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Influence of plasma electrolytic hardening modes on the structure and properties of 65G steel

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This work presented a study of the structure, hardness and wear resistance of 65G steel treated with electrolyte-plasma hardening under different conditions. The electrolyte-plasma hardening technology and a laboratory installation for the realisation of electrolyte-plasma hardening are also described. After electrolyte-plasma hardening, we have established that a modified layer consists of the α-phase (martensite) and M3C cementite. The study results showed that electrolyte-plasma hardening makes it possible to obtain layers on the 65G steel surface that provides an increase in microhardness by 2.6 times, wear resistance by two times, resistance to abrasive wear by 1.7 times compared to the original samples. In addition, local hardening ensures the achievement of technical and economic effects due to the absence of the need to isolate an unwanted site of parts, processing only the areas requiring hardening.

Keywords: structure, phase composition, plasma electrolytic hardening, microhardness, wear resistance.

Introduction

The durability of parts depends not only on the properties of the material determined by the manufacturing technology and volumetric hardening, but also to a large extent on the surface properties. Its role in ensuring the operational properties of products is constantly increasing which, along with the widespread use of traditional methods of chemical and thermal treatment, has contributed to the emergence and development of a new direction - surface engineering by methods of energetic and physicochemical effects. Implementation of this concept when choosing a material will improve the performance properties of parts and in some cases reduce the consumption of expensive materials. Thus due to the use of protective coatings and surface hardening lately more and more low-alloy structural and tool steels are being used and produced, which made it possible to reduce expenses for expensive high-alloy steels and alloys. At the same time, an important role in the use of protective coatings and surface hardening is given to the use of resource-saving technologies that help to reduce the consumption of resources and energy and to increase labor productivity [1, 2].

Recently, studies of plasma electrolytic method of material processing are being conducted quite intensively. As a result, various technologies have been developed for modifying the surface of metals and alloys based on the plasma electrolytic method: oxidation [3], polishing [4], diffusion saturation with nitrogen [5], with carbon [6, 7], with boron [8], multicomponent saturation [9, 10] and surface hardening [11]. Plasma electrolytic hardening (surface hardening) is of particular interest among them [11, 12].

The process of surface hardening was usually carried out by a laser beam, an electron beam and a plasma beam [1, 13]. Compared to these hardening processes, plasma electrolytic hardening is a simple and inexpensive method. All surface hardening methods are commonly used to harden the surface of steels. However, experience shows that under certain conditions during surface hardening a finely dispersed structure is formed, which increases the wear resistance of steel depending on its alloying. In addition, plasma electrolytic hardening (surface hardening) compares favorably with plasma hardening processes (surface hardening) due to the high cooling speed and lower degree of oxide layer formation. As plasma discharges are formed between the surface of the metal and the electrolyte, and the cooling process takes place in a flowing electrolyte. The electrical circuit between the electrodes is closed through an electrolyte (aqueous salt solution). Conversion of electrical energy into heat occurs mainly in the layer adjacent to the product. As a result of heating, this layer turns into a vapor-gas state, in which micro-bridges arise under the influence of the applied voltage. The power density reaches $3 \cdot 10^3$ W/cm^2 [14]. The technology allows varying the rate of heating and cooling and the thickness of the hardened layer within a wide range. By regulating the temperature and speed modes of plasma surface heating and cooling, as well as the use of various electrolytes, it is possible to obtain high rates of the mechanical and tribological characteristics of the surface layer of steels.

In connection with the above, the purpose of this work is to study the influence of the modes of plasma electrolytic hardening on the structure, phase composition, mechanical and tribological properties of 65G steel.

Materials, equipment and experimental techniques

In accordance with the tasks set, structural steel 65G was chosen as the object of the research. The choice of the research material is justified by the fact that spring structural steel 65G is characterized by high wear resistance, while being relatively cheap. These characteristics made this type of steel widely used in mechanical engineering. 65G steel is successfully used for the manufacture of working bodies of tillage machines.

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

Table 1.
65G steel chemical composition in % (GOST 14959-79).

Steel name	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

Plasma electrolytic hardening of steel samples was carried out in a laboratory setup at the Research Center "Surface Engineering and Tribology". The general and schematic view of the installation for plasma electrolytic processing is shown in Figure 1. The installation structurally consists of a power source, a chamber for plasma electrolytic processing of materials.

