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CORROSION AND MECHANICAL WEAR OF NITROGEN STEELS IN ACID ENVIRONMENTS

M. Stechyshyn [1], A. Dykha [1,*], J. Padgurskas [2], N. Stechyshyna [1]

[1] Department of Tribology, Automobiles and Materials Science, Khmelnitsky National University, Ukraine

[2] Institute of Power and Transport Machinery Engineering, Vytautas Magnus University, Lithuania

Abstract: The work is devoted to the study of the tribological properties of steels and cast iron strengthened by anhydrous nitriding in a glow discharge operating in acidic environments. Electrochemical behavior, wear resistance and corrosion resistance of nitrided surfaces in technological acidic environments have not been sufficiently investigated. Without considering the influence of operational factors on wear conditions leads to premature failure of processing and food industry equipment. The results of the study of corrosion-mechanical wear of nitrided and non-nitrided steels (C20; C45; 37Cr4; 41CrAlMo7) in an acidic model environment are given. The dependences of the intensity of wear and the coefficient of friction on the pressure in the frictional contact and on the sliding, speed was obtained. Results were also obtained on the change in the ratio of corrosive and mechanical wear factors depending on the pressure and sliding speed. The analysis of the effect of the temperature of the surface layers on the tribological properties of nitrided samples operating in an acidic environment was carried out. The influence of temperature and sucrose on the intensity of destruction of surfaces was clarified. The obtained results make it possible to make the optimal selection of hardening modes and operating conditions of nitrided surfaces working in acidic production environments.

Keywords: corrosion-mechanical wear, anhydrous nitriding in the glow discharge, the work of friction forces, internal energy

1. INTRODUCTION AND LITERATURE REVIEW

Much attention in the study of the problem of increasing the reliability and durability of machines and equipment is given to the problem of friction and wear, since the over-whelming majority of machines fail due to surface wear of individual parts and assem-blies. In the field of mechanical engineering, financial losses due to friction and wear reach 5% of the national income. At the same time, according to research data, from a quarter to a third of all the energy generated in the world during the year is currently spent on overcoming friction forces in the moving joints of machines. When solving the problem of reducing power costs for friction and wear in machines and increasing their reliability and durability, the main set of measures is related to the choice of materials for friction pairs. The need to maintain a rational ratio between their cost and the appropriate level of operational characteristics will contribute to the growth of the role of various methods of strengthening surface layers, since it is the condition of the surface that determines the durability of machine parts. The structural and phase state of the surface layers of metals determines their wear resistance. One of the effective methods that improves the physical and mechanical properties of structural steel surfaces is chemical heat treatment. Chemical-thermal treatment in controlled oxygen and nitrogen-containing gaseous media pro-vides the formation of a gradient hardened layer. Depending on the physical and mechanical characteristics of the near-surface layer modified with oxygen or nitrogen, it is possible to increase the reliability under different types of loads [1-2]. It is also possible to significantly increase the wear resistance of technological methods [3-4]. It is shown that the increase of corrosion and mechanical strength during friction and cavitation of nitrided structural steels is due to the formation

* Author for contacts: Prof. dr. Aleksandr Dykha
E–mail: tribosenator@gmail.com

of the maximum level of residual compressive stresses [5], grinding subgrain structure and the formation of ordered cell dislocation structure [6]. In [7] the electrochemical and corrosion characteristics of diffusion layers formed in metals nitrided in a glow discharge were studied in acidic model media. A relationship has been established between the thermodynamic potential of nitrided surfaces and their electrode potential. In [8-10] the tribological behavior of steel parts with modified surface layers in different operating conditions during fretting, for reinforcing tools and others is analyzed. Further progressive directions of application of technical surfaces of machine parts strengthened by nitriding in glow discharge and recommendations for practical use are also considered in papers [11-14].
Therefore, there is practically no information in the literature regarding tribological tests of steels nitrided in a glow discharge, which are operated in acidic environment.

