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Investigation of changes in phase composition and tribological properties of 65G steel during electrolyte-plasma hardening

This paper presents the results of studies of phase composition and tribological properties of 65G steel, before and after electrolytic-plasma hardening at different regimes. The technology of electrolyte-plasma hardening and laboratory installation for implementation of electrolyte-plasma hardening are described. It was found that after electroplasma hardening a modified layer consisting of α'-phase (martensite) and cementite M_3C is formed. The developed technological process of hardening of a part made of 65G steel makes it possible to obtain layers on the surface of the part that provide an increase in wear resistance by 2 times and in resistance to abrasive wear by 1.7 times. The carried out investigations have shown perspective and expediency of application of the developed method to increase operational properties of parts working in conditions of friction and wear. This technology can be used to increase the service life of working elements of agricultural machinery.

Keywords: hardening; phase composition; plasma-electrolyte hardening; wear resistance.

Introduction

The durability of parts depends not only on the material properties determined by the manufacturing technology and volume hardening, but also to a large extent on the surface properties. Its role in ensuring the operational properties of products is constantly increasing, which has contributed, along with extensive use of traditional methods of chemical-thermal treatment, to the emergence and development of a new direction — surface engineering by methods of energy and physical-chemical effects. The implementation of this concept in the choice of material will improve the performance properties of parts, and in some cases reduce the consumption of expensive materials. Thus, recently, due to the use of protective coatings and surface hardening, more and more low-alloy structural and tool steels are used and produced, which allowed reducing the cost of expensive high-alloy steels and alloys. At the same time, an important role in the application of protective coatings and surface hardening is the use of resource-saving technologies that help to reduce resource and energy costs and increase labor productivity [1, 2].

Recently, the research on electrolyte-plasma hardening of materials has been conducted quite intensively. As a result, various technologies for surface modification of metals and alloys based on the electrolyte-plasma method have been developed: oxidation [3], polishing [4], diffusion saturation with nitrogen [5], carbon [6, 7], boron [8], multicomponent saturation [9, 10] and surface hardening [11]. Among them, electrolyte-plasma hardening (surface hardening) is of specific interest [11, 12].

The surface hardening process by surface hardening was usually performed by laser beam, electron beam and plasma beam [13, 14]. Compared to these hardening processes, electrolyte-plasma hardening is a simple and inexpensive method. All methods of surface hardening are usually used to increase the surface hardness of steels. However, experience shows that surface hardening under certain conditions forms a fine-dispersed structure that increases the wear resistance of steel depending on its alloying. In addition, electrolyte-plasma hardening (surface hardening) differs favorably from plasma hardening processes (surface hardening) due to the high cooling rate and a smaller degree of oxide layer formation. Since, plasma discharges are formed between the surface of the metal and the electrolyte, and the cooling process takes place in the flowing electrolyte. Electric circuit closes between the electrodes through the electrolyte (aqueous salt solution). The transformation of electrical energy into heat energy occurs mainly in the layer adjacent to the product. As a result of heating, this layer transitions to a vapor-gas state, and micro-arcs are excited in it under the influence of the applied voltage. The power density reaches up to 3×10^3 W/cm^2 [15].

The technology makes it possible to change the heating and cooling rate and the thickness of the hardened layer within a wide range. By adjusting the temperature-rate regimes of plasma surface heating and cooling, as well as the use of various electrolytes, high values of mechanical and tribological characteristics of the surface layer of steels can be obtained.

In connection with the above, the purpose of this work is to study the effect of electrolyte-plasma hardening on the phase composition and tribological properties of 65G steel.

Materials, equipment and methods of experiments.

The object of the study was selected constructional 65G steel, which is used for the manufacture of working elements of tillage machines.

The chemical composition of 65G steel is presented in Table 1.