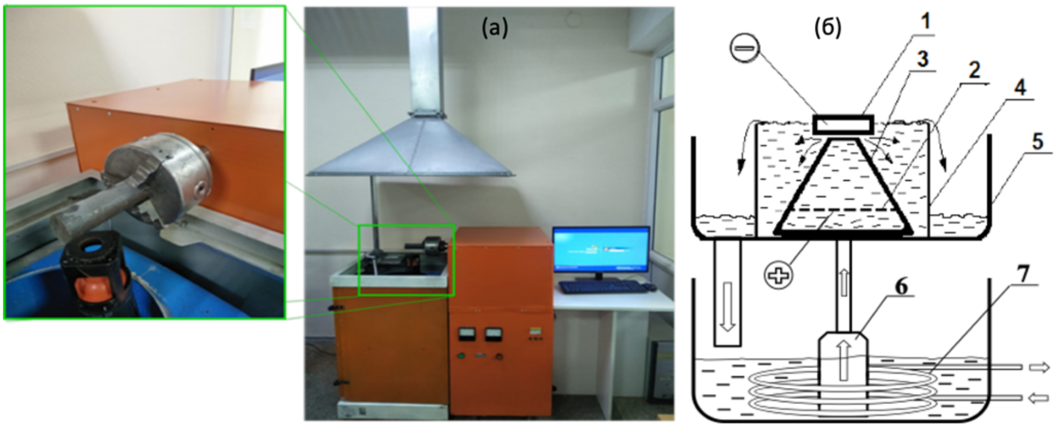

Figure 1. General (a) and schematic (b) view of the installation of plasma electrolytic treatment: 1- processed sample (cathode), 2-stainless steel anode with holes, 3-cone-shaped baffle, 4-working chamber - electrolyte bath, 5-pan, 6-pump, 7-heat exchanger.

The influence of plasma electrolytic hardening modes on the structure and properties of 65G steel was investigated. Samples of 65G steel were processed under different PEH modes. The PEH modes are shown in Table 2. PEH was carried out by variable supply of high (320V), medium (200V) and low (50V) voltages, and also carried out by cyclic exposure. An aqueous solution containing 15% sodium carbonate was used as the electrolyte. Distilled water was used to prepare the electrolyte.

Table 2.
Modes of thermocyclic plasma electrolytic hardening of 65G steel samples.

Sample number	Cycle 1			Cycle 2			Cycle 3			Cycle 4
	320V	200V	50V	320V	200V	50V	320V	200V	50V	320V
10-65G	1 sec	-	3 sec	1 sec	-	-	-	-	-	-
11-65G	2 sec	-	-	-	-	-	-	-	-	-
17-65G	1 sec	3 sec	5 sec	1 sec	1 sec	7 sec	1 sec	-	-	-
18-65G	1 sec	-	3 sec	1 sec	-	7 sec	1 sec	-	12 sec	1 sec

To study the general nature of the structure, we used Altami MET-5S optical microscope of the Research Center "Surface Engineering and Tribology" of EKU named after Sarsen Amanzholov. For metallographic microanalysis of thin sections after polishing, we etched with a 4% alcohol solution of nitric acid, using a paste of chromium dioxide.

The morphology and elemental composition of the sample treated in electrolytic plasma were investigated in the IRGETAS engineering laboratory of EKSTU named after D. Serikbayev on a JSM-6390LV scanning electron microscope made by JEOL (Japan), with an INCAEnergy energy dispersive microanalysis attachment made by OXFORD Instruments.

X-ray structural studies of steel samples were carried out by known methods of X-ray structural analysis on X'PertPRO diffractometers (of National Scientific Laboratory for Collective Use of S. Amanzholov EKSU). Diffractograms were recorded using CuKα radiation (λ=2.2897 Å) at a voltage of 40 kV. Diffractograms were interpreted manually using standard techniques and a PDF-4 database, and quantitative analysis was performed using the Powder Cell software.

The microhardness of steel samples was measured at the National Scientific Laboratory for Collective Use of S. Amanzholov EKSU on the device PMT-3 in accordance with GOST 9450–76, with loads on the indenter P=1 N and holding time at this load of 10 sec [15].

For the obtained coatings, the mechanical properties (Young's modulus, hardness) were investigated using the NanoScan-4D Kompakt nanohardness tester. Tests were carried out at the load of 200 mN. The loading-unloading rate was 3 mN/min. The dependence of penetration on the applied force at the stages of loading and unloading was determined by the Oliver-Pharr method [16].