2. RESEARCH METHODOLOGY

Structural steels of the following grades were selected for the study: C20, C45 - high-quality carbon, 37Cr44 - chromium and 41CrAlMo7-chromium-aluminum with molybdenum of high quality. The experiments were performed in an acidic model environment: 2% citric acid solution $C3H8O7 \times H2O$. Hydrogen nitriding in the glow dis-charge was carried out at the industrial installation BATR, which is additionally equipped with heating elements placed in the gas discharge chamber. This made it possible to arbitrarily change the energy parameters: voltage U and the value of current density g (current ratio to the total area of the cage and suspension). To study the corrosion-mechanical wear resistance of materials in a wide range of loads and working environments, an end friction setup was used (Figure 1).

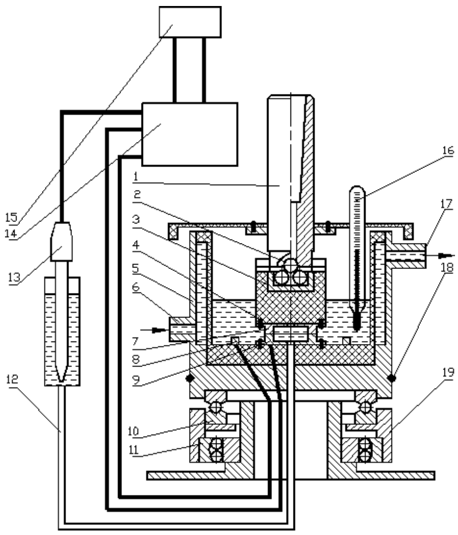

Figure 1. Installation of end friction: cone 1, four balls 2, body 3, screws 4, electrochemical cell 5, upper sample 7, fitting 6-17, electrode 8, lower sample 9, bearings 10-11, tube 12, reference electrode 13, potentiometer 14, potentiometer 15, thermometer 16, housing 18.

The friction machine makes it possible to obtain the kinetics of changes in the electrode potential, frictional and wear-resistant characteristics, the temperature of the friction surface, and the friction characteristic depending on the change in the electrode potential of the system. The installation consists of a rigid bed, a working chamber, a spindle assembly, a drive and a loading device, measuring and recording equipment for measuring and recording electrode potentials, friction characteristics and temperature on the friction surface of the sample.

The kinetics of electrochemical processes in static conditions, with stirring and friction were studied on a P-5827M potentiostat, and polarization curves were recorded with a PDP4-002 potentiometer. Electrode potentials were measured relative to the chlorine-silver reference electrode EVL-1M1 in a saturated KCl solution supplied to the friction zone.

The wear resistance of the friction pair was evaluated by measuring the linear wear of the samples and recording the friction force. The force of friction was measured by the densiometric method. The linear wear of the samples was determined by an indicator with a division price of 1μm. The upper sample made of CT105 steel, hardened to HRC 61…63, had two grooves that provided free access of the medium to the friction surface. The sample of the studied material served as the bottom. The heating of the samples in the contact zone did not exceed 393 K, which excluded the possibility of structural transformations.

The average temperature on the surface of the sample was measured using a KSP-2-23 electronic potentiometer at a distance of 1 mm from the friction surface. For this purpose, a chromel-alumel thermocouple with a diameter of 0.5 mm was used.

Polarization curves in statics and during friction were recorded in the potentiodynamic mode. The sweep rate (change in the potential of the working electrode over time) was constant in all experiments (with a potential multiplier equal to one). A silver chloride electrode immersed in an electrolyte served as a standard electrode. Auxiliary - platinum electrode was placed until the contact was completely immersed in the working solution. The corrosion current was determined by extrapolation of the linear Tafel segments of the polarization curves to the region of low overvoltages. According to the corrosion current, considering the fact that iron passes into solution in the form of divalent ions, the corrosive weight loss of the samples was determined according to the Faraday law. The total wear of the samples was measured on a friction machine. To separate the mechanical component, the calculated corrosion mass losses were subtracted from the total wear.

A comparison of the results of weight loss determined by the gravimetric method and by the corrosion current shows that the relative error at is no more than 7% in an acidic environment. The results obtained indicate a satisfactory correlation between the intensity of corrosion processes obtained by electrochemical and gravimetric methods. Thus, the corrosion current and the mass loss calculated from it are used to quantify the corrosion factor of destruction in this type of wear.

