

Investigating the effect of electrospark alloying parameters on structure formation of modified nitrogen coatings

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Abstract. The quality parameters of surface layers synthesised using electrospark alloying (ESA) technology were analysed in this paper. The main focus was on the influence of equipment energy parameters on structure formation, specifically the effect of discharge energy and productivity. Microstructural analysis of the modified surface of C40 steel after nitriding by ESA using a paste containing nitrogen compounds injected into the interelectrode gap was conducted. The layer structure for all studied ESA parameters includes three areas: the upper “white layer”, the diffusion zone below it, and the substrate. The roughness of the surface is $R_a \sim 0.9 \mu\text{m}$ at low discharge energy $W_p = 0.13 \text{ J}$ and $R_a \sim 6 \mu\text{m}$ at $W_p = 3.4 \text{ J}$. The microhardness, continuity, and surface roughness of the layers varied with W_p . The influence of ESA productivity on the structure was studied. The thickness of the hardened layer and the diffusion zone, as well as the microhardness and continuity, are affected by reduced productivity. For the same discharge energy, the thickness of the hardened layer increases by 10-18% with a decrease in productivity compared to the classical mode. Studies have shown that productivity loss has a positive effect on the quality characteristics of the coatings produced by the ESA process.

Keywords: electrospark alloying; discharge energy; productivity; coating; structure.

1. INTRODUCTION

As a result of scientific and technological progress, the quality of critical parts of dynamic equipment (pump and compressor units, turbines, centrifuges, etc.), which limit their reliability and durability, must be improved. The requirements for the impact strength and ductility of their base and the protective properties of their surface layers increase with their operating parameters (speed, temperature, pressure, radiation exposure).

For example, impulse seal designs with self-adjusting gaps are the most promising components for sealing pump shafts in nuclear power plants [1, 2]. The problem of ensuring the operating characteristics of their working friction surfaces is important [3, 4]. A promising direction for increasing the service life of products is the formation of a surface layer in contact with the external environment with improved operational properties. The most effective and modern solution to this problem is the use of surface modification technologies with concentrated energy and matter flows. These technologies make it possible to obtain protective and restorative coatings with a range of improved physical, mechanical, and operational properties.

2. LITERATURE REVIEW

Today, there are a large number of technologies that can improve the quality parameters of surfaces: increase hardness and wear resistance by applying metal-ceramic coatings [5–7], surfacing coatings of composite materials [8, 9], reinforcing with tungsten carbide [10], chromium in electrolyte [11], etc. In addition, among the technologies aimed at ensuring the operational properties of surfaces and increasing the reliability and durability of parts, those that ensure the required geometry of the product surface layer at the design stage should be highlighted. Thus, a lot of work is devoted to the study of centrifugal scattering devices [12, 13], gravitational transport [14], and cylindrical surfaces of parts [15, 16].

Multilayer coatings that combine lubricating and anti-wear properties can be a promising way to increase the wear resistance of friction pair surfaces [17, 18]. Such coatings can be combined coatings, synthesised by the method of electrospark alloying, combining hard wear-resistant and soft antifriction materials [19, 20].

Electrospark alloying (ESA) of metal surfaces is a phenomenon of electrical erosion and polar transfer of anode material to the cathode during pulsed discharges in a gas environment [21, 22]. The ESA process begins when the anode approaches the cathode and when the distance between them is equal to the break-down distance, a spark discharge of $10^{-7} - 10^{-8} \text{ s}$ begins to develop, which in many cases continues

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and ends after the electrodes come into contact. Local melting and evaporation points are then formed on the surface of the electrodes after the interelectrode area is broken up. The result is the electrical erosion of the electrodes. The preferential transfer of anode material to the cathode ensures the formation of a surface layer on the cathode with the specified performance characteristics and physical and chemical properties. Since the process of transferring anode material takes place in a gas environment, chemical compounds may be formed, as a result of which the particles separated from the anode may not look like anode material. Then the anode (electrode) moves away from the cathode and a layer of anode material remains on the surface of the latter, which is firmly bonded to the cathode. In turn, the value of the changed characteristics is determined by the ESA process parameters, and certain physical and chemical properties will depend on the composition and properties of the electrode materials (EM).