Tribological tests for sliding friction were carried out on a TRB3 tribometer under an agreement with the Institute of Composite Materials LLP using the standard "ball-disk" technique (Figure 2a) (international standards ASTM G 133-95 and ASTM G 99). A ball from a certified material Al_2O_3 with a diameter of 6.0 mm was used as a counterbody. The tests were carried out at a load of 1 N and a linear velocity of 2 cm/s, a radius of curvature of wear of 5 mm, and a friction path was 40.1 m. The tribological characteristics of the modified layer were characterized by the intensity of wear and the coefficient of friction [17].

Abrasive wear tests of samples were carried out on an experimental setup for testing abrasive wear when rubbing against loose abrasive particles according to the "rotating roller-flat surface" scheme in accordance with GOST 23.208-79, which coincides with the American standard ASTM C 6568 (Figure 2b). To test abrasion on a rubber disc, the surfaces of the samples were ground and polished, and they were also cleaned with acetone and dried. A cylindrical rubber roller, pressed by a radial surface against a flat surface of a test specimen with a force of 22 N, rotated at a frequency of 1 s^{-1}. The diagram of the device is shown in Figure 2b. The rate of entry of abrasive particles between the rubber wheel and the sample, that is into the test area, was 41-42 g/min. Electrocorundum with a grain size of 200-250 microns was used as abrasive particles. The wear resistance of the processed test piece was evaluated by comparing its wear with that of a reference piece (non-treated piece). The wear was measured by the weight method on an ADV-200 analytical scale with accuracy up to 0.0001 g. The samples were weighed every minute and tested during three minutes. The total wear length was 28.8 m. Before weighing, the samples were blown with compressed air to remove remaining sand particles on samples. The wear resistance of the test material was assessed by the loss of weight during the test in accordance with GOST 23.208-79.

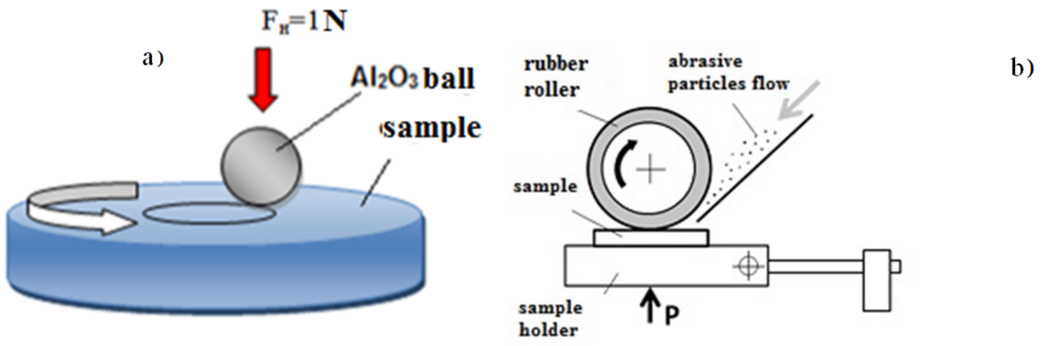

Figure 2. Tribological tests of samples: a)-according to the "ball-disk" scheme, b)-according to the "rotating roller - flat surface" scheme.

Experimental results and their discussion

The influence of plasma electrolytic hardening modes on the structure and properties of 65G steel was investigated. Samples of 65G steel were processed under different PEH modes (Table 2). Figure 3 shows the microhardness data of the samples before and after PEH under different modes. It can be seen that the microhardness of the initial sample is 244 HV. After PEH the microhardness increases depending on the processing mode. At the same time sample No. 10-65G, having passed only one PEH cycle, showed a slight increase (up to 65%) in microhardness. And samples No.17-65G and No.18-65G which passed through 3 and 4 cycles of PEH, showed an increase in microhardness up to two times. The highest level of microhardness of 653 HV is seen in sample No.10-65G, which had passed two PEH cycles. An increase in the microhardness of 65G steel up to 2.6 times after PEH which includes 2 heating cycles shows the promise of the plasma electrolytic technology in the processing of parts made of 65G steel.