Metallographic studies of nitrided samples were performed after etching in 3 percent alcoholic nitric acid solution. Measurements of the thickness of the nitride zone were per-formed on an RX50M microscope. Microhardness was determined on a DuraScan-20 microhardness tester at a load of 1.0 N. When conducting electrochemical measurements, experiments were repeated 3…5 times, and during corrosion and wear tests, they were repeated 5…6 times. Statistical processing of the obtained results was carried out according to standard methods.

3. RESEARCH RESULTS AND DISCUSSION

Figure 1 shows the structure of the surface layer of 37Cr4 steel after applying the technology of anhydrous nitriding in a glow discharge. The structure and phase composition of nitrided layers is determined by a combination of technological and energetic processes of formation of the nitrided layer. Strengthening was carried out according to optimal modes in relation to operating conditions, i.e. increasing the corrosion-mechanical wear resistance of steels in acidic environments (843 K, 75% N2 + 25% Ar, 265 Pa, 4 h), considering the results of work [5]. In the photo, the nitride layer is represented by a white stripe.

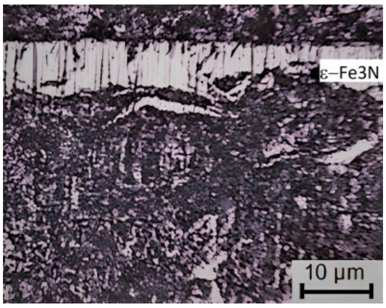

Figure 2. Microplates of nitrided steel 37Cr4 (8.7μm – nitride zone; 300μm – modified zone).

In the initial state, the structure of the nitrided layer consists of a layer of iron nitrides, represented mainly by ε – Fe3N and a diffusion zone consisting of an α-solid solution doped with nitrogen and nitrides of alloying elements.

To analyze the tribological properties of the investigated materials, the results of wear and friction tests were obtained depending on the pressure on the frictional contact and the sliding speed. Figure 3 (a, b), as an example, shows graphical dependences of wear intensity for 41CrAlMo7 steel for samples hardened and not hardened by nitriding.

Based on the analysis of the obtained experimental results, it was established (Figure 3) that with an increase in the pressure in the frictional contact, the intensity of wear of nitrided and non-nitrided materials increases. And for the sliding speed v = 0.5 m/s for nitrided layers, on the contrary, it decreases (Figure 3a). Unhardened steels 41CrAlMo7 and 37Cr4 wear the least at a sliding speed of 0.5 m/s (Fig. 3b).

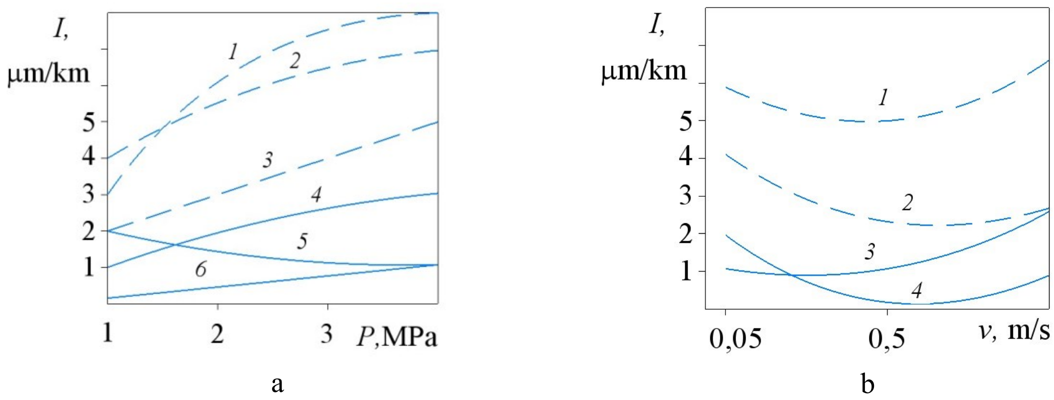

Figure 3. Dependence of the wear intensity I of steel 41CrAlMo7 in an acidic environment on pressure P (a) and sliding speed v (b): dashed lines – without hardening; solid – after nitriding (a: 1, 4 – v = 1 m/s, 2, 5 – 0.05, 3, 6 – 0.5; b: 1, 3 – P = 4 MPa, 2, 4 – 1).