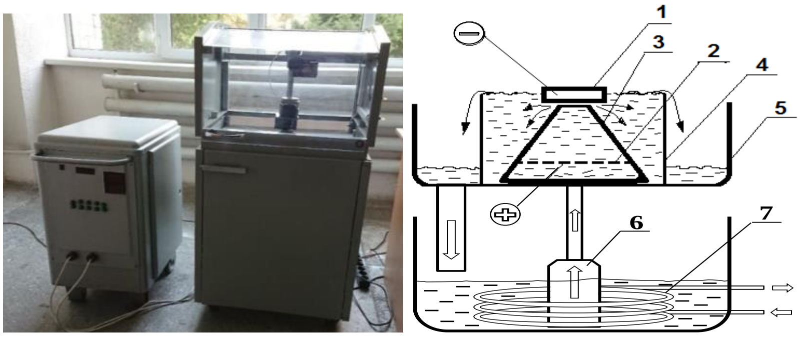
T a b l e 1

Chemical composition in % of steel 65G (GOST 14959 — 79)

Steel grade	**C**	**Si**	**Mn**	**Ni**	**S**	**P**	**Cr**	**Cu**
65G	0.62 — 0.7	0.17 — 0.37	0.9 — 1.2	up to 0.25	up to 0.035	up to 0.035	up to 0.25	up to 0.2

Electrolytic-plasma hardening (EPH) of 65G steel samples was carried out on the laboratory unit in the Research Center “Surface Engineering and Tribology” of S. Amanzholov EKU. The general view and the scheme of installation of electrolyte-plasma processing are shown in Figure 1. The installation structurally consists of a power supply and a chamber for electrolyte-plasma treatment of materials.

EPH of steel samples is carried out as follows. Before starting work, the working bath is filled with electrolyte. Then the electrolyte is pumped into the electrolytic cell by means of a pump installed at the bottom of the working bath. The electrolyte flows out through the opening of the cone-shaped baffle in the form of a jet and fills the electrolytic cell. The electrolyte is then discharged through the edge of the electrolytic cell into the tray and then back into the working bath. Thus, the electrolyte is in circulating regimes. The feed rate of the electrolyte (flow rate) is 4-7 l/min. Feeding rate of cooling flow water into the heat exchanger is 3-6 l/min. The accepted parameters of cooling electrolyte allow maintaining the temperature within 40-70°C when heating the samples to the temperature of 800-900°C. Using the device for fixing the processed product, the processed product is dipped into the electrolyte so that the treated zone of the product was at a distance of 2-3 mm from the hole of the cone-shaped partition. The electrolyte jet is directed through the opening of the cone-shaped partition which is 10-15 mm lower than the height of the electrolytic cell. Then the anode is connected to the positive pole of the power supply, and the processed product — cathode to its negative pole. For heating to the hardening temperature 320 V voltage is applied between the electrodes and the current density is 25-30 A/cm^2. At these voltages an intensively glowing plasma layer is formed in the pre-cathode area and the product is heated at a rate of 450-500 °C/s. In this case an anomalous arc discharge is formed between the electrodes, due to which the workpiece is quickly heated [1, 8].



1 — sample to be treated (cathode), 2 — stainless steel anode with holes, 3 — cone-shaped partition, 4 — working chamber — bath with electrolyte, 5 — pan, 6 — pump, 7 — heat exchanger

Figure 1. General view and diagram of the electrolytic plasma treatment installation

Samples of 65G steel were treated at different regimes of EPH. The EPH regimes are shown in Table 2. The EPH was carried out by alternating high (320 V), medium (200 V) and low (50 V) voltages, as well as by cyclic exposure. An aqueous solution containing 15 percent sodium carbonate was used as the electrolyte. Distilled water was used to prepare the electrolyte.

Table 2

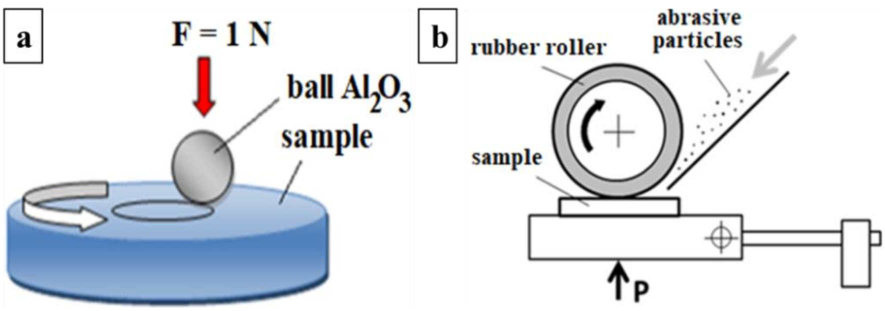
Regimes of thermocyclic electrolyte-plasma treatment of 65G steel sample