Tribological tests of 65G steel samples were carried out before and after plasma electrolytic processing. The tests were carried out according to the "ball-disk"

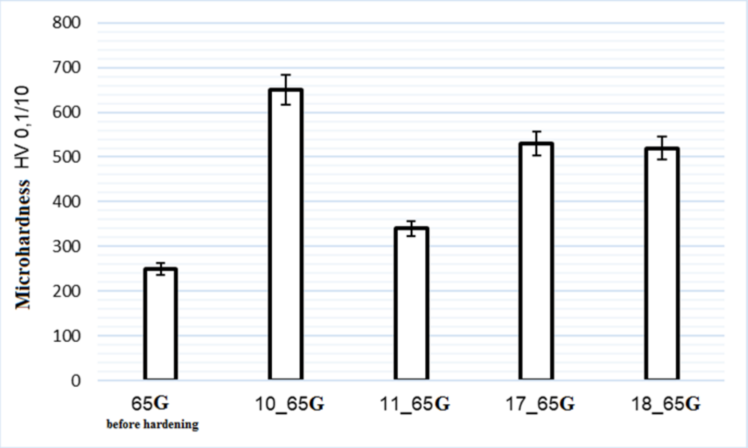

Figure 3. Microhardness data of samples before and after PEH under different modes.

scheme on a TRB3 tribometer (ASTM G99), as well as on a special stand for testing materials for abrasive wear according to the "rotating roller - flat surface" scheme (GOST 23.208-79). As a result of the tests, the tribological characteristics of the samples were determined: the coefficient of friction, the amount of wear during dry friction, and the loss of mass during abrasive wear.

Figure 4 shows the results of tribological tests. Tribological tests were carried out using counterbodies made of Si_3N_4 (a ball with a diameter of 6 mm) and 100Cr6 (a ball with a diameter of 3 mm). The amount of wear was determined by examining the profilogram of the wear tracks. The results showed that after PEH the wear volume decreases. Sample No.11-65G showed a higher value of the wear volume when using a Si_3N_4 counterbody, while when using a 100Cr6 counterbody the wear volume was low compared to the original sample. And sample No.10-65G showed the lowest value of the wear volume in comparison with other samples both when using the Si_3N_4 counterbody and when using the 100Cr6 counterbody.

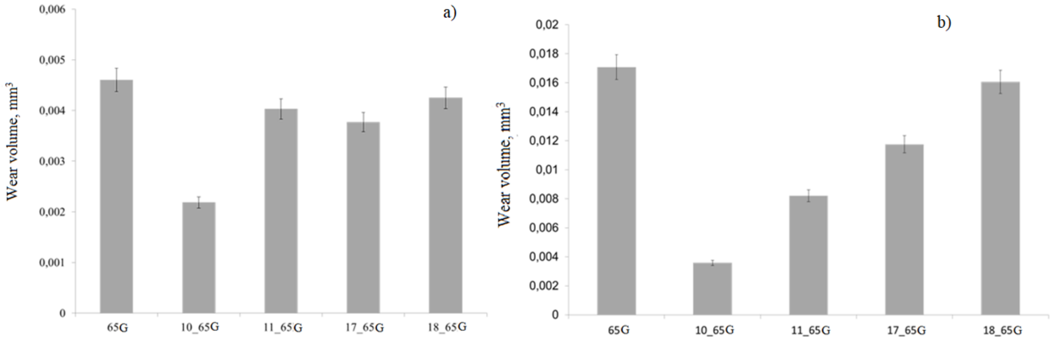

Figure 4. Data on the volume of wear of samples after tests using counterbodies from Si_3N_4 (a) and 100Cr6 (b).

Figure 5 shows the curves of the coefficient of friction of 65G steel samples during wear using counterbodies made of Si_3N_4 (a ball with a diameter of 6 mm) and 100Cr6 (a ball with a diameter of 3 mm). The test results using the Si_3N_4 counterbody showed that the friction coefficient varies greatly depending on the PEH mode. In all samples except for 18-65G, an increase in the coefficient of friction is observed. And when the counterbody 100Cr6 is used, an increase in the coefficient of friction is observed in all samples. Perhaps this is due to the fact that the processed samples had a high roughness in comparison with the original sample.

