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Investigation of corrosion and wear resistance of steels nitrided in a glow discharge in distilled water

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Abstract

The article is devoted to the study of corrosion resistance and wear resistance (sliding friction) of unhardened and glow discharge nitrided (ion or ion-plasma nitriding) structural steels 20, 45, 45X and 38X2MЮA in distilled water. The influence of temperature (T = 793 – 873 K), the composition of the saturating mixture (nitrogen N_2, argon Ar and propane C3H8) and its pressure (P = 80 – 450 Pa), the duration of the process on the structure and phase composition of the nitrided layers was studied (carried out using metallographic and X-ray structural analyses). A comparison of the physical and mechanical characteristics of the surface layer of unhardened and nitrided steels before and after the tests was carried out and it was concluded that nitrided steels have an increased service life due to greater hardness, corrosion resistance and wear resistance. It is recommended to increase the corrosion resistance of the studied steels in distilled water, to carry out their ionic nitriding in a nitrogen-containing atmosphere, and to increase wear resistance - in a carbon-containing atmosphere (carbonitriding).

Key words: structural steels, physical and mechanical properties, ionic nitriding, carbonitriding, distilled water. corrosion, wear.

Objective

The influence of ionic nitriding (IN) and carbonitriding on the corrosion resistance and wear resistance of structural steels 20, 45, 45X, 38X2MЮA in distilled water.

Introduction and formulation of research objectives.

Iron and alloys and its bases are the most widespread metal structural materials. Therefore, the specific features of their corrosion and wear are of primary importance for mechanical engineering.

Description of the research methodology. Materials

For the researched materials (steels 20, 45, 45X and 38X2MЮA), the following heat treatment was applied [1]: normalization (steels 20 and 45), improvement (steels 45X and 38X2MЮA). Samples for testing were made from bars of one melt. The chemical composition was analyzed on a DFS-51 quantometer. The application of selected structural steels can be made more efficient by glow discharge nitriding (IN). But the mechanism of its influence on corrosion resistance and wear resistance in distilled water has not been studied yet. This leads to conflicting results of evaluating this method of chemical-thermal treatment (CTT) on the performance of friction units operating in this environment.

Analysis of recent research and publications. Corrosion of steels in water

It is known that the corrosion of steel in water is mainly controlled by the cathodic reaction, that is, by the delivery of oxygen. The pH of water and its ability to form protective sediments are also important [2]. Small additives in low-alloy steels do not noticeably affect the rate of general corrosion in water [3]. Thus, according to [4], the corrosion rate of steel in distilled water is 0.256 g/m2*h. And, according to [5], the corrosion rate of carbon steel when mixing chemically desalinated water at a speed of 0.5 - 1.0 m/s is (0.17 - 0.24) g/m2*h.

Distilled water (according to DEST 6709-72) still contains salts that provide low electrical conductivity. But this value is enough for water to become a weak electrolyte and provide the possibility of electrochemical corrosion. Another cause of corrosion is gas corrosion, that is, the combination of oxygen atoms dissolved in water with iron atoms. Distilled water is devoid of salts, but it contains all atmospheric gases, of which oxygen is of primary importance [5]. And according to [6], metals of increased thermodynamic instability (Fe, etc.) can corrode even in a neutral water environment in the absence of dissolved oxygen.

Dissolved oxygen in water affects steel corrosion because oxygen reduction is the predominant cathodic reaction that controls the anodic reaction. Thus, all factors that change the content of dissolved oxygen affect metal corrosion. Mixing the solution intensifies the transport of dissolved oxygen, increasing the rate of corrosion [7].

The influence of the speed of movement of the neutral electrolyte on the electrochemical corrosion of metals is complex [8]: it facilitates the diffusion of oxygen.

To date, a large number of methods of protecting metal from corrosion have been developed, which include the use of coatings, HTO, plasma treatment, rational selection of the composition and structural state of alloys, etc.

Wear of steels in water

According to [9], corrosion of low-alloy steels in the presence of moisture is accompanied by the formation of iron oxides. These deposits can play a negative role as an abrasive, increasing wear. The authors of [10] investigated the wear resistance of steel 45 (normalization and hardening) and steel 45X (electrolytic nitriding) when rubbing on foam padding in distilled water. It was established that the wear resistance of normalized steel 45 is determined by the adhesive strength of the films formed on its pearlitic and ferritic components. And hardened steel 45 has a uniform martensitic structure and increased anti-corrosion properties. During friction, oxide films softer than the base are formed on it, which flow over the friction surface without the formation of cracks.