Wear of other materials (hardened by nitriding and unhardened) increases with in-creasing sliding speed (Table 1). As the pressure in the frictional contact increases, the friction coefficient f increases, and at v = 0.05 m/s it remains constant in the entire investigated pressure range (Figure 3a). At a pressure of 4 MPa, it has a maximum at a speed of v = 0.5 m/s, and in other cases, it decreases with increasing sliding speed (Figure 3b).

Table 1 shows an array of data from the experimental results of determining the tribological characteristics of nitrided and unhardened steels depending on operational factors. The numerator shows the values for unreinforced materials, and the denominator shows the values for reinforced ones. These results are averaged after appropriate statistical processing. All obtained results show a positive effect of nitriding hardening on the reduction of wear intensity and friction coefficient.

Table 1. The dependence of the intensity of wear I (μm/km) and the coefficient of friction f of materials in an acidic environment on the sliding speed v and pressure P.

Material		Sliding speed *v*, m/s								
		0.05			0.5			1.0		
		Friction contact pressure *P*, MPa								
		1.0	2.0	4.0	1.0	2.0	4.0	1.0	2.0	4.0
C20	*I*	$\frac{4}{2}$	$\frac{5.5}{1}$	$\frac{7}{0.4}$	$\frac{2}{0.25}$	$\frac{10}{0.5}$	$\frac{14}{1.2}$	$\frac{3}{1}$	$\frac{7}{3.5}$	$\frac{9}{5}$
	f	$\frac{0.32}{0.25}$	$\frac{0.32}{0.25}$	$\frac{0.32}{0.25}$	$\frac{0.20}{0.14}$	$\frac{0.39}{0.25}$	$\frac{0.65}{0.54}$	$\frac{0.07}{0.06}$	$\frac{0.12}{0.10}$	$\frac{0.19}{0.16}$
C45	*I*	$\frac{4}{2}$	$\frac{5.5}{1}$	$\frac{7.0}{0.7}$	$\frac{2.00}{0.25}$	$\frac{8.00}{0.5}$	$\frac{10}{1.1}$	$\frac{3}{1}$	$\frac{7}{3}$	$\frac{9}{4}$
	f	$\frac{0.32}{0.25}$	$\frac{0.32}{0.25}$	$\frac{0.32}{0.25}$	$\frac{0.20}{0.14}$	$\frac{0.35}{0.25}$	$\frac{0.63}{0.53}$	$\frac{0.07}{0.06}$	$\frac{0.12}{0.10}$	$\frac{0.19}{0.16}$
37Cr4	*I*	$\frac{4}{2}$	$\frac{5.5}{1.5}$	$\frac{7}{1}$	$\frac{2.00}{0.25}$	$\frac{3.0}{0.5}$	$\frac{5}{1}$	$\frac{3}{1}$	$\frac{6}{2}$	$\frac{8}{3}$
	f	$\frac{0.32}{0.25}$	$\frac{0.32}{0.25}$	$\frac{0.32}{0.25}$	$\frac{0.20}{0.14}$	$\frac{0.32}{0.24}$	$\frac{0.54}{0.50}$	$\frac{0.07}{0.06}$	$\frac{0.11}{0.09}$	$\frac{0.18}{0.15}$
41CrAlMo7	*I*	$\frac{4}{2}$	$\frac{5.5}{1.5}$	$\frac{7}{1}$	$\frac{2.00}{0.25}$	$\frac{3.0}{0.5}$	$\frac{5.0}{0.9}$	$\frac{3}{1}$	$\frac{6.0}{1.5}$	$\frac{8}{2}$
	f	$\frac{0.32}{0.25}$	$\frac{0.32}{0.25}$	$\frac{0.32}{0.25}$	$\frac{0.20}{0.14}$	$\frac{0.32}{0.24}$	$\frac{0.53}{0.50}$	$\frac{0.07}{0.06}$	$\frac{0.11}{0.09}$	$\frac{0.18}{0.15}$

Numerator - unreinforced materials, denominator - strengthened by nitriding.

The decrease in the intensity of wear of 37Cr4 and 41CrAlMo7 steels with an increase in the sliding speed from 0.05 to 0.5 m/s is explained by the reduction of the contact time of individual contact spots, the interaction of the corrosive environment with juvenile are-as of the surface, and the partial manifestation of the hydrodynamic effect.