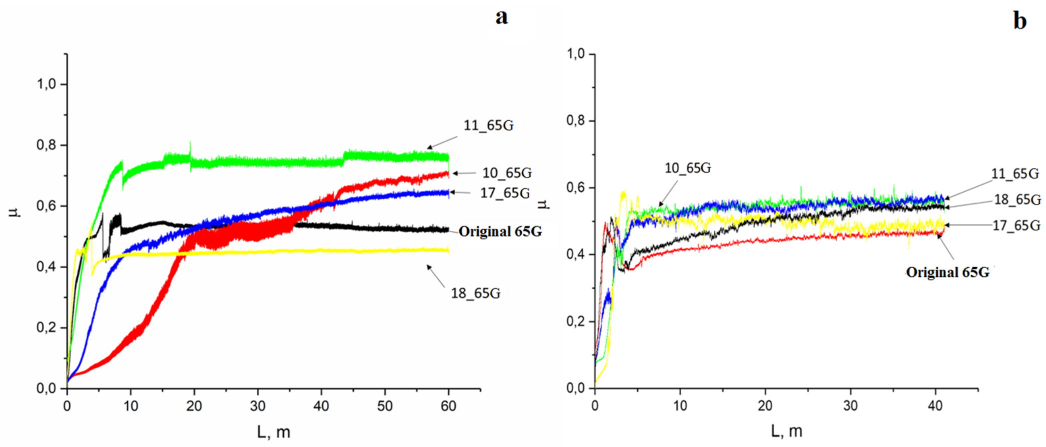

Figure 5. Curves of the friction coefficient of 65G steel samples during wear using counterbodies from Si_3N_4 (a) and 100Cr6 (b).

Samples were tested for abrasive wear on a special stand in accordance with GOST 23.208-79. The wear resistance was assessed by the weight method. The mass of the samples was measured using a Gibertini CRYSTAL 100 CE analytical scale with magnetic compensation. Table 3 shows the sample mass before and after testing. The table shows that the weight loss of all processed samples is low compared to the original sample. At the same time, samples No.10-65G and No.18-65G showed higher resistance to abrasive wear. Thus, it can be claimed that PEH makes it possible to increase the abrasive wear resistance of 65G steel by 1.7 times.

Table 3.
Results of measuring the mass of samples before and after tests.

No.	Sample	Mass before, g	Mass after, g	Mass loss, g
1	Original 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

The data obtained made it possible to choose the optimal mode of PEH for 65G steel. Considering that sample No.10-65G has higher mechanical and tribological characteristics in comparison with other samples, we have identified that the optimal mode of PEH is heating the sample in the first cycle at a voltage of 320 V during 1 sec, followed by a transition to a low voltage of 50 V during 1 sec, then heating in a second cycle at 320V during 1 sec, followed by cooling in a flowing electrolyte by switching off the voltage.

The structural phase states of the hardened surface layers of 65G steel samples were investigated. Figure 6 demonstrates X-ray diffractograms of 65G steel samples before and after PEH. X-ray structure analysis showed that in the initial state only the α-phase is present in the structure of 65G steel. After PEH the cementite reflex (121) is observed in samples No.10-65G, 11-65G and 18-65G. Also, after PEH, the diffractograms indicate broadening of the interference lines of α-phase. The broadening of the α-phase interference lines is associated with an increase in the dislocation density, the formation of martensite and is mainly determined by the tetragonality of martensite [18-20]. In sample 17-65G a slight broadening of the interference line 110 is observed, and the formation of cementite was not detected in this sample. Apparently, this is due to the fact that this sample is characterized by a fine-grained ferrite-bainite structure, which is formed upon partial melting of the surface.

