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ELECTROLYTIC-PLASMA MODIFICATION FOR THE SURFACE HARDENING OF A LOW-CARBON STRUCTURAL STEEL FOR FRICTION AND WEAR-RESISTANT APPLICATIONS.

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Abstract

An alternative technology of surface hardening by the electrolytic-plasma modification (EPM) of a low-carbon alloy steel has been developed. The processing of 20KH (207) alloy steel samples to achieve surface hardening was conducted using electrolytic-plasma surface modification in a Na₂CO₃ solution electrolyte. The optimal processing modes of the electrolyte-plasma modification treatment was determined experimentally using voltage and applied current measurements. The quenching of the steel was performed in the electrolyte stream. The plasma was ionized after excitation. A mathematical model using thermal conductivity equations and regression analysis relating the key parameters of the hardening process was also carried out. Our experimental and mathematical model results both confirmed that the EPM processing clearly leads to a significant reduction in the time for hardening relative to other traditional hardening methods for steels. We observed that electrolyte-plasma modification (EPM) processing promotes the transformation of coarse-grained pearlite-ferrite microstructure into quenched martensite in 20KH (207) alloy steel samples using inverted light optical microscopy. Our Vickers micro-hardness indentation measurements showed an increase in hardness relative to the initial state of the steel samples after electrolyte-plasma surface treatment. The major advantage of the method of electrolytic-plasma treatment includes a low energy consumption at high quenching rates and the possibility of a local surface treatment. They also provide an overall cost reduction in the surface treatment of steels for various industrial applications.

Key words: Electrolyte-plasma modification, microstructure, hardening, hardness.

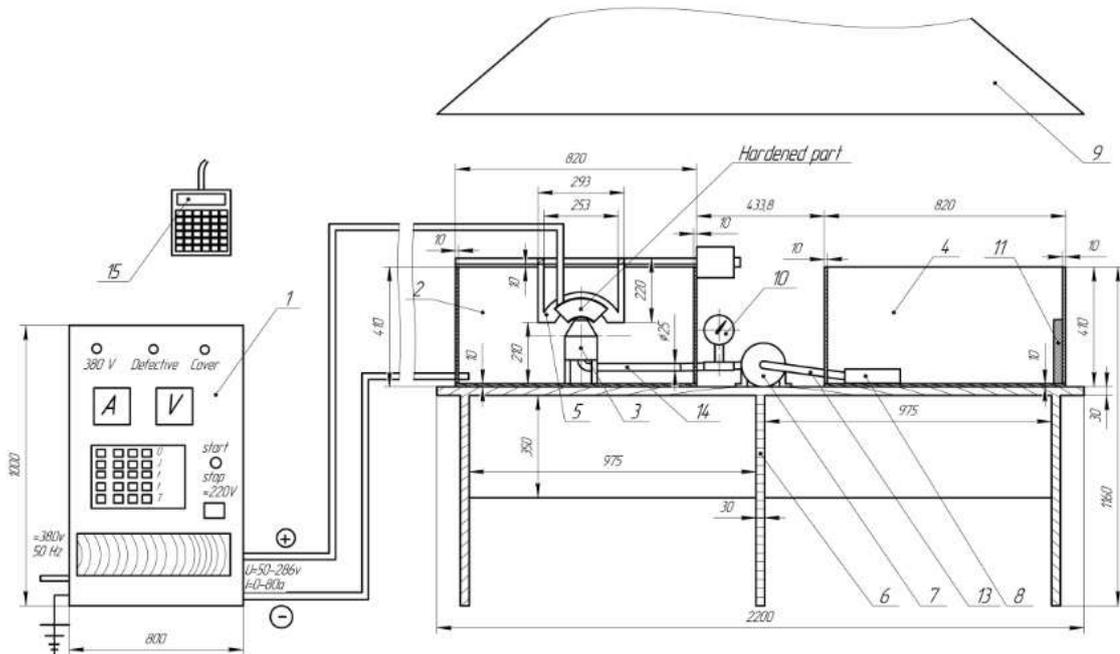
Introduction

An important issue facing the global machine-building industry is the introduction of innovative technologies and the accelerated modernization of the machine-building complex. Surface hardening and wear-resistance are essential for engineering components used in mining, metallurgical engineering and the oil and gas production industries [1]. One of the activities of machine-building enterprises in the oil and gas is the production of pipeline valves and oil-field equipment. The valves are manufactured with low-carbon alloy steels by machining and parts are welded together in most cases. Wedge Column Equipment Strapping (CES) used in the

process of pumping oil is exposed to high contact stresses and impact fatigue wear. During the process of pumping oil, wedge string equipment piping is subjected to high contact stresses and impact fatigue wear. Strengthening of CES dies from low-carbon steel 20Kh GOST 33260-2015 (207) is usually carried out using the traditional method inside an electric furnace by carburizing in a solid carburizer (charcoal) followed by quenching [2]. However, this process is accompanied by high heat losses, which reduce the efficiency, large expenditures of time (especially when heating the furnace to the required temperature), high energy consumption (power consumption is 60-100 kW) [3]. The penetration rate of carbon is low, and for depths of 1-2 mm, it becomes necessary to heat the workpiece in a carburizer for 8-10 hours at a certain temperature, followed by quenching [4]. At present, along with traditional methods of mechanical and thermal treatment, methods of high-energy local hardening are becoming more and more widely used. In our current investigation, the electrolyte-plasma process of diffusion saturation of low-carbon steel used for oil and gas fittings is considered. The saturation time takes several minutes and is easily combined with quenching in the same electrolyte without reheating. The aim of our current investigation is to study the effect of electrolytic-plasma modification (hereinafter referred to as EPM) of low-carbon alloy steel, to improve the productivity, processing efficiency, surface quality, as well as the structure and properties of the treated steel surface.