No.	Sample	Cycle 1			Cycle 2			Cycle 3			Cycle 4		
		320 V	200 V	50 V	320 V	200 V	50 V	320 V	200 V	50 V	320 V	200 V	50 V
	10-65G	1 s	-	1 s	1 s	-	-	-	-	-	-	-	-
	11-65G	2s	-	-	-	-	-	-	-	-	-	-	-
	17-65G	1s	3s	7s	1s	1s	7s	1s	-	-	-	-	-
	18-65G	1s	-	1s	1s	-	7s	1s	-	12s	1s	-	-

X-ray studies of steel samples were carried out using the well-known methods of X-ray structural analysis on X'PertPRO diffractometers. Diffractograms were taken using CuKα-radiation (λ=2.2897 A^0) at 40 kV. The diffractograms were manually transcribed using standard techniques and the PDF-4 database, and quantitative analysis was performed using Powder Cell software.

Tribological sliding friction tests were performed on a TRB^3 tribometer using the standard ball on disk technique (Figure 2a) (ASTM G 133-95 and ASTM G 99 international standards). A 6.0 mm diameter ball of certified Al_2O_3 material was used as a counterbody. Tests were conducted at a load of 1 N and a linear velocity of 2 cm/s, a wear radius of curvature of 5 mm, and a friction path of 40.1 m. Tribological characteristics of the modified layer were characterized by wear intensity and friction coefficient [12].

The samples were tested for abrasion on an experimental installation for abrasion testing in friction against non-rigidly fixed abrasive particles according to the scheme "rotating roller — flat surface" in accordance with GOST 23.208-79, which coincides with the American standard ASTM C 6568 (Fig. 2 b). To test the abrasion on the rubber wheel, the surfaces of the samples were ground and polished, as well as they were cleaned with acetone and dried. A cylindrical rubber roller pressed by the radial surface to the flat surface of the test sample with a force of 22 N was rotated at a frequency of 1 s^{-1}. The scheme of the device is shown in Figure 2b. The rate of arrival of abrasive particles between the rubber wheel and the sample, i.e. in the test zone was 41-42 g/min. As abrasive particles, electrical corundum with granularity of 200-250 μm was used.



a — according to the scheme "ball on disk", b — according to the scheme "rotating roller — plane surface"

Figure 2. Tribological tests of samples

The wear resistance of the tested treated sample was evaluated by comparing its wear with the wear of the reference sample (untreated sample). Wear was measured by weight method on analytical scales ADV-200 with accuracy to 0.0001 g. Samples were weighed every minute and tested for three minutes, the length of the whole wear was 28.8 m. Before weighing, the samples were blown with compressed air to remove any remaining sand particles on the samples. Wear resistance of the tested material was evaluated by weight loss of the samples during the test according to GOST-23.208-79.

Tests of samples on shock-abrasive wear were carried out on the experimental bench according to GOST 23.207-79. For comparative evaluation of wear resistance of 65G steel samples before and after electrolyte-plasma treatment at different regimes. Tests were carried out in the following regimes: impact energy E = 3.3 J, impact velocity υ = 1 m/s and impact frequency n = 200 min^{-1}. The scheme of the device is presented in Figure 3. The rate of abrasive particles entering the test zone was 75-80 g/min. Electro-corundum with granularity 200-250 μm was used as abrasive particles. The wear resistance of the tested samples was evaluated by comparing the wear of the hardened sample with the wear of the unhardened sample. Wear was measured by weight method on analytical scales ADV-200 with accuracy 0.0001 g. Samples were tested for five minutes. Before weighing, the samples were blown with compressed air to remove the remaining sand particles on the samples.