For unstrengthened steels 20C, 45C, the maximum intensity of wear was recorded at v = 0.5 m/s and P = 4 MPa, which was caused by the activation of the mechanical factor of destruction. For nitrided materials, the minimum wear resistance corresponds to a speed of 1 m/s and a pressure of 4 MPa.

It is obvious that the high coefficient of friction (Figure 4a) and low intensity of wear for alloyed steels 37Cr4 and 41CrAlMo7 (Figure 4b) at speeds v = 0.5 m/s can also be explained by the formation of dense protective films, the strength of which is much higher than on carbon steels.

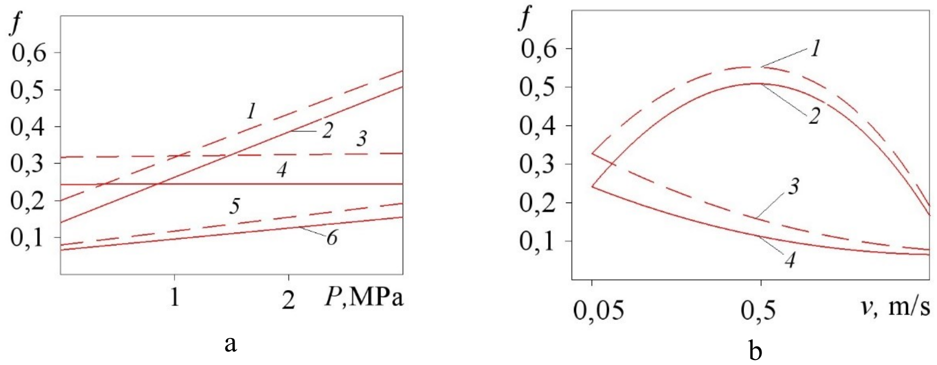

Figure 4. Dependence of the friction coefficient f of steel 37Cr4 in an acidic environment on pressure P (a) and the sliding speed v (b); dashed lines – without hardening; solid – after nitriding (a: 1, 2 – v = 0.15 m/s, 3, 4 – 0.05, 5, 6 – 1; b: 1, 2 – P = 4 MPa, 3, 4 – 1).

The ratio of corrosive and mechanical components in the general process of wear under the action of a cold environment was also investigated. The results of such studies are shown in Table 2 and Figure 4.

Table 2. Corrosion and mechanical wear ratio depending on pressure during friction of 37Cr4 steel in an acidic environment.

Pressure, MPa	Corrosion current, A/m^2	Corrosion rate	Intensity of mechanical wear	Total wear rate	The percentage of corrosion destruction, %
		g/(m^2 · h)			
1	1,77	1,84	0,16	2	92
2	0,86	0,9	0,6	1,5	60
4	0,41	0,43	0,57	1	43

Figure 5 shows a graphical interpretation of surface wear rates versus corrosion and mechanical components as the contact pressure increases. The graphs show that increasing the load reduces corrosion, but activates mechanical wear.

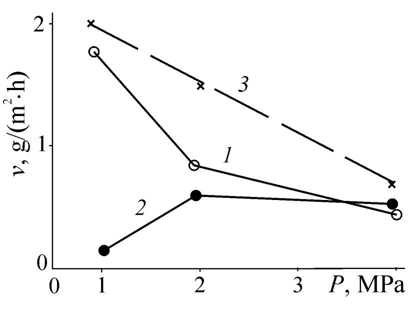

Figure 5. Dependence of the corrosion rate (curve 1), mechanical (2) and total (3) wear during friction of steel 40X in an acidic environment on the pressure is the friction contact P (v = 0.05 m/s).

The next block of research is devoted to the analysis of the influence of the temperature of the surface layers on the tribological properties of nitrided samples operating in an acidic environment. Table 3 shows how the working load in frictional contact affects the formation of the surface temperature.

Table 3. The temperature of the nitrided surface of steel 37Cr4 when rubbed in an acidic environment depending on the load conditions on the friction contact.

v = 0,5 m/s			v = 1 m/s		
P, MPa			P, MPa		
1	2	4	1	2	4
298/298	318/303	333/313	298/298	333/313	363/338

The numerator is normalization, the denominator is nitriding.