Experimental Methods and Techniques

To perform the EPM, a specially designed experimental setup shown in Figure 1 was used. A programmable power supply 1 (hereinafter referred to as PS) converts the energy of a three-phase AC network with a frequency of 50 Hz into the energy of a pulsed single-phase high voltage direct current.



1- Power supply; 2 - working bath; 3- cone nozzle; 4- reservoir; 5 - clamping mechanism; 6 - table; 7- pump; 8 - filter; 9- hood; 10- manometer; 11- thermometer; 12 - Ball valve; 13, 14 - high pressure hoses; 15 - control panel.

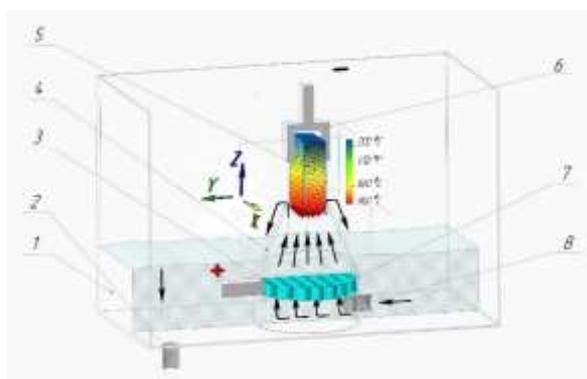
Figure 1 - Experimental setup for electrolyte-plasma modification.

The test sample (part) is installed and clamped in the clamping mechanism 5, which allows adjusting the required depth of immersion of the cathode (sample, part) into the Na_2CO_3 solution

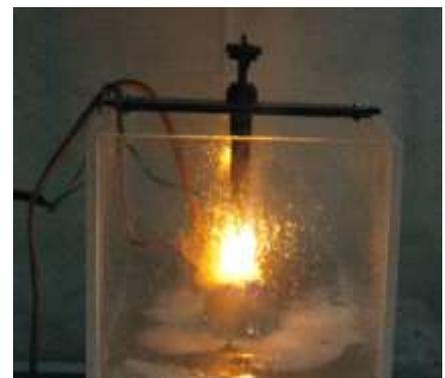
electrolyte. Cone nozzle 3 is a working device in which a vapor-air plasma is excited between the liquid anode and the part - the cathode. The electrolyte from the reservoir 4 is supplied by the pump 7 directly by the high-pressure hose 14 to the nozzle 3 of a conical shape, into which the anode of stainless steel 12Kh18N10T GOST 2590-2006 (X6CrNiTi18-10) is inserted. The electrolyte pressure is controlled by a pressure gauge 10 and is regulated by a ball valve 12. The operating temperature of the electrolyte (at the level of 20 - 60 ° C) is controlled by a thermometer 11. From the working bath 2, the used electrolyte is fed back through the high-pressure sleeve 13 into the reservoir 4. The mode of electrolyte-plasma treatment is determined experimentally: rated voltage $U = 200$ V, current $I = 10$ A, processing time 4-6 sec, hardening 4-10 sec, total time 4 min. Quenching is performed in an electrolyte stream. Strengthening is carried out by periodic heating due to the electric potential in the plasma layer and cooling the surface of the hardened sample [1], created between the liquid electrode (electrolyte) and the cathode surface (sample). The most important element of the EPO is nozzle 3, the design of which determines the density of the plasma, the stability of its ionization, as well as the speed and nature of the outflow of the electrolyte.

The cone nozzle, Figure 2 a, is placed in a dielectric working bath 1. Anode 3 is made in the form of a cylindrical plate with transverse holes 7, 2 mm in diameter [5]. The anode is inserted into the conical nozzle 4, and the hardened sample / part is used as the cathode 5. From the reservoir through the nozzle 8 electrolyte 10% soda ash (Na_2CO_3 solution) is fed into the nozzle 4. During the processing, it circulates back to the reservoir through the tube 2. When a power source is connected, a vapor-gas shell is formed between the cathode and the liquid electrolyte, which is accompanied by film boiling [6]. In this short period of time, the constituent components of the electrolyte are ionized, and the electrolyte plasma is excited, Figure 2 b. As is known, the plasma temperature is much higher than the temperature of structural phase transformations, and therefore, we determined the modes of electrolyte-plasma treatment experimentally. Quenching is carried out in an electrolyte flow; the best result is achieved with cyclic processing [7].

The studies were carried out on specimens made of steel 20Kh GOST 33260-2015 (207): C 0.17-0.23%; Si 0.17-0.37%; Mn 0.5-0.8%; Cr 0.7-1.0%, cut from a CES die with dimensions of 10x10x25 mm.



a) EPM 3D model



b) EPM processing

1 working bath; 2 tube for return of electrolyte; 3 anode plate; 4 cone nozzle; 5 cathode - sample / detail; 6 clamping mechanism; 7 holes on the anode; 8 nipple for electrolyte supply.

Figure 2. Electrolytic-plasma modification of a sample in a working bath.