The analysis of literature data on electric spark alloying of carbon, alloyed, and high-speed steels showed that the authors use a wide range of EMs, mainly high-melting compounds [23].

In practice, hard alloys based on tungsten and titanium carbides are mainly used as alloying electrodes, but they do not always fulfil the surface and EM for ESA, as they are expensive, and do not transfer well to the surface. In addition, hard alloy coatings increase hardness and wear resistance, but they do not provide corrosion resistance, heat resistance, special tribotechnical properties, etc.

The environmental and economic issues of electrode production should be noted separately – the existing methods used at metallurgical and chemical plants are in most cases environmentally hazardous.

This unjustifiably limited number of materials in the literature also does not allow us to fully reveal the capabilities of this progressive method.

In this regard, ESA coating technologies, such as cementation, sulphidation, borisation, metallisation, and their combinations, are worthy of attention [24–27]. Relatively cheap materials and technologies for their production are used as EM.

The researchers applied coatings to both the front and back surfaces, as well as the inner and outer surfaces of tools and machine parts, in their experiments to strengthen cutting tools and machine parts. They proposed new methods of surface modification using ESA with the possibility of transferring conductive materials to the treated surface. The source of the alloying element is a special technological environment (STE). Before doping it is applied in the form of a paste on the treated surface. The use of STE makes it possible to obtain surface structures with unique physical, mechanical, and tribological properties at the nanoscale. In ESA, STE can be used to produce single-component coatings such as alitisation [24], cementation [25], nitriding [26], and multicomponent coatings [19, 20].

One of the ways of intensifying and unifying ESA processes is the use of aluminium, copper, etc. as an intermediate sublayer [28]. An additional sublayer before ESA with hard materials such as hard alloys, metal ceramics, tungsten, chromium, etc. helps to improve the quality of the coating, increases the continuity of the coating, reduces the roughness of the treated surface,

and increases the thickness of the hardened layer. The interlayer performs an auxiliary role, the diffusing element saturates the surface through the interlayer, mixing of the interlayer and alloying electrode materials is possible and the adhesion between the coating and the substrate is improved. In addition, ESA with aluminium creates intermetallic phases that contribute to a significant increase in microhardness (up to 12 000–14 000 MPa) [24]. A 10–15 μm layer between the substrate and the deposited electrode material has been shown to have a positive effect on the coating properties in service, including cracking and brittle fracture under friction [28]. The ESA method using a suitable electrode or other surface modification methods such as laser alloying, PVD, etc. are recommended for obtaining a sublayer. The use of surface treatment methods with concentrated energy flows, including ESA, promotes the intensification of diffusion processes and improves the quality of the coating.

Thus, using the application of STE containing nitrogen-containing components to the hardened surface, nitriding of steel parts surfaces can be conducted, and preliminary ESA of the treated surface with aluminium can be useful for improving the quality parameters of their surface layer.

The analysis of the ESA with both the compact tool electrodes and the STE showed that the quality parameters of the formed surface layers were studied mainly in dependence on the main parameter of the equipment operation – the discharge energy (W_p). Simultaneously, the value of machining productivity, i.e. surface area treated per unit time, was not considered. It is also known that for most materials the thickness of the layer formed on the cathode (part) is limited.

In [19] it is argued that during the ESA process, the deposition of anode material slows down and stops over time. This is attributed to the formation of oxides and nitrides in the surface layer. These prevent the interaction of the newly deposited anode material on the cathode with the previously deposited material and lead to embrittlement and destruction of the formed layer. As a result, the transfer intensity is maximum in the first few minutes of the process, then it decreases. Finally, at certain values of W_p , the transfer is replaced by erosion of the already deposited layer and the increase becomes negative.