Regarding studies of corrosion resistance and wear resistance of non-strengthened and nitrided test steels in distilled water, they were not found.

Nitriding in a glowing discharge

Experimental studies were carried out on the UATR-63 glow discharge nitriding unit, designed and manufactured at the Podilskyi Scientific Physics and Technology Center of the Khmelnytskyi National University.

The nitrogenized layer is characterized by the following main parameters: thickness, surface and depth hardness, phase composition, structure, etc. They depend on the grade of steel and the technological parameters of the process: temperature (T), the composition of the medium in % (nitrogen N_2, argon Ar and propane C_3H_8), the pressure of the gas medium (P) and the duration (D) of diffusion saturation (in all studies, the duration of ionic nitriding (IN) or carbonitriding was □ = 4 hours). These factors under the conditions of ionic nitriding are practically independent [11].

Thus, as a result of IN, a high complex of properties of the strengthened layer (hardness, plasticity, etc.) is ensured, which affects the strength characteristics and operational characteristics of structural elements.

Structure

The study of structural strength properties of nitrided layers was carried out with the help of metallographic and X-ray structural analysis, which made it possible to establish correlations between the studied properties and the observed macro-submicrostructure of the hardened layer.

The sub-microscopic structure of the nitride zone (size, number and nature of discharges) was studied on a SEM-200 scanning electron microscope. X-ray structural phase analysis of the surface layers of the samples was carried out on a DR0N-3 X-ray diffractometer using chromium α-radiation. A semi-quantitative analysis was performed based on the ratio of the intensities of the (101) ε-phase, (111) γ'-phase, and (110) α-phase lines. The calculation of the periods a and c of the ε-phase was carried out by the position of the maxima (110) and (101), the content of the introduced element was determined by the ratio c/a.

The cross-sectional structure of the diffusion layer and the distribution of microhardness over the thickness of the layer were studied on etched sections using an MIM-10 optical microscope. The microhardness of the surface was measured separately and the fragility of the strengthened layer was assessed according to the VINM four-point scale. To measure the microhardness of all studied materials (performed on a microhardness tester PMT-3 in accordance with DEST 2999-75; a Vickers diamond tip was used) in order to obtain comparable data, a load of 0.98 N was adopted, which provides the smallest relative measurement error. The value of microhardness was recorded both on the surface and at a certain distance from it deep into the sample. The thickness of the nitride zone was measured at ten different locations. The uniformity of indicators was judged by the scatter area.

Corrosion research

Corrosion and electrochemical properties of materials were studied by gravimetric and potentiostatic methods by taking polarization curves, as well as changes in potential over time. Samples for testing (Fig. 2.4; working surface area 1 cm^2) were made on a lathe from one unit from one rod, subjected to heat treatment, polished on a flat grinding machine and refined on an M20 sandpaper. After grinding, the side surface of the samples was insulated with 88NP glue, and the end surface was cleaned with benzene, degreased with acetone, and dried with filter paper. The samples were placed in a desiccator and kept for at least a day.

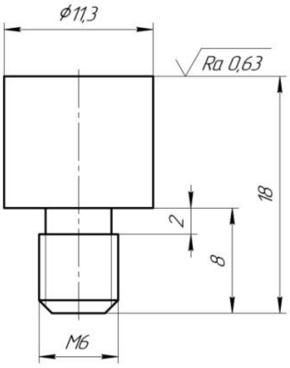


Fig. 1. Sample for studying the electrochemical properties of materials

The kinetics of electrochemical processes that took place in the sample (working electrode) - electrolyte system were studied using a P-5827M potentiostat in a YASE-2 three-electrode cell. The solutions in these experiments were mixed with a magnetic stirrer. Before taking the polarization curves, the samples were kept in the test environment for a time sufficient to establish the rate of change of the potential of the working electrode by no more than 10 mV in 30 min before the start of measurements. Potentials were recorded: immediately after immersion, after 0.5, 1, 2, 3, 4, 5, 30, 60, 90, 120 min. The last measurement was carried out after 96 hours.