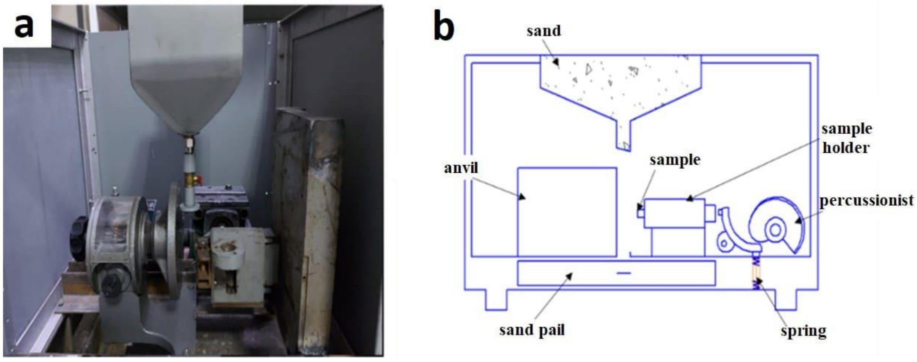


Figure 3. Experimental bench for shock-abrasive wear according to GOST 23.207-79

Bench tests of 65G steel samples on air-erosion resistance of coatings were carried out on a special bench designed and manufactured in the Research Center "Surface Engineering and Tribology" in accordance with ASTM G76 standard. The general view of the bench and the test chamber is shown in Figure 4.

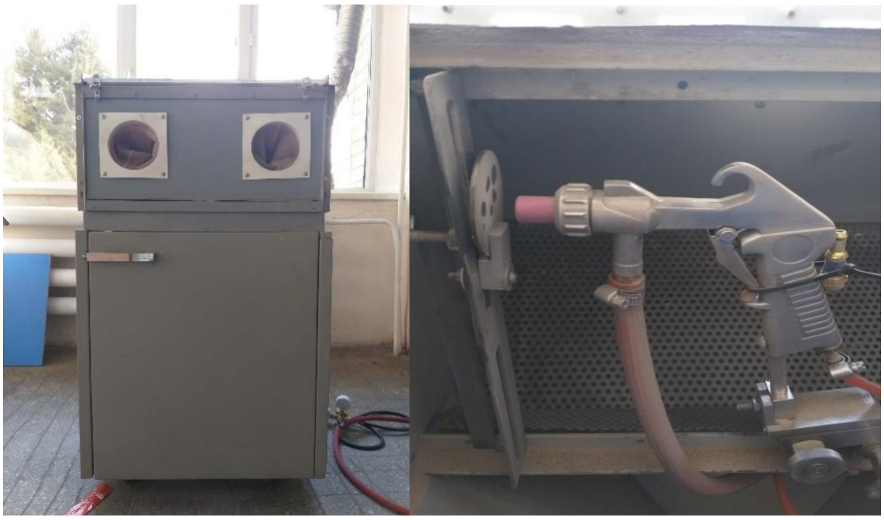


Figure 4. Bench for erosion testing

Tests on air-erosion resistance of samples were carried out as follows: air-abrasive jet is supplied to the sample from the nozzle with diameter of 5 mm. Air pressure at nozzle inlet is 0.4 MPa, distance from nozzle to sample surface is 11 mm. The duration of exposure to air-abrasive jet was 60 seconds.

Results and discussion.

Figure 5 shows the results of the tribological tests. Tribological tests were performed using Si_3N_4 counterbodies (ball diameter of 6 mm) and 100Cr6 (ball diameter of 3 mm). The amount of wear was determined by examining the profilogram of the wear tracks. The results showed that the wear volume decreased after the EPH, sample 11-65G showed a higher wear volume when the Si_3N_4 counterbody was used, while the 100Cr6 counterbody had a low wear volume compared to the initial sample. And sample 10-65G showed the lowest wear volume values compared to the other samples with both the Si_3N_4 and 100Cr6 counterbodies.