The percentage share of the corrosion factor of wear in the general process of nitrided steel 37Cr4 decreases with increasing pressure. Based on the results of the research (Table 2), the dependence of the corrosion and mechanical factors of destruction on the pressure shows (Figure 5) that at v = 0.05 m/s the rate of total mass loss v_t (up to P = 4 MPa) is con-trolled by the rate of corrosion loss, which decreases with increasing pressure in the frictional contact. It is obvious that at the initial moment of corrosion-mechanical impact, the formed juvenile surfaces are covered with protective films (increase in v_{mech} to P = 2 MPa), which shield the friction surface from the influence of an acidic environment (decrease in v_{corr} to P = 2 MPa). At the same time, the forces in frictional contact are insufficient for their destruction.

With an increase in the pressure in the frictional contact from 1 to 2 MPa, the influence of the corrosive factor is sharply weakened and the mechanical factor increases up to P = 4 MPa. Further, the mechanical factor of destruction begins to prevail over the corrosive factor.

One of the factors that have a significant impact on the behavior of nitrided layers is the friction surface temperature. As can be seen from Table 3, increasing the pressure on the friction contact leads to an increase in temperature at the friction surface. The temperature on the nitrided surface is much lower than on the surface after the normalization process due to the higher heat capacity of iron nitrides compared to iron. This creates the conditions to prevent temperature flares on the surface during friction.

In Figure 6 shows graphs for the intensity of wear of steel depending on the parameters of ionic nitriding. From Figure 6 it follows that the most optimal structure of the nitrided surface layer is formed at T = 843 - 873K, nitrogen content of 75% and gas pressure of 265 MPa.

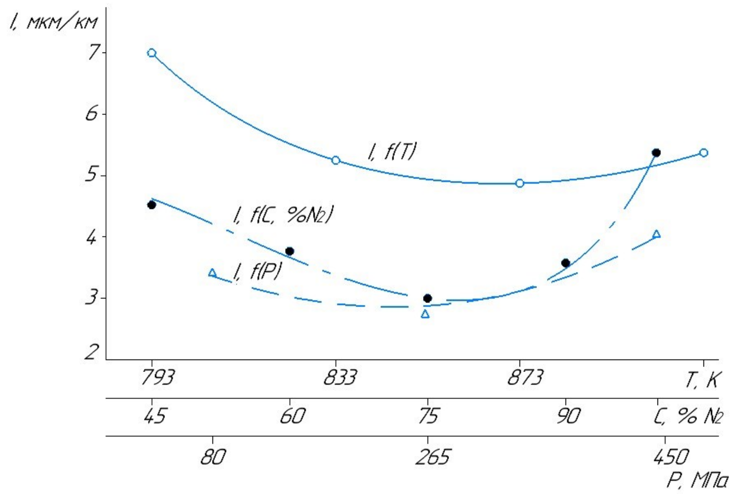

Figure 6. Dependence of the intensity of wear I of steel 37Cr4 in an acidic environment on the parameters of ionic nitriding (wear test mode: sliding speed v = 1m/s, pressure P= 4 MPa): 1- I=f(T);2- I=f(C,% N2); 3- I=f(P).

It was found that steel 37Cr4 has the lowest wear resistance at a nitriding temperature of 793 K. ε-phase, which is formed in this case on the steel surface has increased brittleness, which significantly reduces the wear resistance of the surface layer. Increasing the nitriding temperature to 873 K increases the stability of steel, which is caused by an increase in the relaxation capacity of the surface layers due to a decrease in the concentration of nitrogen in it. A further increase in temperature to 913 K leads to a decrease in wear resistance. The decrease in wear resistance with increasing pressure of the nitrogen-argon mixture to 450 MPa and an increase in its nitrogen content is also due to an increase in the content of ε -phase in the surface layer.

Temperature dependences for the coefficient of friction are shown in Figure 7.