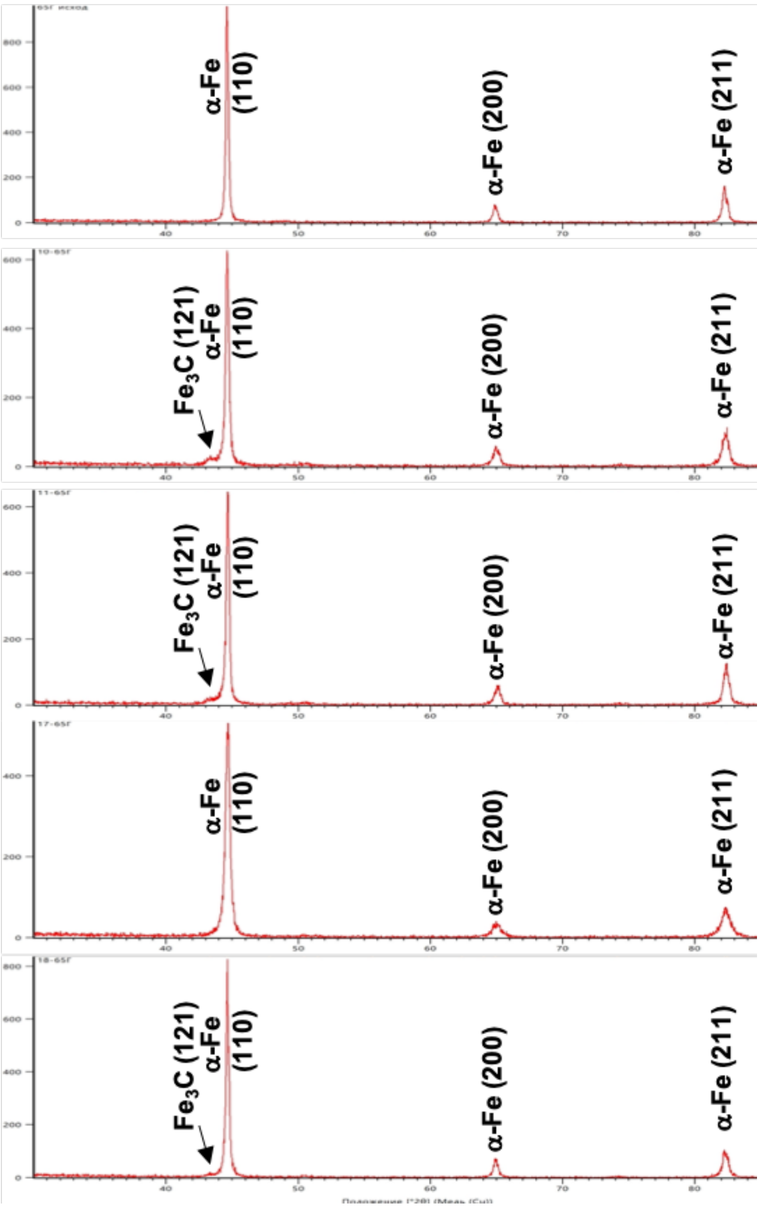

Figure 6. Diffractograms of samples.

Metallographic analysis showed that in the initial state the structure of 65G steel consists of a ferrite-pearlite structure, and after PEH there is a change in the microstructure of 65G steel depending on the processing mode (Figure 7). Samples No.10-65G and 11-65G consist of martensite and ferrite. Whereas, samples No.17-65G and 18-65G consist of a ferrite-bainite fine-grained structure. The formation of a ferrite-bainite structure is connected with a low cooling rate of samples 17-65G and 18-65G in comparison with samples 10-65G and 11-65G.

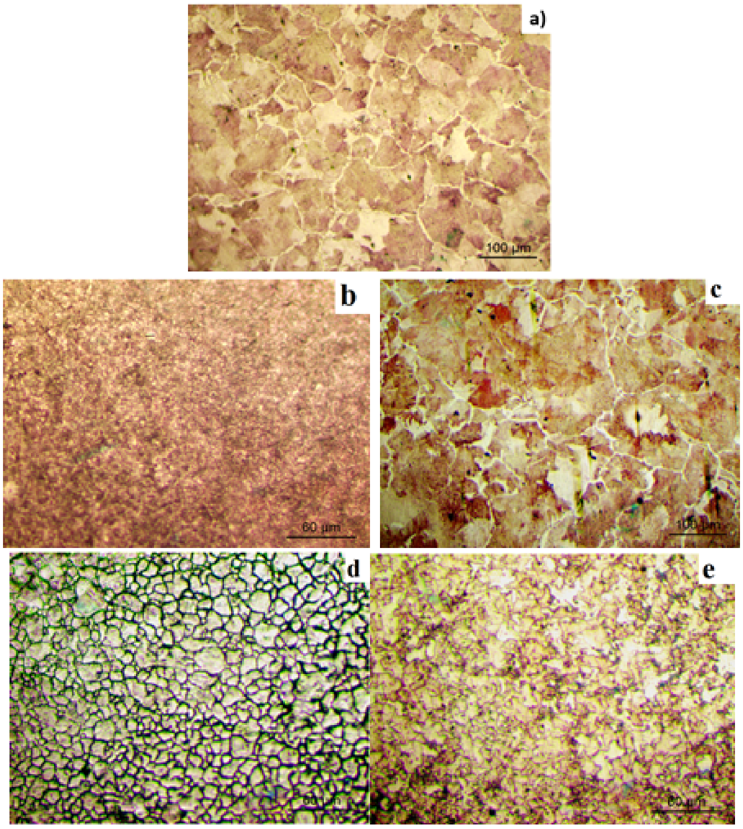

Figure 7. Surface microstructure of 65G steel samples before (a) and after PEH (b)-10-65G, (c)-11-65G, (d)-17-65G, (e)-18-65G).

Thus, the main advantage of PEH is the possibility of obtaining a modified layer of martensite on the steel surface. Wherein, the basis of the material does not change, i.e. the part retains its viscous 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 the parts.

As shown by the results of the study the considered steel has high wear resistance, microhardness and strength characteristics after plasma electrolytic surface hardening.