Polarization curves were recorded in potentiodynamic mode. The speed of the sweep (change in the potential of the working electrode over time) in all experiments was constant (with a multiplier of the potential speed equal to one). A silver chloride electrode of the EVL-1M1 type, immersed in the electrolyte by 10...20 mm, served as a standard. The auxiliary platinum electrode was placed until the contact was completely immersed in the working solution. The current between the working and auxiliary electrodes was measured with a M2020 millivolt ampmeter.

Corrosion current was determined by extrapolation of rectilinear (Tafel) sections of polarization curves in the region of small overvoltages. According to the corrosion current, taking into account the fact that iron passes into the solution in the form of divalent ions, according to Faraday's law, the corrosion loss of the mass of the samples was determined.

For a deeper study of the dissolution process of materials, the corrosion products in the solution were studied. Qualitative determination of divalent iron ions was carried out using a solution of potassium hexacyanoferrate, trivalent - using a solution of potassium rhodate.

The influence of temperature on the rate of corrosion processes was studied. The temperature of the electrolyte was measured directly near the sample.

To compare the data of electrochemical measurements, the corrosion resistance was studied by means of an open glass with the samples fully immersed in the solution, the amount of which was 4*106 g mm3 per 100 mm^2 of the sample surface. Tests were conducted at room temperature and natural aeration for 720...1100 hours. Corrosion products were removed with a strong stream of water while wiping the sample with a glass stick with a rubber tip. Corrosion losses were determined by weighing the samples on analytical scales VLR-200g before and after the tests, the mass index of corrosion was determined by the formula:

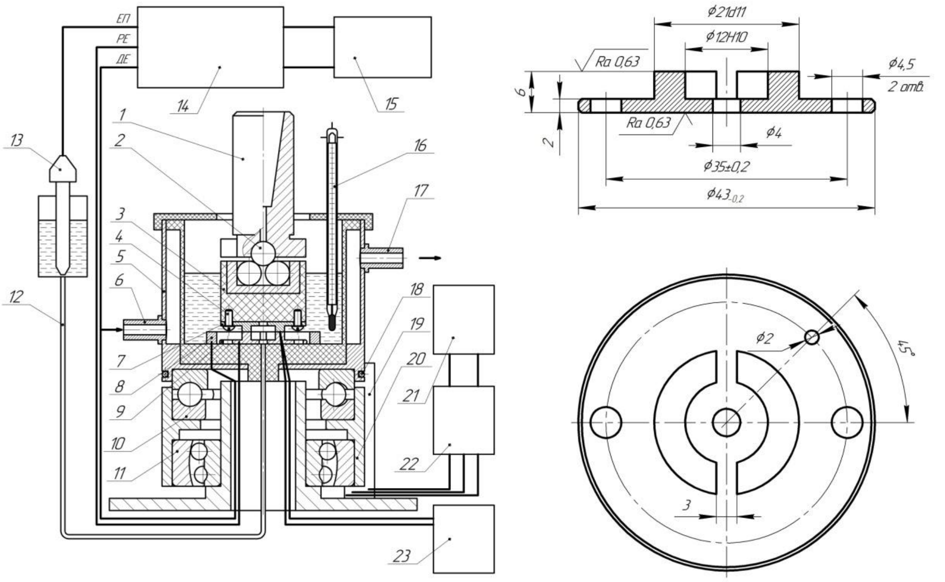
$$K = (m_0 - m)/S\tau$$

where $(m_0 - m)$ – loss or gain of mass, g;
S – sample area, m^2;
τ– duration of tests, hours.

Corrosion-mechanical wear (CMW)

For a comprehensive study of the CMW of materials in a wide range of external loads and working environments of different chemical composition and properties, a laboratory installation of end friction has been designed, which makes it possible to study the change in electrode potential, frictional and wear characteristics, temperature on the friction surface of the sample, friction characteristics depending on the electrode potential system, take polarization cathode and anode curves [11].

The installation (Fig. 2, a) consists of a rigid bed on the basis of a 2M112 machine, a working chamber, a spindle assembly with 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 (Fig. 2, b).



a b

Fig. 2. Scheme of the installation (a) and sample (b) for the study of CMW materials

The rotational movement and vertical force from the conical shank I (Fig. 2.2, a) is transmitted through four balls 2 to the body 3, to which the upper sample 7 is attached with the help of two screws 4. The lower sample 9 is attached with two screws to the electrochemical cell 5 - a three-electrode system , in which the working electrode (WE) is a friction pair, and the auxiliary electrode (AE) is a ring electrode 8 made of 12Х1Ш9Т steel, which forms a polarization chain with WE.