Samples for metallographic analysis, electron microscopic studies, were subjected to preliminary grinding, polishing and etching. For these purposes, the treated samples, after washing and drying, were poured with epoxy resin into a plastic mold (Figure 3), and subjected to grinding and polishing to a mirror finish. Then, the polished surface of steel samples was etched with a 5% solution of nitric acid in ethyl alcohol (Kurpatov reagent) for 5–7 seconds [8]. The degree of etching was monitored on a microscope with fixation with ethyl alcohol and rinsing in running water. The metallographic study of the substrate was carried out on an Axioscop-2MAT inverted reflected light photomicroscope with a Sony digital camera [9]. The microhardness was measured by the Vickers method, in accordance with the requirements of ST SEV 469-77 and ISO 6508-86.

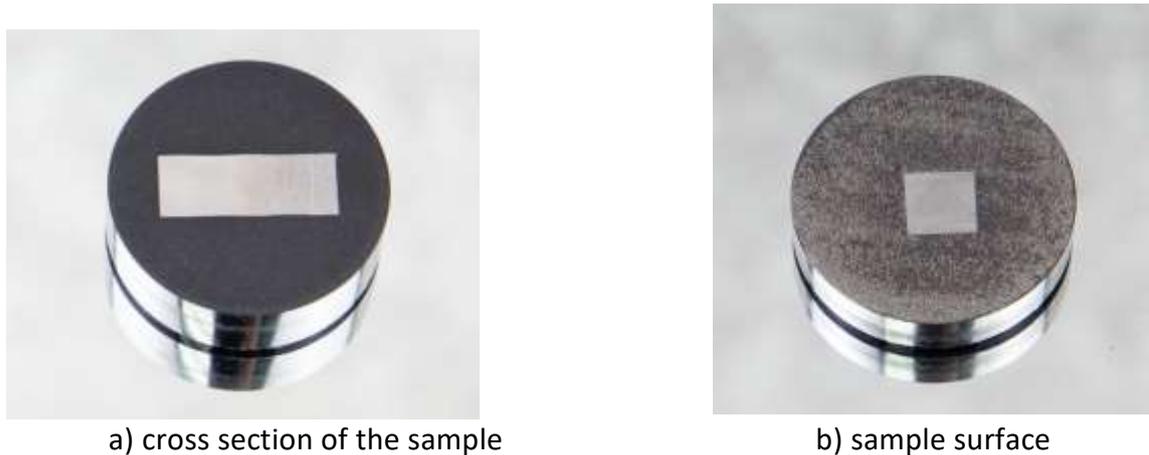


Figure 3 - Preparation of samples for research.

A conical diamond tip with an apex angle of 120° was used as an indenter; the value was rounded up to 0.5 hardness units [10].

Experimental results and discussion

In the initial (hot-rolled state) the microstructure of 20Kh (207) steel is a coarse-grained pearlite-ferrite structure shown in Figure 4. The forms of ferrite and pearlite grains are closed by a thin shell of iron carbide, which form the cementite phase. Chromium alloying elements have a finely dispersed distribution throughout the entire volume of steel. In low carbon steels, ferrite predominates, while pearlite grains account for a small proportion. During furnace (slow and prolonged) heating, it has been previously reported that carbon concentrated in pearlite grains have time to become evenly distributed over the entire volume of the metal as a result of diffusion [11]. Its concentration in comparison with the concentration in pearlite grains (0.8%) decreases to the brand value of 0.1–0.2% [12]. This prevents low-carbon steels from effectively hardening when quenched from a furnace.

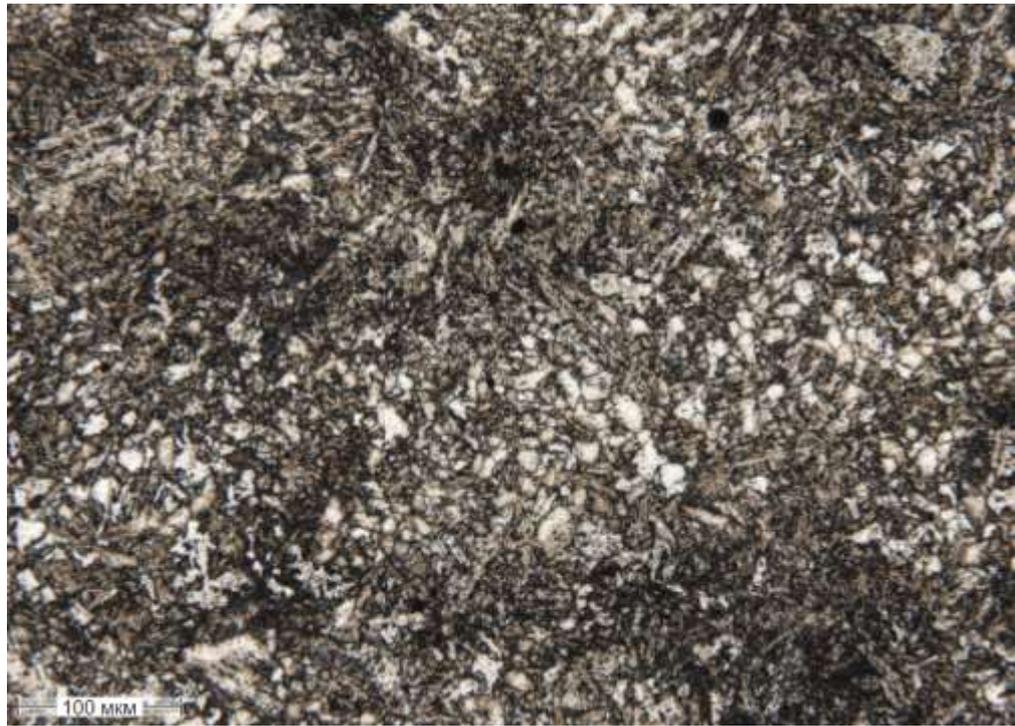


Figure 4. Microstructure of steel 20Kh (207) in the initial state, optical microscopy.