Based on the study of the structure and phase composition, it was found that after PEH a modified layer is formed, consisting of the α-phase (martensite) and cementite M3C. An increase in the microhardness and wear resistance of 65G steel after PEH is associated with the formation of martensite, as well as the formation of a defective substructure.

As a result of experimental studies of changes in the structural-phase states and tribological properties of 65G steel during PEH and on the basis of the developed method of plasma electrolytic hardening and selected optimal modes, a technological process of PEH of the working surface of 65G steel parts has been developed, including:

- preliminary preparation of the workpiece surface to be treated, including the removal from the surface of visible layers of organic contaminants such as residues of oils, lubricants and grease stains using ethyl alcohol or acetone;

- loading the workpiece into the chamber the way that the treated area is under the electrolyte layer;
- plasma electrolyte hardening of the working surface;
- cooling the processed part in electrolyte.

Figure 8 depicts a diagram of the process of plasma electrolytic hardening of a part made of 65G steel.

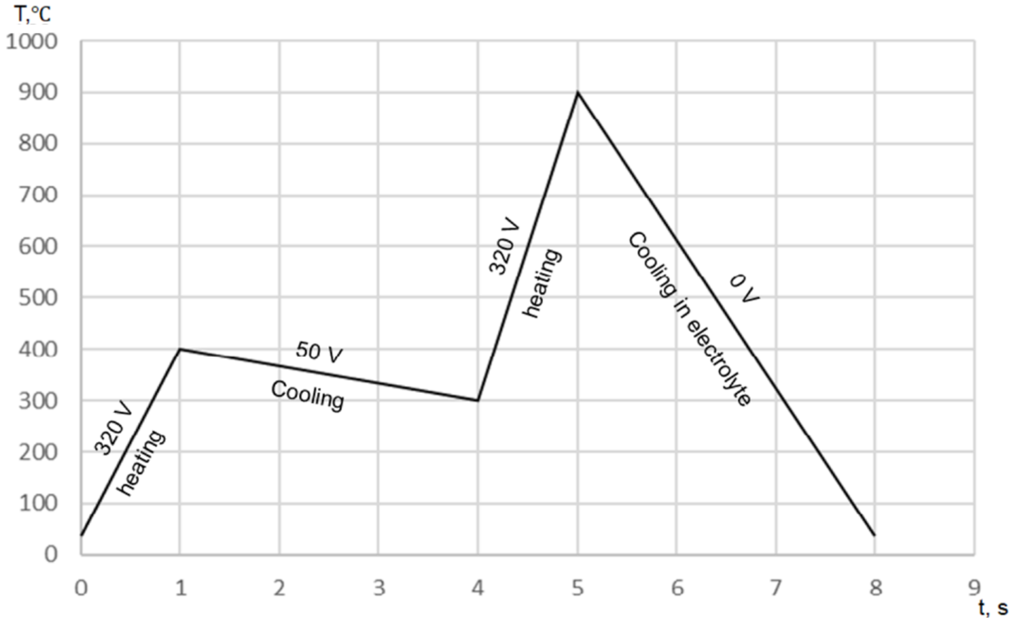

Figure 8. Diagram of the process of plasma electrolytic hardening of a part made of 65G steel.

The sequence of operations during the technological process is as follows:

Before starting work the bath is filled with electrolyte. Then the electrolyte is fed to the electrolytic cell by means of a pump installed at the bottom of the bath. Wherein, the electrolyte flows out through the opening of the cone-shaped baffle in the form of a jet and fills the electrolytic cell. Then the electrolyte is drained through the electrolyte drain holes in the electrolyte cell into the pan and then back into the bath. Thus, the electrolyte is in circulation mode. The flow rate of electrolyte 2 (consumption) is 4-7 l/min. For cooling and maintaining the required temperature of the electrolyte there is a heat exchanger at the bottom of the bath. The flow rate of cooling running water into the heat exchanger is 3-6 l/min. The adopted parameters of electrolyte cooling allow maintaining the temperature in the range of 40-70 ° C when the sample is heated to a temperature of 900-1000 ° C. With the help of a device for fixing the workpieces being processed, the workpiece is immersed in the electrolyte the way that the processed part of the workpiece is under the electrolyte layer at a depth of 10-15 mm. At the same time, a stream of electrolyte is supplied to the treated area through the opening of the cone-shaped baffle. Then the anode is connected to the positive pole of the power supply, and the processed piece-cathode-is connected to its negative pole. To heat the surface of the part to the hardening temperature a voltage of 300-320 V is applied between the electrodes and the current density is 20-25 A/ cm^2 . At such voltages an intensely glowing plasma layer is formed in the cathode region and the tool is heated at a rate of 450-500 ° C/sec. An abnormal arc discharge is formed between the electrodes, due to which the tool being processed quickly heats up [21, 22]. In the first PEH cycle a voltage of 320V is applied during 1 second to heat the surface to a temperature of 400-450 ° C. After reaching this temperature (after 1 sec), the voltage is sharply reduced to 50 V for 1 sec for slow cooling, and then there come the second cycle which switches to heating at 320 V within 1 sec, followed by cooling in the electrolyte. The PEH process consists of two cycles and this, in turn, does not allow the parts to be exposed to erosion, roughness deterioration, and also provides the ability to control the heating temperature of the workpiece.