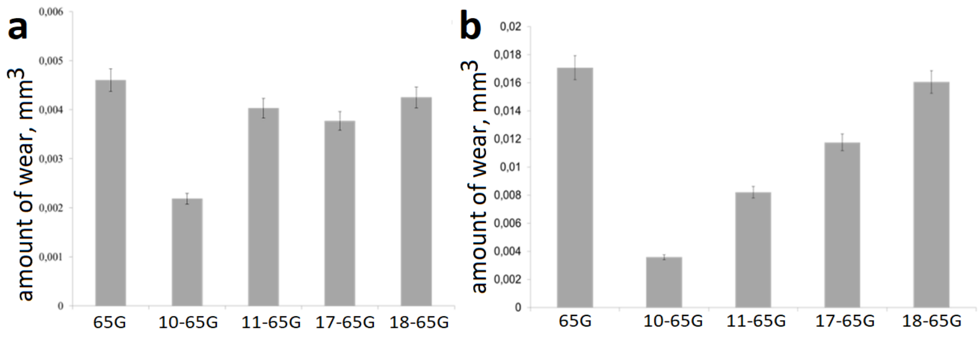


Figure 5. Data on the wear volume of samples after test with counterbodies of Si_3N_4 (a) and 100Cr6 (b)

Figure 6 shows the friction coefficient curves of 65G steel samples after wearing with counterbodies made of Si_3N_4 (ball with diameter of 6 mm) and 100Cr6 (ball with diameter of 3 mm). The test results when using Si_3N_4 counterbodies showed that the friction coefficient varies greatly depending on the EPH regime. An increase in the friction coefficient was observed in all samples except 18-65G. And when the 100Cr6 counterbody was used, an increase in the friction coefficient was observed in all samples. This may be due to the fact that the treated samples had a high roughness compared to the initial sample.

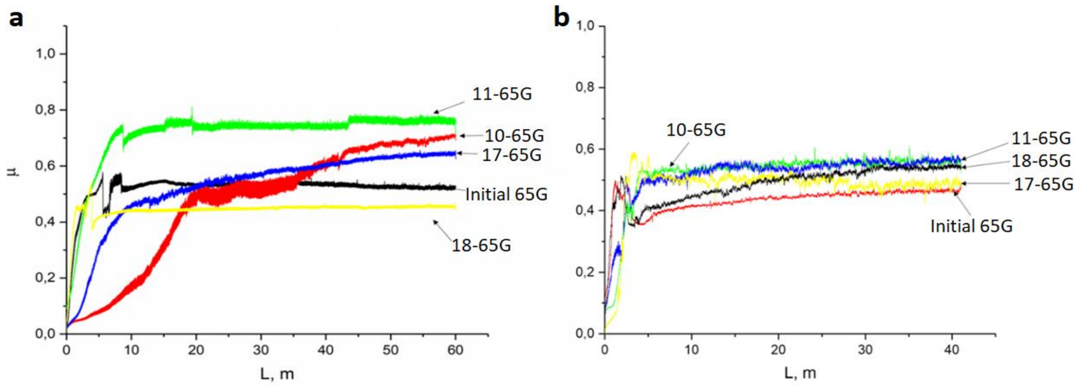


Figure 6. Friction coefficient curves of 65G steel samples at wear with counterbodies of Si_3N_4 (a) and 100Cr6 (b)

Wear tests of samples on abrasion on the special bench according to GOST 23.208-79 were carried out. Wear resistance was estimated by weight method. Weight of samples was measured by means of analytical scales Gibertini CRYSTAL 100 CE with magnetic compensation. Table 3 shows the masses of the samples before and after the tests. From the table it is seen that the weight loss of all treated samples is low compared to the initial sample. At the same time, samples 10-65G and 18-65G showed higher resistance to abrasive wear. Thus, it is possible to assert that EPH allows to increase resistance of steel 65G to abrasive wear by 1.7 times.