Therefore, to strengthen the CES dies from low-carbon alloy steel, surface saturation with carbon is carried out - by carburizing on a solid carburizer followed by quenching [13]. This heat treatment process is very time-consuming, labor-intensive and energy consuming.

The development of plasma hardening technologies makes it possible to reconsider the prevailing process on the strengthening of low-carbon steels. EPM processes are best described by the volt-temperature characteristic with the superposition of the real time of the process of one cycle as shown in Figure 5. When it is considered that the electrolyte circulated from the reservoir to the nozzle and through the working bath at room temperature of about 20 °C, then the plasma will be expected to be excited in a few seconds [13]. At low voltages (section AB), the cathode immersed in the electrolyte will experience an electrical resistance. Due to the localization of resistance on the sample, an increase in voltage leads to heating of the solution in the zone of contact with the electrolyte to boiling and the appearance of a current interruption (section BC). In this part of the curve, a pulsed character of current passage is observed due to periodic condensation processes and the formation of a vapor layer around the cathode / workpiece [14]. In this case, the occurrence of spark discharges, accompanied by crackling and luminescence, is characteristic. The electrolyte plasma at point C becomes stable, and the temperature rises, the system goes into heating mode (section CD). In this case, the temperature rises with an increase in voltage to 150–280 V due to an increase in the power released in the electrolyte plasma [15].

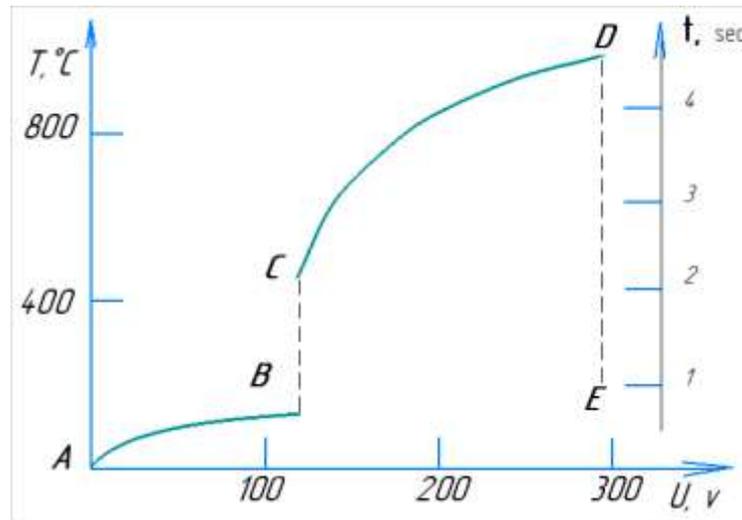


Figure 5. Volt-temperature characteristic of EPM, in the time interval of one cycle.

The section DE is characterized by a sharp decrease in the cathode temperature caused by the cooling of the electrolyte. The most significant characteristics of the EPM process are temperature and heating rate. A high heating rate classifies EPM as a high-speed method of chemical-thermal treatment [16]. So, for the cathodic version of the EPM at a constant voltage value, the heating rate of the part reaches up to 200 deg/s [17], and for the anode one - up to 250 deg/s [18]. The heating temperature largely depends on the applied voltage, the size of the electrode, the temperature and the rate of circulation of the electrolyte. The dependence of the anode temperature has a maximum in the heating mode on the value of the applied voltage [18]. An increase in the anode diameter leads to a decrease in its temperature at a constant voltage, and an increase in height (immersion depth), on the contrary, to an increase in temperature. This is due to the peculiarities of the distribution of heat flows in the system. Thus, the maximum heating temperature of the sample from the plate in the Na_2CO_3 solution reached at the lowest electrolyte temperature, and the minimum heating temperature - at the highest electrolyte temperature.

The problem of optimizing the heating and heat treatment process during electrolytic-plasma treatment can be considered as a function of $Q (k_1, k_2, k_3, k_4)$. The factor ranges of the numerical values k_i of the parameters under study are presented in Table 1.

Table 1 - Variable factors during electrolyte-plasma modification

Factors	Physical values of factors	Factor levels	
		min	max
k_1	Heating time of the workpiece from the plasma temperature, sec	1	15
k_2	DC voltage, V	180	300
k_3	Cooling time in electrolyte flow, sec	1	10
k_4	Number of processing cycles	20	40

To carry out a comparative analysis of the technological modes influence on the part material strengthening, it was decided to switch to a three-factor model of the experiment, fixing the number of processing cycles at the level of $k_4 = 30$ cycles. We thus experimentally established the

main factors that determine the quality of steel hardening at EPM as *heating time, hardening time, and voltage*.

The processes of heating and heat treatment during EPM are directly related to the change in the temperature of the substance T according to the thermal conductivity equation (1).

$$\frac{dT}{dt} = \lambda \Delta T \quad (1)$$

where λ – coefficient of thermal conductivity; t is time.

$$\Delta T = \frac{d^2T}{dx^2} + \frac{d^2T}{dy^2} + \frac{d^2T}{dz^2}$$

where x, y, z – coordinates allow the spatial case of the sample to be considered as shown in Figure 2 a.