It should be noted that the presence of STE between the anode and cathode, mainly in a pasty (liquid) state, changes the magnitude of spark discharge and the mass transfer process differs significantly from traditional.

It is known [29, 30] that the quantitative and qualitative characteristics of the surface layer formed in the ESA process depend on the power of the pulsed spark discharges; the duration of the treatment; the type of electrode materials; the composition and condition of the environment in which the treatment is conducted; the relative position and movement of the electrodes; the use of STE; the position of the anode and cathode electrodes (anode at the top, cathode at the bottom and vice versa); the shape of the electrodes; the alloying technology (manual vibrator and mechanised); the number of electrodes in simultaneous contact, and others. The ESA method is therefore multi-parametric. The effect of pulsed discharges on the surface of conductive materials during ESA leads to complex structural and phase transformations which subsequently determine a wide range of properties

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of these surfaces. The most studied processing parameter is the effect of pulsed spark discharge power or discharge energy on the structure, and phase state and properties of ESA coatings. As the discharge energy increases, the intensity of material transfer to the electrode tool increases, and maximum cathode growth is achieved in a relatively brief time. Other processing parameters, such as processing time or processing power per unit area, have not received sufficient attention.

The literature usually provides recommended (optimal) treatment durations depending on the discharge energy. In our previous studies, we also used traditional treatment modes for ESA [19,20,24]. However, during the development of new ESA technologies using STE, the authors have concluded that productivity and discharge energy are important parameters of ESA processing that quantitatively and qualitatively influence the parameters of the formed surface layer. The paper [31] shows that during ESA aluminisation, a decrease in the productivity of the ESA process contributes to the deterioration of the quality parameters of the resulting coatings.

Therefore, this paper aims to study the structural state of nitrogen-containing coatings obtained by the ESA method with STE under different conditions (discharge energy and productivity) of alloying.

3. MATERIALS AND METHODS

In order to evaluate the effect of productivity on the quality parameters of the coatings obtained, we studied the classical processing modes (Table 1), as well as modes with reduced productivity by a factor of two, three, and four. That is, the processing time (τ) per unit area (labour intensity of the ESA process) was increased by two, three, and four folds (Table 2).

Table 1
Traditional processing modes [31]

Discharge energy (Wp), J	0.13	0.52	3.4
Productivity, cm ² /min	0.5–0.6	0.8–1.0	1.7–2.0

Table 2
The modified processing modes studied

Discharge energy (Wp), J		0.13	0.52	3.4
Productivity, cm ² /min	1st option	~0.3	~0.6	~1.0
	2nd option	~0.2	~0.3	~0.5
	3rd option	~0.1	~0.2	~0.3
Intensity (τ), min/cm ²	1st option	~3.3	~1.7	~1.0
	2nd option	~5.0	~3.3	~2.0
	3rd option	~10.0	~5.0	~3.3

The ESA nitriding process was conducted as follows. An aluminium sub-layer was formed on C40 steel specimens (EN 10277-2: 2008) using the ESA method with an aluminium electrode (ENAW-1050A, diameter of 3.0 mm). Samples were

15×15×8 mm in size. An Elitron-52A unit and a discharge energy of 3.40 J were used [26]. Next, a nitrogenous paste based on petroleum jelly containing urea (urea powder ~ 90% Vaseline ~ 10% mass.) was applied to the treated surface in a continuous layer of 0.2–0.3 mm. The alloying was then conducted with a metal electrode with a diameter of 3.0 mm, made of C40 steel according to the modes in Tables 1 and 2.

The roughness of the treated surface was determined using a profilograph-profilometer. The preparation of the specimens for metallographic studies was conducted according to the standard method. A Neofot-2 optical microscope was used. Hardness was determined using a PMT-3 device. Microhardness was tested at a load of 0.05 N according to GOST 9450-76. The continuity of the coating was determined by the metallographic method with statistical data processing.

X-ray diffraction studies were performed on a DRON-3 diffractometer in $CiK\alpha$ radiation. The voltage and anode currents were 30 kV and 24 mA, respectively. X-ray diffraction patterns were taken by step-by-step scanning with an exposure for 2 s at every point.