The temperature of the processed piece is controlled by a thermocouple. After selecting the optimal processing parameter for each type of part, the PEH process can be carried out in an automated mode. The selected processing parameters (voltage and duration of the process in the first and second heating cycle, the rate of electrolyte supply) are set by the computer, which controls the hardening process.

The developed technological process of hardening a part made of 65G steel makes it possible to obtain layers on the part surface that provide an increase in microhardness by 2.6 times, wear resistance by 2 times, abrasive wear resistance by 1.7 times, as well as to ensure a uniform distribution of all phase formations in a thin surface layer, which in general will lead to an improvement in the performance characteristics of the 65G steel part. In addition, local hardening ensures the achievement of a technical and economic effect due to the absence of the need to isolate unwanted areas of the part, processing only the areas requiring hardening.

The developed method of hardening makes it possible to obtain a fine-grained martensitic structure on the surface of the part, which provides an increase in wear resistance. It makes it possible to carry out surface hardening of parts with large dimensions without deteriorating the quality (roughness) of the surface.

The technical result from the use of the developed technology is in strengthening the cutting surface of the working bodies of agricultural machinery (plowshares, plowshares of cultivators) leaving the core untouched and obtaining a uniformly distributed heterophase layer that provides increased wear resistance and improved mechanical characteristics of the workpiece.

Compared with the existing methods, the proposed method is carried out on inexpensive equipment, the composition of the electrolytes used is environmentally friendly and can be used repeatedly without additional energy costs. The hardening process is controlled by a personal computer. It becomes possible to automate the hardening process and include it into the general flow of parts processing.

Thus, the studies carried out have demonstrated the prospects and feasibility of using the developed method to improve the operational properties of parts operating under conditions of friction and wear. It can be claimed that the use of plasma electrolytic hardening instead of the technology of laser and electron-beam hardening allows to save the main equipment and production areas, reduce energy consumption and overhead costs.

Conclusion

Analyzing the experimental results obtained in this work, the following conclusions can be drawn:

As shown by the results of the study the considered steel has high wear resistance, microhardness and strength characteristics after plasma electrolytic surface hardening. Based on the study of the structure and phase composition, it was found that after PEH there forms a modified layer witch consists of the α-phase (martensite) and cementite M3C. An increase in the microhardness and wear resistance of 65G steel after PEH is associated with the formation of martensite, and also with the formation of a defective substructure.

The optimal mode has been determined and the technological process of hardening a part made of 65G steel has been developed. The developed technological process of hardening a part made of 65G steel makes it possible to obtain layers on the surface of the part that provide an increase in microhardness by 2.6 times, wear resistance by 2 times, resistance to abrasive wear by 1.7 times, as well as to ensure a uniform distribution of all phase formations in a thin surface layer, which in general will lead to an improvement in the performance characteristics of the 65G steel part. In addition, local hardening ensures the achievement of a technical and economic effect due to the absence of the need to isolate unwanted areas of the part processing only the areas requiring hardening.

Thus, the studies carried out have shown the prospects and feasibility of using the developed method to improve the operational properties of parts operating under conditions of friction and wear. The studies carried out have demonstrated that the PEH technology, which makes it possible to increase the hardness and wear resistance of 65G steel, can be used to increase the resource of the working bodies of agricultural machinery. This PEH method is recommended to be used to strengthen the working bodies of agricultural machinery made of 65G steel without additional heat treatment. PEH ensures the achievement of a technical and economic effect due to the use of simple equipment, inexpensive aqueous solutions, a reduction in the duration of processing, and also an increase in wear resistance and microhardness of steels.

Acknowledgments

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