For objects of different shapes, different coordinate systems can be considered, and equation (1) can be represented as

$$\frac{dT}{dt} = \frac{1}{x^n} \frac{d}{dx} \left(x^n \lambda \frac{dT}{dx} \right), \quad (2)$$

where for $n = 0$, equation (2) is investigated in a rectangular coordinate system, for $n = 1$ - in a cylindrical one, and for $n = 2$ - in a spherical coordinate system.

The uniqueness of the solution is provided by the initial and boundary conditions.

Boundary conditions in the general setting are as follows:

$$\begin{aligned} (A_1 T + A_2 \frac{dT}{dx}) \Big|_{x=0} &= A; \\ (B_1 T + B_2 \frac{dT}{dx}) \Big|_{x=0} &= B; \\ (C_1 T + C_2 \frac{dT}{dx}) \Big|_{x=1} &= C; \\ (D_1 T + D_2 \frac{dT}{dx}) \Big|_{x=0} &= D. \end{aligned} \quad (3)$$

By setting parameters $A_1, A_2, B_1, B_2, C_1, C_2, D_1, D_2$ in equation (3), the boundary conditions of the problem can be varied.

The influence of EPM modes and the quality indicators of hardening of steel 20Kh (207), depend on parameters such as the heating time T_{heating} from ionized plasma, the quenching time $T_{\text{quenching}}$, and the DC voltage U between the electrodes, summarized in in Table 2.

Table 2 - EPM modes during hardening of steel 20Kh (207)

Parameters	EPM processing modes			
	A	B	C	D
$T_{\text{heating, sec}}$	2	4	8	15
$T_{\text{hardening, sec}}$	2	4	8	15
U, V	180	200	250	300

The heating temperature is the main parameter of phase transformations, for steel 20Kh (207), which is 840-860 °C [19]. When the ionized plasma is excited (the plasma temperature is in the range from 6000 K to 30,000 K), a vapor-gas layer appears on the surface of the sample, resulting in the dissociation of the electrolyte. The high plasma temperature in a few seconds heats the sample, Figure 6 a, and when the power source is turned off, the sample is quenched in the electrolyte flow, Figure 6 b.