4. RESULTS AND DISCUSSION

Figure 1 shows the optical microscope microstructures of the surface layer of the samples after surface modification according to the classical regime (discharge energy of 0.52 J and productivity of 0.8–1.0 cm²/min).

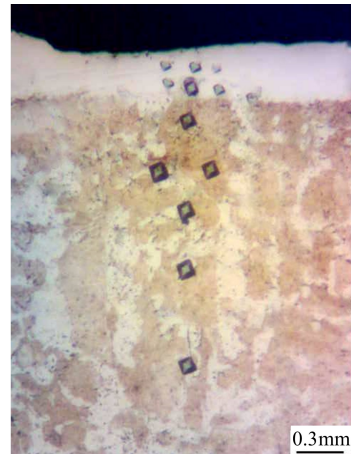


Fig. 1. Microstructure of the nitrided surface layer obtained by ESA with a discharge energy of 0.52 J and traditional productivity (0.8–1.0 cm²/min)

According to ESA using traditional processing performance, the coating consists of three areas – “white layer” (WL), diffusion zone, and substrate.

In [29] it is shown that the WL is formed from the liquid phase by mixing with the substrate and interpenetration diffusion. It usually consists of the alloyed electrode material and the elements of the inter-electrode medium. The diffusion zone is formed as a result of the diffusion of chemical elements of the anode materials and interelectrode medium into the cathode material (surface) and also as a result of thermal influence.

The diffused zone of ESA coatings merges smoothly into the substrate structure (Fig. 2). Metallographic analysis, as well as local micro-X-ray spectral analysis, revealed that ESA coatings are characterised by a continuous coating-substrate transition, which is an important advantage of electrospark alloying compared to other methods [26,29].

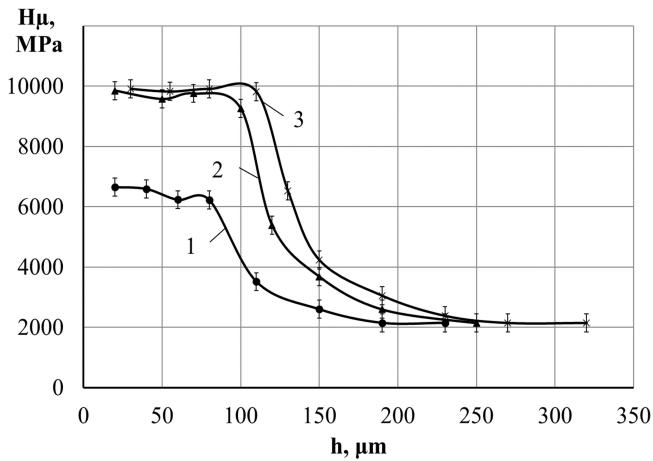


Fig. 2. Surface microhardness distribution after ESA at discharge energy: 1 – 0.13; 2 – 0.52 and 3 – 3.40 J and traditional productivity

WL is placed as a light band along the boundaries of the substrate. The diffusion zone is formed underneath the WL. The microhardness of the doped modified layer decreases from the surface and further into the substrate. The maximum hardness

values are 9910 MPa at the WL and 4870 MPa at the diffusion zone at discharge energy 3.40 J (Table 3).

This research presents an analysis of the change in phase composition of the modified surfaces. Figure 3 shows the diffractograms of the surface samples after alitisation with an aluminium electrode and nitriding with an aluminium sublayer. The discharge energy was 0.52 J, and the productivity was 0.8–1.0 cm²/mm. Alitisation results in the formation of solid solutions of aluminium in iron and aluminium oxides. As the discharge energy increases, free aluminium is deposited [24]. The phase composition of the surface changes during the nitriding of a steel surface according to the technology presented here.