The cell is installed on the thrust bearing 10 and, together with the housing 20, on the double-row spherical bearing 11. The angular fixation of the cell is ensured by the cable 18 attached to the finger 19. To isolate the samples from the installation, the cell 5 and the housing 4 are made of kaprolon, and the non-working surfaces of the samples together with the fastening screws are covered with 88NP glue.

During the friction of metals in electrolytes, the flow of adsorption, diffusion and other processes on their surfaces is determined by the electrode potential of the system. Being a fundamental energy characteristic of the double layer, the electrode potential shows in which stage – active or passive dissolution – the steel is and can affect its friction and wear in electrically conductive environments. The kinetics of electrochemical processes of materials in static conditions, during mixing and friction were studied by the potentiodynamic method using the P-5827M potentiostat (14), and the polarization curves were recorded with a self-writing potentiometer PDP4-002 (13 - Fig. 2, a). Static and stirring tests were performed with a distance between the working surfaces of the samples of 3 mm. The electrode potentials were measured relative to the silver chloride reference electrode EVL-1M1 (13) in a saturated solution of KCl, which is fed into the friction zone using a polymer tube 12 through an axial hole in the lower sample. Thoroughly cleaned, degreased and aged for at least a day in a desiccator, the samples were immersed in a solution with a specified pH value. The volume of the solution was 2 * 105 mm^3, and its temperature was controlled by an electric contact thermometer 16 and maintained by a thermostat. Through the fittings 6 and 17 (Fig. 2, a), thermostated water was supplied and discharged. The average surface temperature of the sample during the tests was measured using a self-recording electronic potentiometer KSP-2 (23) at a distance of 1 mm from the friction surface (Fig. 2, a). To obtain reliable results, an artificial thermocouple with a hot permanent junction (round head) of chromel-alumel thermoelectrodes with a diameter of 0.5 mm was used. The wear resistance of the friction pair was evaluated by friction force (friction moment) and linear wear of the samples. The force of friction was measured by the tensometric method. The signal generated by the deformation of the 19 strain gauges glued to the finger was transmitted through the strain amplifier 8АНЧ-7М (22) to the KSP-2 potentiometer (21). The established coefficient of friction was calculated on the basis of the diagram of the moment of friction, which is recorded by a self-recording device; linear wear of the samples was determined by an indicator of the hour type MKM with a division price of I μm.

The lower sample made of U8A steel, hardened to (61 – 63) HRCe, had two grooves 3 mm wide on the working surface, which ensured free access of the medium to the friction surface. The upper one was a sample from the researched material. The initial roughness of the working surfaces of the samples was Ra = 0.63. The heating of the samples at the point of contact did not exceed 393 K, which excluded the possibility of significant structural changes in the material. Experiments were conducted at sliding speeds of 0.05, 0.6, and 1 m/s in the pressure range of 1–48 MPa.

Presentation of the main material and obtained scientific results

It was established (Table 1) that the corrosion rate of unreinforced steels is approximately the same. Since the anodes and cathodes are located close to each other in the low-conductivity medium under investigation, the OH- ions formed at the cathode are always close to the Fe^{2}+ ions formed at the anode. As a result, a film of $Fe(OH)_2$ appears, which is adjacent to the metal surface and is an effective diffusion barrier. Increasing the speed of movement of distilled water improves the access of oxygen to the surface and contributes to the removal of corrosion products, thereby increasing the corrosion rate (Table 1 - the obtained data coincide with those given in [5].