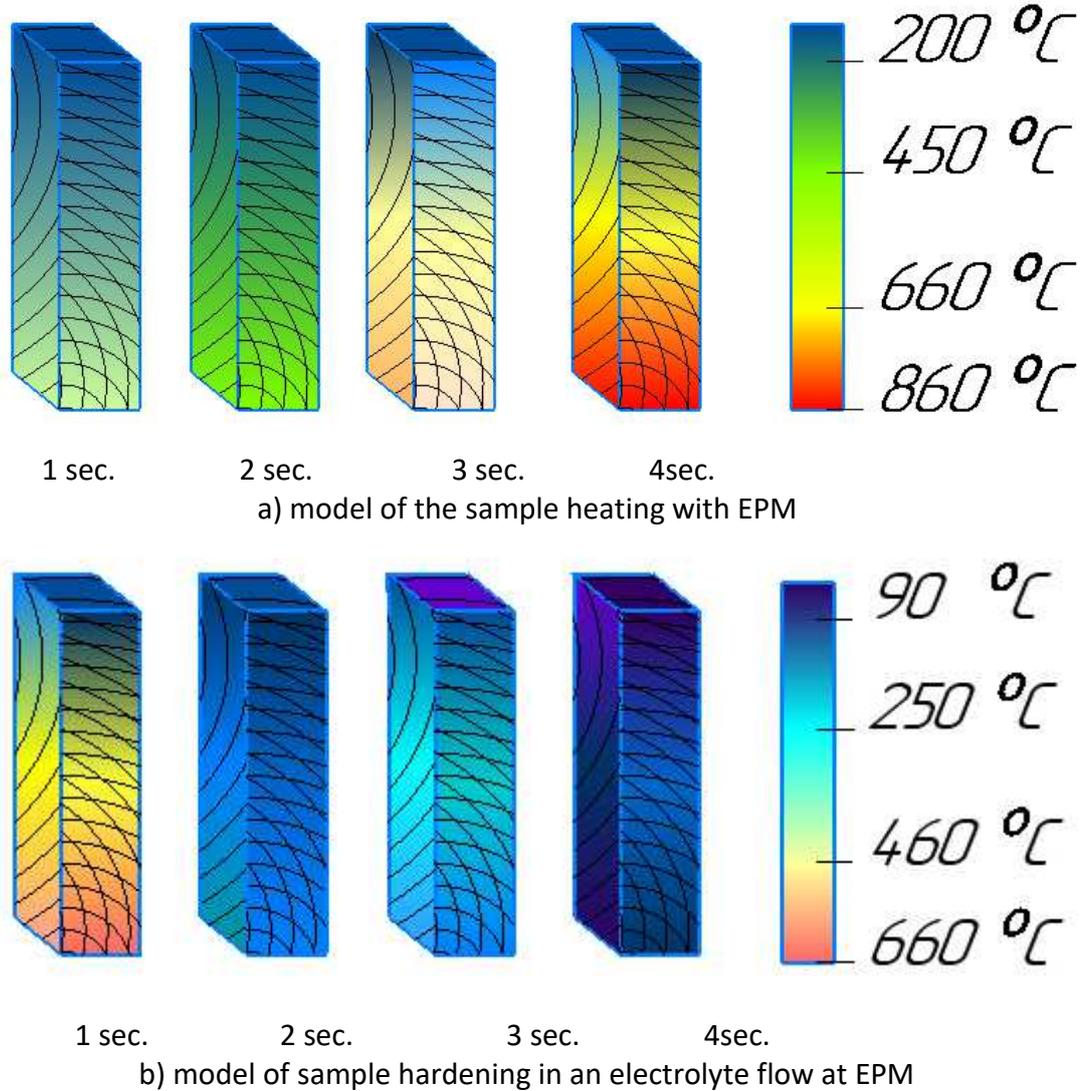


Figure 6 – The Mathematical model estimate of the temperature profile dependence on the quenching time of the modelled steels in the electrolyte.

Taking into account the accepted assumptions, the thermal conductivity equation can be reduced to the thermal equation of the two-phase zone model (4):

$$\Psi(T) \frac{dT}{dt} = \lambda(\varepsilon_V, \varepsilon_\sigma) \nabla^2 T + F(qL) \quad (4)$$

where Ψ - the dimensionless effective heat capacity, which takes into account the release of the latent heat of the phase transition; T - the absolute temperature; t - time; λ - coefficient of thermal conductivity; ε_V and ε_σ - the density characteristics of the hardened layer, defined as the volume fraction of the density and the fraction of the hardened layer in a flat section, respectively; $F = \alpha qL$ - the intensity of the volumetric heat source associated with the power of the plasma effect

at different depths y of the powder layer. Here, q_L and α - the plasma radiation energy flux density and the absorption coefficient of light radiation in the local volume of the hardened layer, respectively. The coefficient α depends on both the temperature and the phase composition of the local volume, determining in the model the change in the penetration depth of plasma radiation during particle melting and changes in the morphology of the body being hardened.

The vapor-gas layer prevents electrolyte from getting on the overheated surface. This leads to a slowdown in the cooling rate, which excludes the formation of thermal (hardening) cracks. As a result, the service life of the steel is increased.

A mathematical model was developed to describe the change in the key parameters of the EPM hardening process, namely the heating temperature T . The logarithmic dependence of temperature T on the main factors is expressed by the following regression equation, with time expressed as t (5):

$$\ln(T) = A \cdot k1 \cdot \ln(t_{heat}) + B \cdot k2 \cdot \ln(U) + C \cdot k3 \cdot \ln(t_{cool}) \quad (5)$$

Parameter D modes are excluded because the sample melts from the plasma temperature. Coefficients for equation (5) were found using logarithm in the Deductor Studio Academic program. Then the equation (5) of the dependence of the heating temperature on the heating time, cooling time and voltage was transformed into a power law (6):

$$T = 4.5 \cdot t_{heat}^2 + 4.8 \cdot U - 25 \cdot t_{cool} \quad (6)$$

where T is the temperature of steel heating, t_{heat} - the heating time, t_{cool} - cooling time in the flow of electrolyte, U - voltage [22].

The experimentally determined optimal modes of steel hardening by the EPM method ($t_{heat} = 4$ sec., $t_{cool} = 4$ sec., $U = 200$ V) correlate well with the established relationship (6).

It is in this mode that the sample/part is heated to temperatures of 840–860°C, that is, sufficient for the cathodic phase transformation of steel 20KH (207).

The microstructure of a 20KH (207) steel sample treated with an electrolytic-plasma modification indicates the presence of acicular quenched martensite as shown Figure 7.

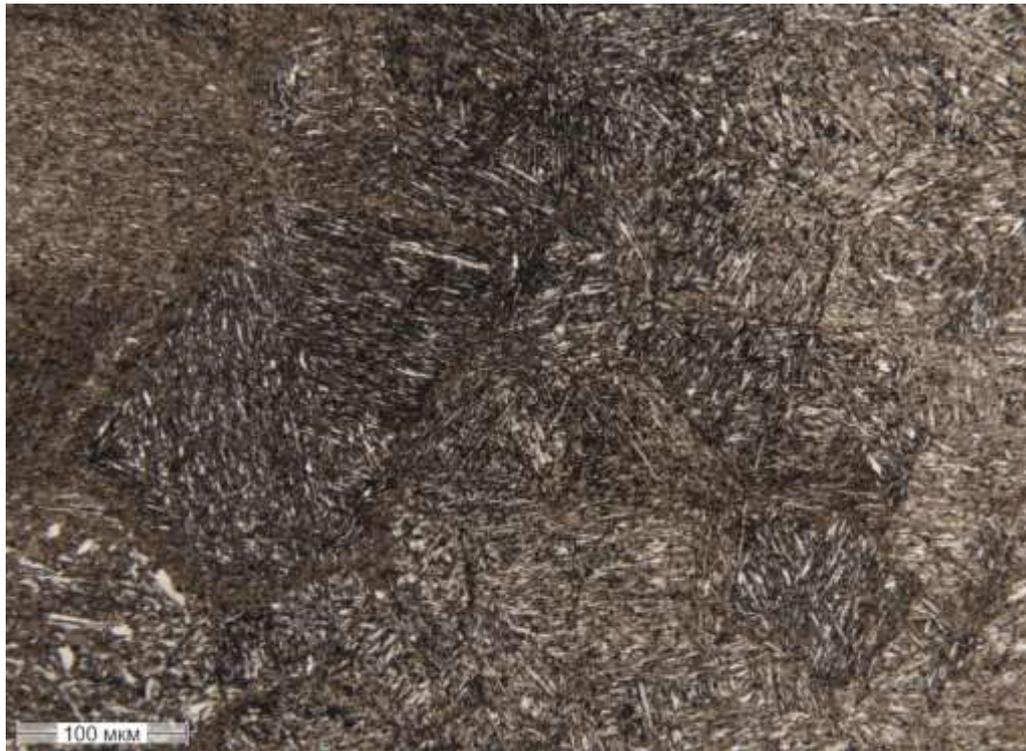


Figure 7. Microstructure of the surface layer of steel 20Kh after electrolytic-plasma modification, $U=200$ V, $t=4$ min.