Studies of the phase composition of the coatings obtained on C40 steel have shown that when nitrided using the ESA method and traditional productivity, it is represented by ferrite (BCC solid solution) and cubic iron nitride (Fig. 3). Obviously aluminium is dissolved in the ferrite and nitride. There is also the possibility of a sublayer of aluminium-containing phases between the substrate and the nitrogen-containing layer.

In steel nitriding, pre-ESA with aluminium increases the thickness, microhardness, and continuity of the WL, while the roughness changes insignificantly. In addition, X-ray diffraction analysis proves that nitriding by the ESA method using nitrogen-containing paste as an interelectrode area is effective. Such technology can be used for hardening surface layers of critical parts and their elements of compressor and pumping equipment. However, the proposed technology requires improvement with regard to the assurance of high productivity of the ESA process. Therefore, we investigate the influence of ESA pro-

Table 3

Parameters of the modified layer after nitriding by ESA

Discharge energy, J	Productivity, (Q) cm ² /min	Thickness of the hardened layer, μm	Microhardness, MPa		Roughness, Ra, μm	Continuity of the WL, %
			WL	diffusion zone		
Classical technology (according to the modes of Table 1)						
0.13	0.5–0.6	155	6650	4350	0.9	85
0.52	0.8–1.0	165	9850	4550	1.3	90
3.40	1.7–2.0	230	9910	4870	5.9	95
1st option						
0.13	0.3	170	7100	4500	1.0	90
0.52	0.6	180	9980	4570	1.4	95
3.40	1.0	245	10 080	4970	6.1	100
2nd option						
0.13	0.2	180	7130	4540	1.1	100
0.52	0.3	190	9990	4630	1.4	100
3.40	0.5	255	10 100	4980	6.0	100
3rd option						
0.13	0.1	185	7140	4540	1.0	100
0.52	0.2	195	10 010	4590	1.4	100
3.40	0.3	260	10 110	4920	6.2	100

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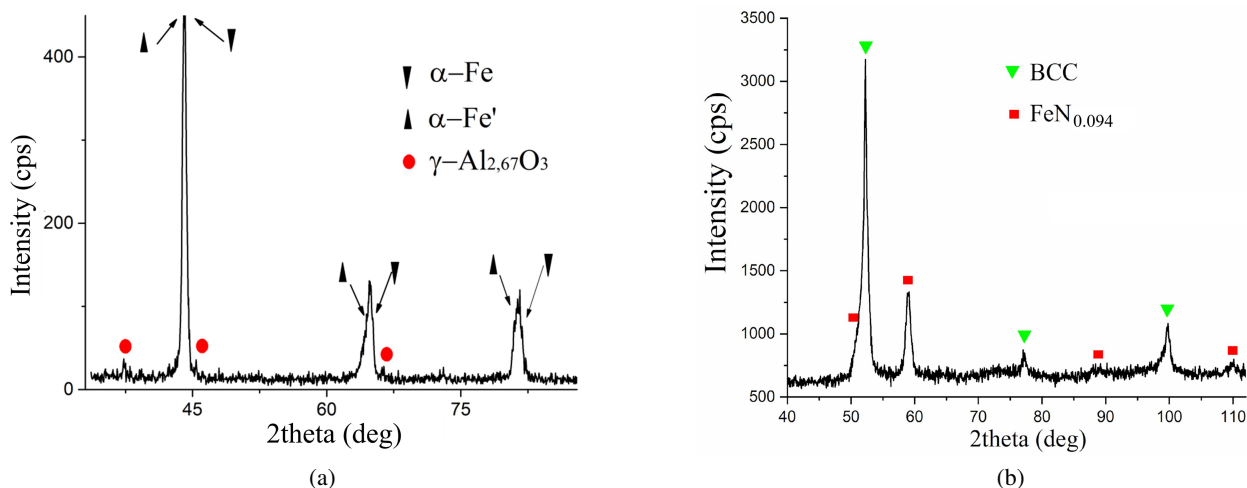


Fig. 3. Surface layer X-ray diffraction results of C40 steel samples after ESA at 0.52 J and traditional productivity: (a) after alitisation; (b) after nitriding with an aluminium sublayer

ductivity on forming the structure and properties of nitrided layers.