Table 3

Results of measuring the weight of samples before and after testing

No.	Samples	Weight before, g	Weight after, g	Weight loss, g
1	Initial sample-65G	60.084	60.0746	0.0094
2	10-65G	57.159	57.1509	0.0081
3	11-65G	58.148	58.1425	0.0055
4	17-65G	62.130	62.1223	0.0077
5	18-65G	54.7845	54.7789	0.0056

Figure 7 shows X-ray diffractograms of 65G steel samples before and after EPH. X-ray diffraction analysis showed that in the initial state in the structure of 65G steel only α-phase is present. After the EPH in samples 10-65G, 11-65G and 18-65G a reflex (121) of cementite is observed. Also after the EPH, broadening of the -phase interference lines is observed on the diffractograms. The broadening of the -phase interference lines is associated with the growth of dislocation density, martensite formation and is determined mainly by the martensite tetragonality [16-18]. In sample 17-65G, a slight broadening of interference line 110 is observed, and no cementite formation is detected in this sample. Apparently, this is due to the fact that this sample is characterized by a fine-grained ferrite-bainite structure formed during partial melting of the surface.

Thus, the main advantage of EPH is the possibility of obtaining a modified martensite layer on the steel surface. In this case, the base of the material does not change, i.e. the part retains its ductile core. The formation of a modified layer of fine-grained martensite with a small amount of cementite in the surface layers will have a positive effect on the performance properties of parts.

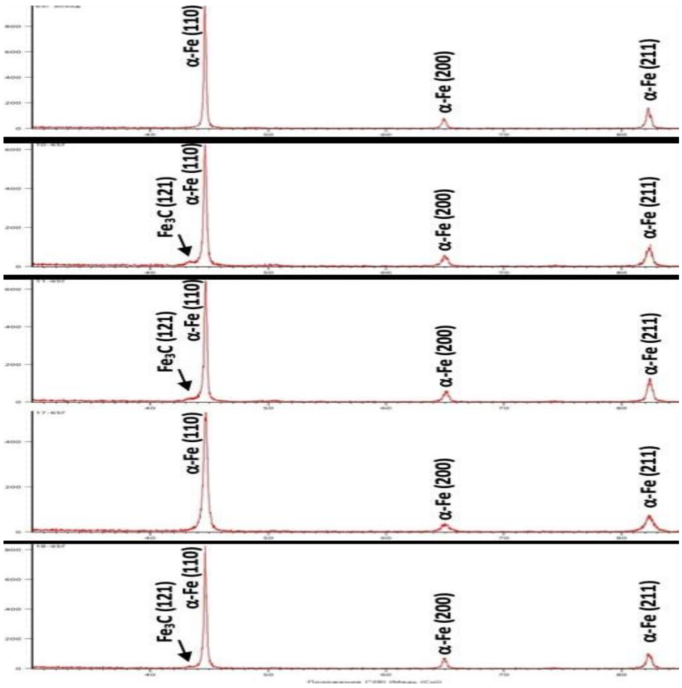


Figure 7. Diffractograms of 65G steel samples

As the results of the study after electrolyte-plasma surface hardening the steel has high wear resistance and strength characteristics.

On the basis of phase composition study it was found that after EPH a modified layer consisting of α'-phase (martensite) and cementite M_3C is formed. The increase in wear resistance of 65G steel after EPH is associated with the formation of martensite as well as the formation of defective substructure.

The bench tests of 65G steel samples on the test bench for air-erosion resistance and on the test bench for shock-abrasion wear were carried out.

The test results are shown in Table 4. It can be seen from the table that the weight loss of the treated samples is almost 2 times less than that of the initial samples. Thus, test results show that all samples that underwent electrolyte-plasma hardening are characterized by sufficiently lower erosion wear than the initial samples.

Table 4

Results of air-erosion resistance tests

Sample No.	Material	Erosion wear, g/min
10-65G initial	fragment of lancet paws made of 65G steel	0.045
10-65G		0.027

Table 5 shows the results of the shock-abrasion test. It can be seen that the weight loss of the hardened sample is less than that of the initial sample, which indicates an increased resistance to impact-abrasive wear.

The high resistance of the hardened samples to shock-abrasive wear is caused by the formation of martensitic structure.