Under traditional production conditions, the saturation of the surface of low-carbon steel with carbon and a similar microstructure is obtained over a significantly long period of time with high energy costs. It should be noted that the EPM was carried out in cyclic processing for a total of 4 minutes. The interval of plasma heating is 4 seconds, quenching in an electrolyte flow is 4 seconds and, accordingly, the number of cycles is 30. Previously, studies were published on the experimental determination of the optimal modes of EPM processing and on the saturation of metal with carbon from the decomposition ions of the Na_2CO_3 components of the electrolyte and the anode. Diffusion saturation of steel with carbon and other elements is possible not only from the electrolyte, but also from the elements of the anode, for which the stainless steel 12Kh18N10T GOST 2590-2006 (X6CrNiTi18-10) is used [23].

There are models about the positive effect of the electric field in the cathode surface on the transfer of carbon anions as shown Figure 8. The requirements for the composition of electrolytes for EPM are determined by several conditions. It is necessary to ensure that the parts are heated to temperatures sufficient for effective diffusion of carbon. The solution must have a minimum electrical conductivity that ensures the creation of an EPM. The saturating capacity of various electrolytes is determined by the decomposition reactions of carbon-containing components and the adsorption of the products of this decomposition on the treated surface [24].

The electrolyte circulation rate determines the intensity of stirring and cooling and quenching of the sample [25]. The regularities of heat transfer in the near-cathode zone, depending on the hydrodynamic conditions in the conical nozzle, significantly affect the features of the release and transfer of saturating components to the surface of the workpiece. These regularities also depend on the compositions of electrolytes, which, together with the processing modes, determine the properties acquired by materials after EPM.

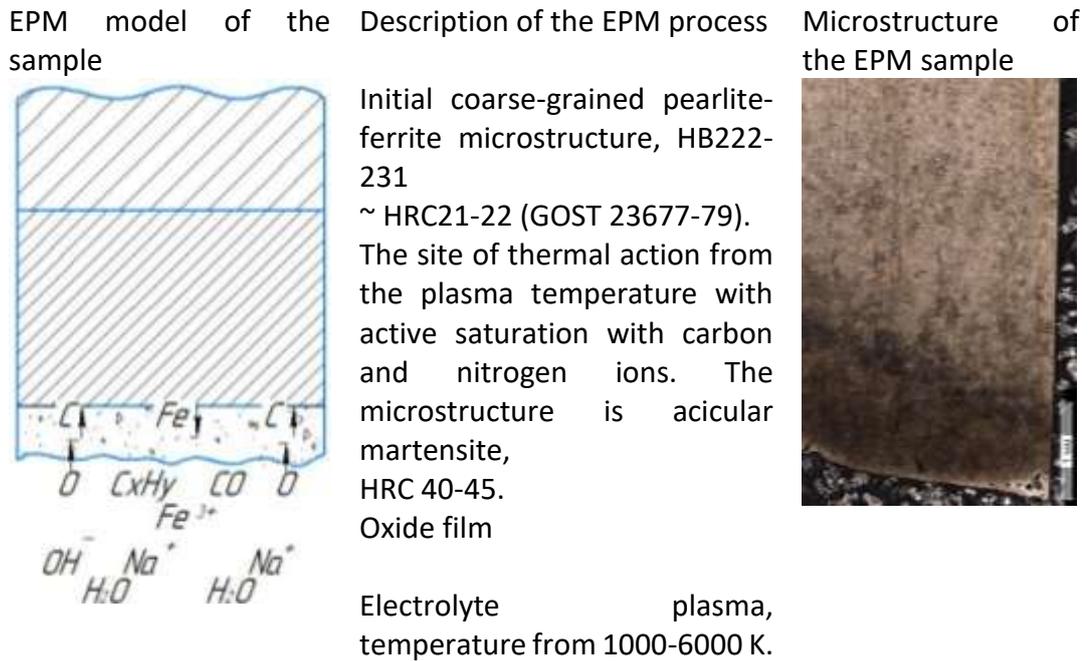


Figure 8. Model of the electrolytic-plasma treatment of the sample / part surface.

A highly concentrated plasma temperature forms on the surface of the steel substrate creating a with a high carbon concentration gradient. A typical surface investigated from micro-hardness variation after EPM processing is as shown Figure 9. Cyclic quenching in the electrolyte flow creates a zone of thermal effect on the steel microstructure. In this section, a phase transformation occurs with the formation of fine-acicular martensite. There are no clearly defined boundaries of the sections, but the total depth of the hardened layer reaches 2 mm, which smoothly goes over to the original pearlite-ferrite structure [26].

