardness of the surface subjected to the nitriding under conditions 1 is by 2.3–3.9 GPa higher than that of the surface subjected to the nitriding under conditions 3 due to more intensive nitride formation. Although the ion nitriding is carried out under the lower pressure of nitrogen (0.6×10^{-3} Pa) than nitriding carried out under conditions 3, it ensures a higher microhardness of the surface of 7.4 GPa.

The HHT, which precedes nitriding, enhances the surface hardening effect. The use of the HHT leads to an increase in not only the microhardness of the surface, but
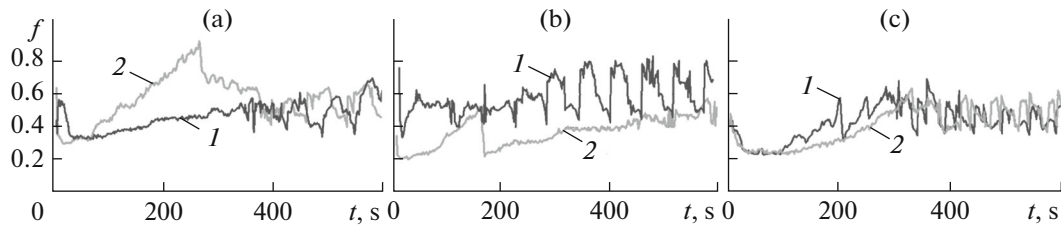
Fig. 3. Kinetics of the coefficient of friction of 12Kh18N10T steel in pair with VT6 alloy subjected to nitriding under various conditions without preliminary hydrogen heat treatment: (a) 750°C, 1 h, 10^5 Pa N_2; (b) 750°C, 1 h, 1 Pa N_2; and (c) ion nitriding; (*1*) dry friction and (*2*) lubrication with 0.9% NaCl solution. Contact pressure is 0.354 kg/mm^2.

also its gradient in the subsurface layer (Table 1). The nitriding carried out under conditions 1 after the HHT increases the increment in the microhardness of the surfaces of the specimens, which is equal to 0.4–1.2 GPa, compared to that obtained for the specimens produced without the use of the HHT. The HHT followed by the nitriding carried out under conditions 3 increases the microhardness of the surface by 1.1–3.4 GPa. This is related to the intensification of the diffusion processes due to the HTT and the effect of the HTT on the structural and phase state of the subsurface layers [14]. After the ion nitriding of the specimens subjected to the HHT, the increase in the microhardness of the surface and the gradient of the microhardness in the thin surface layer decrease, while the average values of these characteristics are still higher than those obtained for the specimens produced without the use of the HHT. This can be explained by nitrogen deficiency in the strongly rarefied gas atmosphere due to the increasing diffusion-assisted removal of nitrogen from the surface. The HHT and the decrease in the nitriding temperature from 750 to 700°C (conditions 2) do not influence the quality of the hardened surface ($R_a \approx 0.105$ μm); they reduce the microhardness of the surface only slightly, while the gradient of the microhardness in the thin subsurface layer remains unchanged (Table 1).

Physicomechanical characteristics of hardened surface layers govern the behavior of these layers under friction.

The tribological behavior of the VT6 alloy, which was subjected to nitriding with and without preceding HHT, was studied under dry friction conditions and in a working fluid, which was a 0.9% NaCl solution. The tribotests were carried out using the linear contact of the specimens in accordance with the stationary cylinder end (counterbody)–oscillating plate (specimen) arrangement, which was implemented in the developed tribometer. The counterbody was made of the 12Kh18N10T steel with the Rockwell hardness $HRA = 52 \pm 1.5$. The tests were performed under a load of 0.354 kg/mm^2, a frequency of oscillations of 30 min^{-1}, a length of stroke of 10 mm, and a temperature of

24°C. The friction pair was lubricated by immersing it into the 0.9% isotonic NaCl solution.

During the dry friction of the specimens nitrided under conditions 1 against the steel, the coefficient of friction rises sharply from 0.36 to 0.55 upon 45 s of the test, then decreases to 0.33 after which it monotonously increases to 0.51 during a period of 300 s (Fig. 3a, curve *1*). With a further increase in the duration of the test, fluctuations in the coefficient of friction arise the amplitude and perod of which increase with increasing duration of the test. This is indicative of the continuous fracture of the nitride film, as well as the cyclic seizure of the specimen and the counterbody. The coefficient of friction averaged over the chosen duration of the test is equal to 0.46.