Table 1

Electrode potentials and corrosion rate of the investigated materials in static conditions (numerator) and when stirring at a circular speed of 0.05 m/s (denominator)

Steel	Electrode potential, mV						Corrosion rate, g/m^2*h	
	After 24 hours exposure			After 96 hours exposure				
	Without hardening	Nitriding*	Nitriding**	Without hardening	Nitriding*	Nitriding**	Without hardening	Nitriding*
20	-578 -386	+42 +37	+37 +35	-584 -390	+78 +77	+48 +44	0.218 0.35	0.0003 0.0005
45	-572 -378	+76 +50	+60 +56	-582 -390	+86 +85	+65 +60	0.210 0.35	0.0003 0.0005
45X	-525 -362	+102 +102	+80 +78	-538 -374	+118 +118	+84 +84	0.151 0.28	0.0003 0.0005
38Kh2MYuA	-520 -355	+122 +122	+85 +82	-535 -363	+131 +130	+88 +88	0.112 0.28	0.0002 0.0004

*one-stage (793 K; 75% N2 + 25% Ar; 265 Pa);

**combined (793 K; 75% N_2 + 25% Ar – 3 hours and 90% N_2 + 10% C_3H_8 – 1 hour, 265 Pa) – carbonitriding.

IN of the studied steels significantly increases their corrosion resistance (Fig. 3), which, in particular, is indicated by a significant improvement of the electrode potential of the surface (Table 1, Fig. 4).

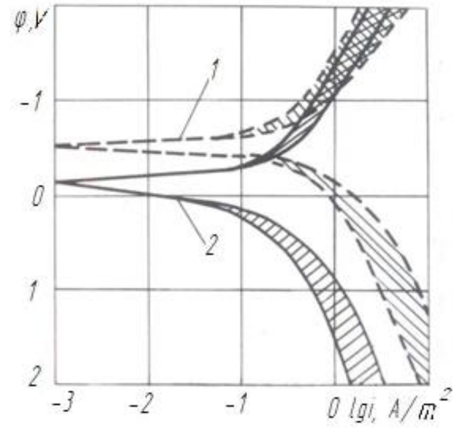


Fig. 3. Polarization curves of unhardened (1) and nitrided (2) at temperature T = 793 K in the single-stage mode of the studied steels

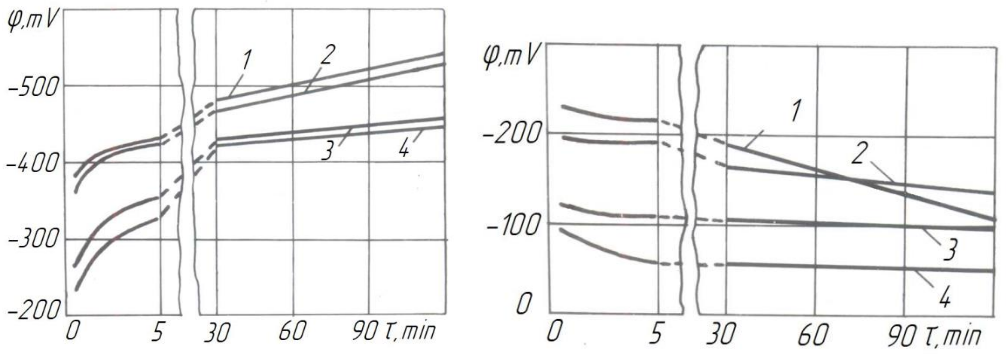


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Fig. 4. Curves change over timeτelectrode potentialφunhardened (a) and nitrided (b) at T = 793 K in the one-stage regime of steels 20 (1), 45 (2), 45X (3), 38Kh2MYuA (4)

X-ray structural analysis established that regardless of the composition of the gas atmosphere (table PP.2), ε-$Fe_{2-3}N$, γ′-Fe_4N and α-phases are formed in the surface layer. At the same time, the phase composition and ratio of phase structures in the nitrided layer can be adjusted by changing the nitriding parameters [1].

From Fig. 5, as well as given in table. 1 and 2 of the data shows that with an increase in the content of the ε-phase in the surface layer of materials, their corrosion resistance increases. Therefore, to reduce the corrosion rate of materials in distilled water, a nitrogen-containing atmosphere is better than a carbon-containing one.

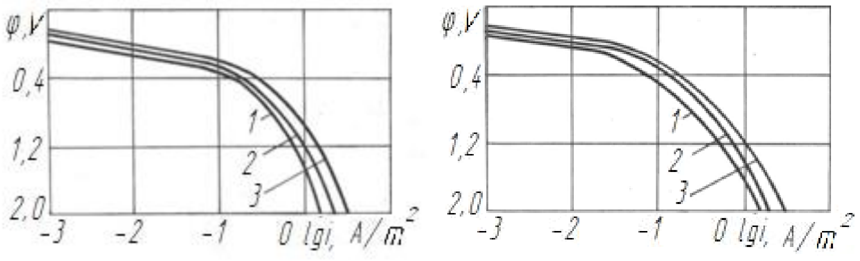

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Fig. 5. Anodic polarization curves in distilled water in static conditions: a) nitrided steel 45X at temperatures of 793 (I), 833 (2) and 873 (3) K, pressure 265 Pa, in an atmosphere of 100% N_2; b) nitrided steels 38Kh2MYuA (I), 45X (2), 45 and 20 (3) at parameters of 843 K, 75% N_2 + 25% Ar, 265 Pa