In view of the small size of the sample with a small thickness, by varying the applied voltage, it is possible to promptly control the temperature of the cathode - the workpiece being processed in the range from 400 to 1100°C [27], which is characterized by a nearly linear dependence of the temperature on the value of the applied voltage [28]. High rates of electrolytic-plasma heating not only shorten the time to reach the specified temperature of the sample / part, but also accelerate the formation of diffusion layers, having a beneficial effect on some stages of the process. Slow heating creates conditions for grain growth, while high-speed heating may increase the temperature to accelerate diffusion.

To determine the depth of the hardened layer during EPM, we measured the microhardness from the cross section of the sample with a step of 200 μm. The results of measuring the microhardness made it possible to reveal an increase in surface hardness relative to the initial value ~ 3000 till ~7500 MPa as shown in Figure 10. The distribution of hardness after EPM treatment completely correlates with the distribution of carbon concentration in steel 20Kh (207) and corresponds to

hypoeutectoid steel, which is consistent with saturating ability electrolytes [29]. This is due to the maximum total concentration of carbon, which form a supersaturated solution in austenite at saturation temperature and martensite after quenching.



Figure 9. A surface on which measurement of the cross-section microhardness after EPM was conducted.

Cathodic cyclic hardening of structural steels by EPM in an electrolyte containing 10% soda ash at 840-860 °C makes it possible to double their hardness in 4 minutes of treatment, which is much less than when carburizing on a solid carburizer followed by quenching. In addition, the possibility of reducing the time for heat treatment is shown, which in turn reduces energy consumption for heating an industrial furnace and, in general, the cost of manufacturing a part is reduced.

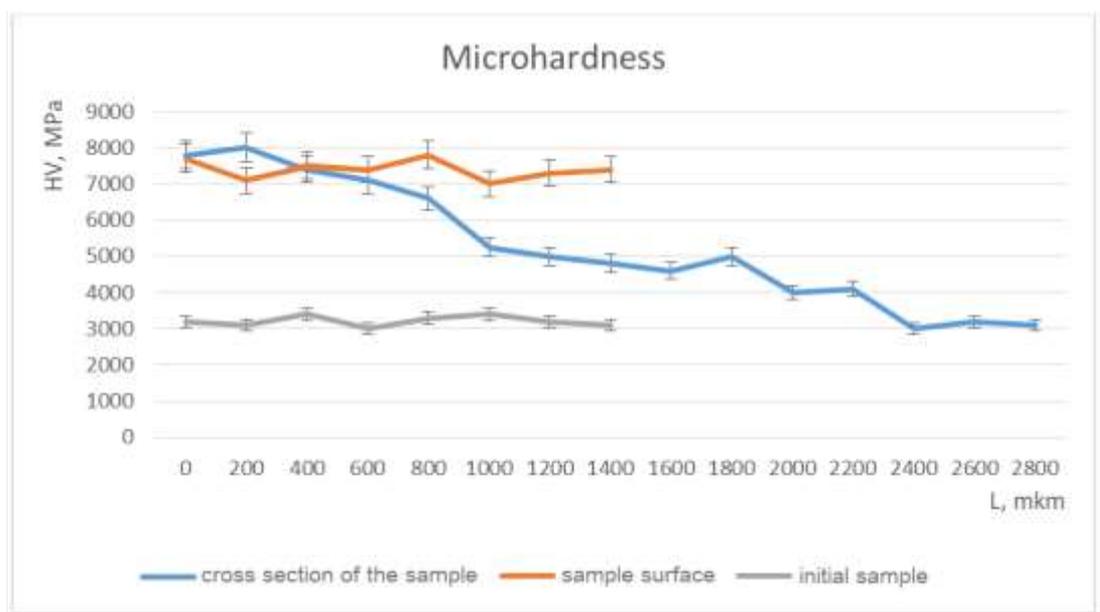


Figure 10. Comparative graph of the surface hardness of steel 20KH (207).

To exclude potential calcination and increased brittleness, it is recommended that parameters EPM with cyclic quenching by electrolyte flows is determined empirically and confirmed by the calculation as part of the planning of experiments [30]. The introduction of the relatively new EPM technology will provide an opportunity to obtain new information about the laws of nature

of electrolyte-plasma hardening. From the point of view of practical use, the EPM mechanism is necessary for a reasonable choice of processing modes and electrolyte compositions, as well as for creating the necessary equipment.

Conclusions

The results of our experimental electrolytic-plasma modification (EPM) investigation of a low-carbon alloy steel 20KH (207) and a mathematical model using thermal conductivity equations and regression analysis relating the key parameters of the hardening process, shows substantial promise for EPM as a surface hardening process for steels. The characteristic of the EPM treatment in our investigation indicates that the heating time of the workpiece from the high-temperature electrolyte plasma is 4 minutes, which is significantly less than the time for traditional carburizing followed by quenching. Our EPM surface hardening investigation further revealed that the sample surface has a shallow layer on it, which is a carbon-saturated hardened layer of martensitic origin. This layer smoothly transformed the initial pearlite-ferrite structure. We also established that the surface microhardness of the 20KH (207) steel sample after EPM increases by a factor of 2 relative to the initial state. Our Investigation further confirms the EPM as competitive process in terms of cost, processing time and energy efficiency, as a surface hardening process compared to the traditional heat-treatment surface hardening processes for steels that need to be updated for efficiency and competitiveness.

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Competing interests

The authors declare no conflict of interests.

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