The dependence of the coefficient of friction on the duration of the test shows that the wear resistance of the nitrided surface of the VT6 alloy is lower for the specimen subjected to the thermal-diffusion saturation with nitrogen under conditions 3 (Fig. 3b, curve *1*). This can be explained by a decrease in the thickness of the surface nitride film and to the phase composition of this film (ε titanium nitride Ti_2N) (Fig. 2) and, therefore, a decrease in the surface hardening (Table 1). As early as the initial stage of the test of the VT6 alloy nitrided under conditions 3–steel friction pair, the coefficient of friction is characterized by slight fluctuations, which upon 290 s of the test become larger and periodical (Fig. 3b, curve *1*). The coefficient of friction averaged over the chosen duration of the test for the specimens nitrided under conditions 3 is ~1.25 times higher than for specimens nitrided under conditions 1 (Table 2).

The coefficient of friction of the specimens of the VT6 alloy nitrided under conditions 1 in pair with the steel in the 0.9% NaCl solution increases from 0.30 to 0.92 for a period of testing of 270 s (Fig. 3a, curve *2*). During the subsequent 110-s period of testing, the coefficient of friction decreases to 0.60. With a further increase in the duration of the test, fluctuations in the coefficient of friction with regard to its average value of 0.53 arise; the amplitude of the fluctuations increases with increasing duration of the test. In general, the coefficient of friction of the nitrided VT6 alloy in pair

Table 2. Coefficient of friction averaged over 600 s of tests of nitrided VT6 alloy−12Kh18N10T steel pair carried out under contact pressure of 0.354 kg/mm^2

Conditions of nitriding		Coefficient of friction f		
		conditions of friction	preliminary treatment	
			without HHT	without HHT
1	750°C, 1 h, 10^5 Pa N$_2$	Dry friction	0.46	0.44
2	700°C, 1 h, 10^5 Pa N$_2$		–	0.46
3	750°C, 1 h, 1 Pa N$_2$		0.58	0.53
Ion nitriding, 580°C, 40 min, 0.6 × 10^{-3} Pa N$_2$			0.43	0.41
1	750°C, 1 h, 10^5 Pa N$_2$	0.9% NaCl solution	0.57	0.58
2	700°C, 1 h, 10^5 Pa N$_2$		–	0.35
3	750°C, 1 h, 1 Pa N$_2$		0.38	0.50
Ion nitriding, 580 °C, 40 min, 0.6 × 10^{-3} Pa N$_2$			0.41	0.24

with the steel in the working fluid is ~1.2 times higher than under dry friction (Table 2).

In general, the character of variations in the coefficient of friction of the nitruded layer produed under conditions 3 in pair with the steel in the working fluid is similar to that observed for the nitruded layer produced under conditions 1. Thus, during the first 160 s of the test the coefficient of friction increases from 0.21 to 0.53 and then sharply drops to 0.24 after which it monotonously increases with slight fluctuations, which arise upon 470 s of the test (Fig. 3*b*, curve *2*). The coefficient of friction averaged over the chosen duration of the test is equal to 0.38. However, we note that, in this case, the coefficient of friction in the working fluid is 1.5 times lower than under dry friction (Table 2).

The working fluid has also a positive effect on the coefficient of friction of the pair in which the surface of the VT6 alloy has been hardened using ion nitriding. This effect is observed in the dependences of the coefficient of friction on the duration of the test as the 130-s increase in the period of the monotonous growth in the coefficient of friction without fluctuations

(Fig. 3c, curve *2*); in this case, the average coefficient of friction is lower than under dry friction (Table 2).

The HHT, which precedes the nitriding, has little or no qualitative effect on the above-considered regularities of variations in the coefficnet of friction during the tests of the friction pairs under study (Figs. 3, 4). However, substantial differences arise in the coefficients of friction of the pairs that contain the nitrided alloys subjected and not subjected to the preliminary HHT (Table 2).