Table 5

Results of the shock-abrasion test

No.	Samples name	Material	Mass loss, mg (shock abrasion)
2	10-65G initial	fragment of lancet paws made of 65G steel	0.0475
3	10-65G		0.0426

Conclusion

Analyzing the experimental results obtained, the following conclusions can be made:

1. As shown by the results of the study the considered steel after electrolyte-plasma surface hardening have high wear resistance and strength characteristics. Based on the study of the phase composition it was found that after EPH a modified layer consisting of α'-phase (martensite) and cementite M_3C is formed. The increase in wear resistance of 65G steel after EPH is connected with formation of martensite as well as formation of defective substructure.

2. The developed technological process of hardening of a part made of 65G steel makes it possible to obtain layers on the surface of the part ensuring a twofold increase in wear resistance, a 1.7-fold increase in resistance to abrasive wear, as well as to ensure uniform distribution of all phase formations in a thin surface layer, which, in general, will result in improvement of operating characteristics of the 65G steel part. In addition, local hardening ensures the achievement of technical and economic effect due to the absence of the need to isolate undesirable areas of the part, treating only the areas requiring hardening.

Thus, the conducted studies have shown the prospects and feasibility of using the developed method to improve the operating properties of parts working in conditions of friction and wear. The conducted studies have shown that EPH technology, which allows increasing hardness and wear resistance of 65G steel can be used to increase the service life of working elements of agricultural machinery. It is recommended to apply this method of EPH for hardening of working elements of agricultural machinery made of 65G steel without additional heat treatment. The EPH provides achievement of technical and economic effect due to the use of simple equipment, not expensive aqueous solutions, reduction of processing time, as well as due to increase of wear resistance, microhardness of steels.

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Электролитті-плазмалық өңдеу кезінде 65Г болатының фазалық құрамы мен трибологиялық қасиеттерінің өзгеруін зерттеу

Жұмыста әртүрлі режимдерде электролиттік-плазмалық шынықтыруға дейінгі және одан кейінгі 65Г маркалы болаттың фазалық құрамы мен трибологиялық қасиеттерін зерттеу нәтижелері келтірілген. Электролиттік-плазмалық қатайту технологиясы және электролиттік-плазмалық қатайтуды жүзеге асыруға арналған зертханалық қондырғы сипатталған. Электроплазмалық қатайтудан кейін α'фазасынан (мартенсит) және М3С цементиттен тұратын модификацияланған қабат пайда болатыны анықталды. 65Г болаттан жасалған бөлшекті қатайтудың дамыған технологиялық процесі бөлшектің бетінде тозуға төзімділікті 2 есе және абразивті тозуға төзімділікті 1,7 есе арттыруды қамтамасыз ететін қабаттарды алуға мүмкіндік береді. Жүргізілген зерттеулер үйкеліс пен тозу жағдайында жұмыс істейтін бөлшектердің пайдалану қасиеттерін арттыру үшін әзірленген әдісті қолданудың келешегі мен мақсаттылығын көрсетті. Бұл технологияны ауылшаруашылық техникасының жұмыс органдарының қызмет ету мерзімін ұзарту үшін пайдалануға болады.

Кілт сөздер: қатайту, фазалық құрам, плазмалық-электролиттік қатайту, тозуға төзімділік.

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Исследование изменений фазового состава и трибологических свойств стали 65Г при электролитно-плазменном упрочнении

В работе представлены результаты исследований фазового состава и трибологических свойств стали марки 65Г до и после электролитно-плазменной закалки при различных режимах. Описаны технология электролитно-плазменного упрочнения и лабораторная установка для осуществления электролитно-плазменного упрочнения. Было обнаружено, что после электролитно-плазменного упрочнения образуется модифицированный слой, состоящий из α'-фазы (мартенсита) и цементита М3С. Разработанный технологический процесс упрочнения детали из стали 65Г позволяет получить на поверхности детали слои, обеспечивающие повышение износостойкости в 2 раза и стойкости к абразивному износу в 1,7 раза. Проведенные исследования показали перспективность и целесообразность применения разработанного метода для повышения эксплуатационных свойств деталей, работающих в условиях трения и износа. Эта технология может быть использована для увеличения срока службы рабочих органов сельскохозяйственной техники.

Ключевые слова: трение, износ, фазовый состав, плазменно-электролитическое упрочнение, износостойкость.

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