Table 2

Crystal lattice parameters (a and c), nitrogen concentration in the surface layer (Ka) and phase composition of nitrided (843 K; 75% N_2 + 25% Ar; 265 Pa) steels

Indicators	Steel 20	Steel 45	Steel 45X	Steel 45X*	Steel 38Kh2MYuA
a, кX	2.775	2.757	2.761	2.762	2.764
c, кX	4.443	4.441	4.439	4.447	4.436
c/a	1.613	1.611	1.608	1.611	1.605
K_a, %	7.3	7.6	8.3	7.6	8.7
ε - $Fe_{2-3}N$, %	19	21	31	20	45
γ′– Fe_4N, %	56	62	55	9	55
α - Fe, %	25	17	14	71	–

*843 K; 75% N_2 + 25% Ar – 3 hours and 90% N_2 + 10% C_3H_8– 1 hour; 265 Pa

The established regularities indicate that with a decrease in the nitriding temperature, the corrosion resistance of materials in distilled water increases due to an increase in the nitrogen content in the diffusion layer. At the same time, the fragility of the surface area increases, which reduces wear resistance. Therefore, in order to increase the wear resistance of the studied steels in distilled water, their nitriding was carried out at T = 793 K, which helps to increase the saturation of the diffusion layer with nitrogen (Table 3), according to the combined mode of saturation (75% N_2 + 25% argon, 3 hours and 90 % N_2 and 10% C_3H_8, 1 hour; 265 Pa), which increases the plasticity of the surface zones.

Table 3

Dependence of crystal lattice parameters (a and c), nitrogen concentration in the surface layer (Ka) and phase composition of the diffusion layer of steel 45X on IN regimes for 4 h.

Indicators	Temperature, K*			N_2 content, %**					Pressure, Pa***		
	793	833	873	45	60	75	90	100	80	265	450
a, кX	2.775	2.768	2.757	2.759	2.761	2.762	2.765	2.768	2.762	2.762	2.763
c, кX	4.413	4.428	4,447	4.441	4.440	4.439	4.433	4.428	4.440	4.439	4.438
c/a	1.590	1.600	1.613	1.610	1.608	1.607	1.603	1.600	1.608	1.607	1.606
K_a, %	10.8	9.0	7.3	7,6	8.1	8.4	8.8	9.0	8.4	8.4	8.5
ε - $Fe_{2-3}N$, %	63	42	20	22	27	32	38	42	29	32	35
γ'– Fe_4N, %	30	46	59	66	61	56	50	46	59	56	53
α - Fe, %	7	12	21	12	12	12	12	12	12	12	12

*100% N_2; 265 Pa;

**833 К; 265 Pa;

***833 К;75%N_2+ 25% Ar

When wearing materials in distilled water, it is characteristic that it is both a corrosive agent and a friction lubricant. The presence of a low-viscosity aggressive medium causes the appearance of boundary and semi-fluid friction. However, for the accepted test conditions (V = 1 m/s, P = 4 MPa), the surfaces of unreinforced materials seize (Fig. 6). At the same time, the friction coefficient reaches a value of 0.19. Analyzing presented in the table. 4 data, it can be seen that when rubbing in distilled water, the use of unhardened alloyed steels does not give a significant advantage over unalloyed ones.