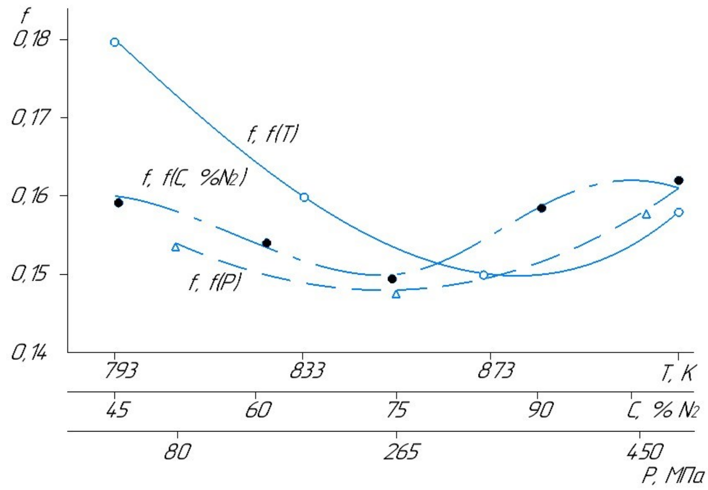

Figure 7. Dependence of friction coefficient of steel 37Cr4 in an acidic environment on the parameters of ionic nitriding (wear test mode: sliding speed v = 1m/s, pressure P= 4 MPa): 1- I=f(T);2- I=f(C,% N2); 3- I=f(P).

From Figure 7 it follows that the lowest coefficient of friction corresponds to a nitriding temperature of 873 K, a nitrogen content of 75% and a medium pressure of 265 MPa.

It is known that tightly packed hexagonal lattices tend to be oriented so that the base (hexagonal) plane is parallel to the direction of sliding. At the same time, the bonding forces between adjacent hexagonal base planes are relatively small, which reduces the coefficient of friction and prevents the occurrence of setting processes. Increasing the nitrogen content in the gaseous medium leads to a decrease in the content of Fe3N nitride in the surface layer and, accordingly, to increase the resistance to corrosion and mechanical wear of nitrided surfaces.

Further, experimental studies were carried out on the influence of the quantitative and qualitative content of sucrose in the medium solution on the tribological properties of 37Cr4 steel.

Many sugar intermediates (diffusion, sulfated, saturated, defecated juices, syrups, massecuite, molasses) contain significant amounts of sucrose. To develop sound recommendations for the choice of materials that work in these environments, it is necessary to determine the effect of sucrose on the corrosion and mechanical wear resistance of nitrided steels. Listed in table 4 data show that the presence of sucrose in solutions increases the stability and reduces the coefficient of friction.

Table 4. Influence of sucrose on corrosion-mechanical wear resistance of 37Cr4 steel (v = 1 m / s, P = 4 MPa).

Tribological parameters	Distilled water	Distilled water+ 15% sucrose	Citric acid buffer solution	Citric acid buffer solution+15% sucrose
Wear intensity, *I*, μm / km	12/1.4	2.8/0.8	8.0/2.6	5.3/1.8
Coefficient of friction, *f*	0.19/0.15	0.16/0.14	0.18/0.15	0.15/0.14

The numerator is without hardening, the denominator is nitriding (843 K, 75% N2 + 25% Ar, 265 Pa, 4 h).

The increase in the strength of materials in sucrose solutions is due to a decrease in their electrical conductivity, as well as high lubricating properties of sucrose. Thus, the addition of 15% sucrose increases the stability of improved and nitrided steel Cr37 in distilled water, respectively, 1.8 and 4.3 times, and in a buffer solution of citric acid in 1.4 and 1.5 times.

3. CONCLUSIONS

1. The obtained dependences of the amount of wear and the coefficient of friction of nitrided and normalized steels when working in an acidic environment on the working pressure and sliding speed. It was established that surfaces strengthened by nitriding have better tribological characteristics compared to non-strengthened ones. Recommendations for choosing a range of operating pressures and speeds to ensure the re-quired wear-resistant and anti-friction indicators are defined.
2. It was experimentally determined that the temperature on the friction surfaces for the nitrided surface is significantly lower than on the surface after normalization due to the higher heat capacity of iron nitrides compared to iron.
3. The optimal ranges of ion nitriding parameters (temperature, nitrogen content, pressure of the environment) are established, which ensure the best tribological characteristics of steels when working in an acidic environment.
4. It was established that the increase in stability and decrease in the coefficient of friction of steels nitrided in the glow discharge during operation in sucrose solutions occurs due to a decrease in their electrical conductivity, as well as the high lubricating properties of sucrose.

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