The use of the preliminary HHT leads to a decrease in the average coefficient of the dry friction of the specimens nitrided using conditions 1 (Table 2). The coefficient of friction in the working fluid remains at the previous level of 0.57−0.58. While using the preliminary HHT and retaining the gas-dynamic parameters of saturaton, the decrease in the nitriding temperature from 750 to 700°C (conditions 2) enhances the tribological characteristics of the surface of the alloy in the working fluid; the average coefficient of friction decreases 1.7 times (Table 2) apparently due to the level of the hardening (Table 1) and the quality of the nitrided surface. The tribological characteristics of
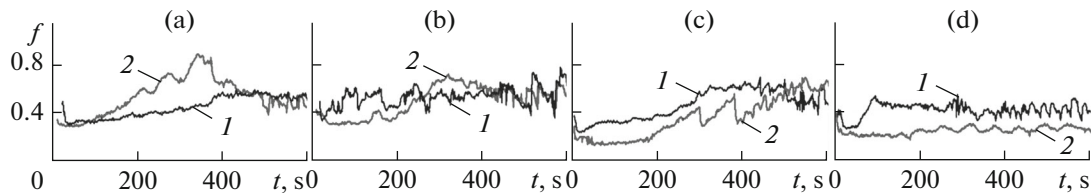
Fig. 4. Kinetics of coefficient of friction of 12Kh18N10T steel in pair with VT6 alloy subjected to nitriding under various conditions with preliminary hydrogen heat treatment: (a) 750°C, 1 h, 10^5 Pa N$_2$; (b) 750°C, 1 h, 1 Pa N$_2$; (c) 700°C, 1 h, 10^5 Pa N$_2$; and (d) ion nitriding; (*1*) dry friction and (*2*) lubrication with 0.9% NaCl solution. Contact pressure is 0.354 kg/mm^2.
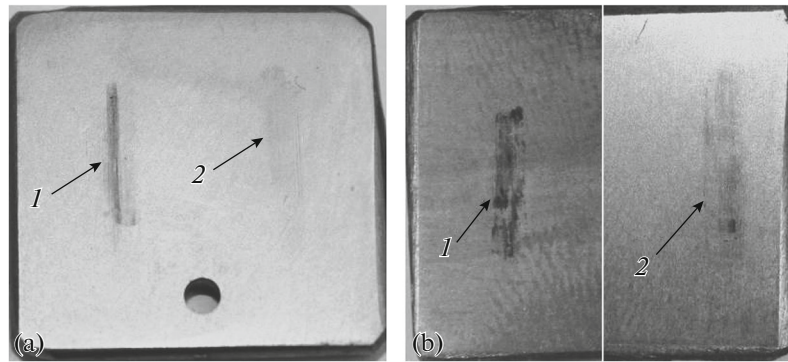
Fig. 5. Photos of friction tracks on surface of nitrided VT6 alloy after friction against 12Kh18N10T steel: (a) 1-h nitriding at 700°C under 10^5 Pa N_2 with preliminary hydrogen heat treatment ((*1*) dry friction and (*2*) lubrication with 0.9% NaCl solution), as well as (b) ion nitriding (*1*) without and (*2*) with preliminary hydrogen heat treatment.

the surface under dry friction deteriorate, which is confirmed by a decrease in the coefficient of friction (Table 2) and the photos of the friction tracks (Fig. 5a).

The use of the HHT, which precedes the nitriding, leads to a decrease in the coefficient of friction under the dry conditions and, especially (~1.7 times) in the working fluid (Table 2). The efficiency of the influence of the working fluid when using the HHT, which precedes the nitriding, is also confirmed by the photos of the friction tracks (Fig. 5b) in which less severe damage to the surface is observed. Upon 260 s of the dry tribotest, fluctuations in the coefficient of friction arise (Fig. 4d, curve *1*). The presence of the working fluid favors a substantial increase in the period of the fluctuations and a decrease in their amplitude, which ensures a fairly steady coefficient of friction during the entire test period (Fig. 4d, curve *2*).

CONCLUSIONS

The preliminary HHT enhances the surface hardening effect when using the nitriding of the alloy in the molecular nitrogen. This leads to an increase in not only the microhardness of the surface, but also the gradient of the microhardness in the subsurface layer. When using ion nitriding, both the microhardness of the surface and the gradient of the microhardness in the thin (up to 1 μm) subsurface layer increase.

The preliminary HHT and the nitriding of the specimens of the VT6 alloy under all conditions lead to a decrease in the coefficient of friction of this alloy in pair with the 12Kh18N10T steel in the dry tribotests by 4–9%.

The HHT and the nitriding of the specimens of the VT6 alloy in molecular nitrogen under atmospheric pressure lead to a slight (by ~2%) increase in the coefficient of friction of the alloy in pair with the steel in the 0.9% NaCl solution. Under the same test conditions, the tribological characteristics of the friction pair with the alloy nitrided in the rarefied dynamic

flux of nitrogen deteriorate more severely; i.e., the coefficient of friction increases by ~30%. The HHT and the ion nitriding of the specimens of the VT6 alloy lead to a decrease in the coefficient of friction of the alloy in pair with the 12Kh18N10T steel in the 0.9% NaCl solution by ~40%.

NOTATION

P pressure of gas, Pa

I_1 intrinsic leakage to vacuum system, Pa dm^3 s^{-1}

R_a arithmetic average roughness, μm

I intensity of XRD reflections, arb. units

2θ diffraction angle, deg

H_μ^s microhardness of the surface, Pa

f coefficient of sliding friction

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