Table 4

Wear intensity (µm/km) of steels (V = I m/s, P = 4 MPa)

Steel 20	Steel 45	Steel 45X	Steel 38Kh2MYuA
$\frac{14}{1.7}$	$\frac{13}{1.5}$	$\frac{12}{1.2}$	$\frac{12}{1.0}$

Numerator – without hardening, denominator – combined nitriding (carbonitriding)

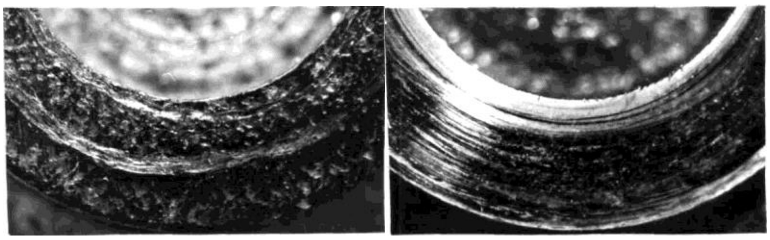

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Fig. 6. Friction surfaces of non-hardened (a) and nitrided according to the combined mode (b) 45X steel after CMW (x7.5)

Research has also shown (Table 4) that nitrided steels are characterized by significantly higher wear resistance compared to normalized and improved steels. There is no sticking of nitrided friction surfaces, the friction coefficient is within 0.15. Obviously, this is explained by the increased corrosion resistance of the nitrided layer, the presence of compressive stresses in it [1], as well as the intermetallic structure of Fe_3N nitride, which has a low tendency to cold welding and good antifriction properties. Steel 38Kh2MYuA, which has the highest hardness, has the maximum wear resistance. For unhardened steels, the intensity of wear is directly proportional to the friction path, and for nitrided steels 45X and 38Kh2MYuA, it increases as the diffusion layer wears. Wear of the nitride layer of nitrided steels 20, 45 exposes a zone with reduced hardness (Fig. 7), which leads to an increase in the intensity of wear.

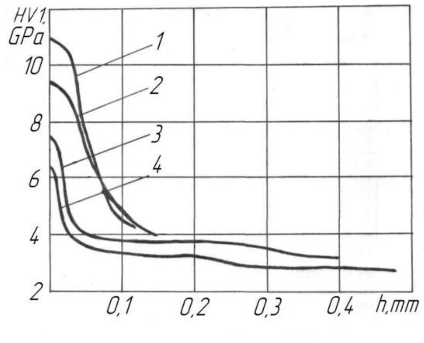

Fig. 7. Distribution curves of microhardness HV1 along the thickness h of the diffusion layer of nitrided steels 38Kh2MYuA (1), 45X (2), 45 (3), 20 (4)

Conclusion

1. X-ray structural analysis established that the phase composition and the ratio of structural components of the strengthened layer can be adjusted by changing the parameters of the IN.

2. With an increase in the content of the ε-phase in the surface layer of the studied steels, their corrosion resistance in distilled water increases. Therefore, from this point of view, a nitrogen-containing atmosphere is better than a carbon-containing one.

3. When rubbing in distilled water, the use of unhardened alloyed steels does not give a significant advantage over unalloyed ones.

4. To increase the wear resistance of nitrided test steels in distilled water, a carbon-containing atmosphere is better than a nitrogen-containing one.

5. In order to determine the maximum wear resistance of steels, further research should be aimed at clarifying the propane content at IN.

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Рудик О.Ю., Каплун П.В., Голенко К.Е., Гончар В.А., ПобережниЙ М.М. Дослідження корозійної та зносостійкості азотованих у тліючому розряді сталей у дистильованій воді

Стаття присвячена дослідженню корозійної стійкості та зносостійкості (тертя ковзання) незагартованих і тліючим розрядом азотованих (іонним або іонно-плазмовим азотуванням) конструкційних сталей 20, 45, 45Х і 38Х2МЮА в дистильованій воді. Вплив температури (Т = 793 – 873 К), складу насиченої суміші (азот N2, аргон Ar і пропан C3H8) та її тиску (Р = 80 – 450 Па), тривалості процесу на структуру та досліджено фазовий склад азотованих шарів (здійснено методами металографічного та рентгеноструктурного аналізів). Проведено порівняння фізико-механічних характеристик поверхневого шару незагартованої та азотованої сталі до та після випробувань і зроблено висновок, що азотована сталь має підвищений термін служби за рахунок більшої твердості, корозійної стійкості та зносостійкості. Рекомендується для підвищення корозійної стійкості досліджуваних сталей у дистильованій воді проводити їх іонне азотування в азотовмісній атмосфері, а для підвищення зносостійкості – у вуглецевмісній атмосфері (карбонітрування).

Ключові слова: конструкційні сталі, фізико-механічні властивості, іонне азотування, карбонітрування, дистильована вода. корозія, знос.