Figure 4 shows the microstructures of the surface layer of a C40 steel sample after nitriding by the ESA method, at productivity according to the 1st, 2nd, and 3rd options, respectively, Table 2. Analysis of the microstructures showed that, as in the case of alloying by traditional modes, it consists of a surface “white layer” (WL), which is identified by a single light strip, under which there is a diffusion zone with a structure of incomplete phase recrystallisation and the substrate. The typical characteristic of ESA coatings is a step-down in hardness from the top layer to the substrate, which describes the ESA method as one that allows the formation of coatings with high adhesion. Changing the processing parameters does not affect the characteristic features of the electrical spark coating microstructure but affects the quality indicators (Table 3). With the increase in the energy modes of processing, the thickness of the WL and the diffusion zone increases. In addition, at fixed discharge en-

ergy, with a decrease in ESA productivity, the microhardness, geometric parameters of the formed structural zones, and the continuity of the surface layer increase, but the surface quality deteriorates, which leads to an increase in its roughness.

Figure 5 shows the dependence of hardness and layer thickness on the alloying modes - productivity and discharge energy. Studies have shown that a decrease in productivity has a positive effect on the quality characteristics of coatings obtained by the ESA method. However, the intensity of the growth of indicators slows down with an increase in the degree of productivity reduction.

Thus, for the same discharge energy, the thickness of the hardened layer increases by ~ 10% with a two-fold decrease in productivity (1st option), by ~ 15% with a three-fold decrease in productivity (2nd option), and by ~ 18% with a four-fold decrease in productivity (3rd option) compared to the classical mode. The microhardness changes less. At $W_p = 0.52$ J and a change in productivity according to Table 2, the microhardness

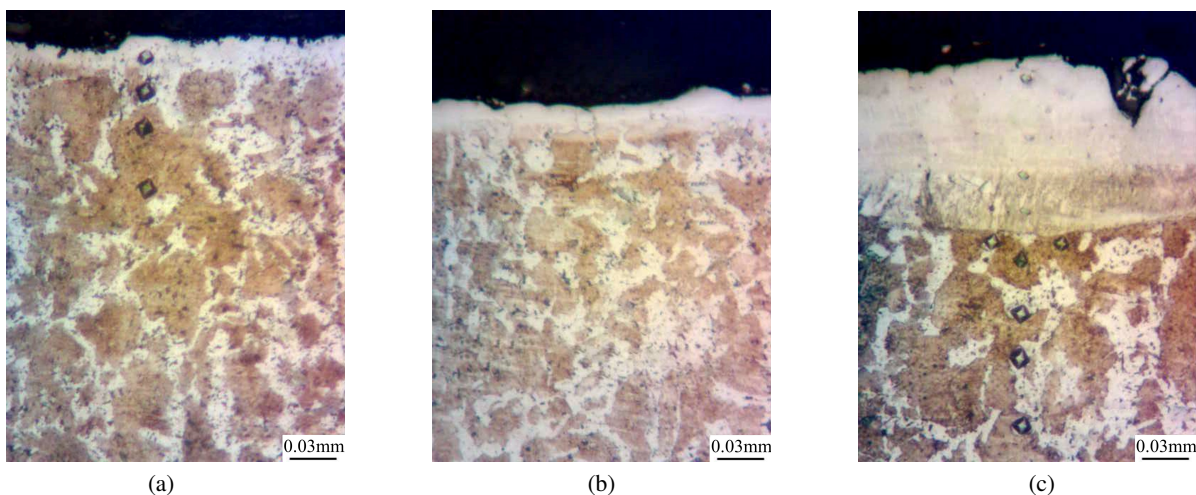


Fig. 4. Microstructures of the nitrided surface layer of C40 steel samples under ESA at discharge energies: (a) 0.13; (b) 0.52 and (c) 3.40 J. Performance values for the 2nd option (Table 2)

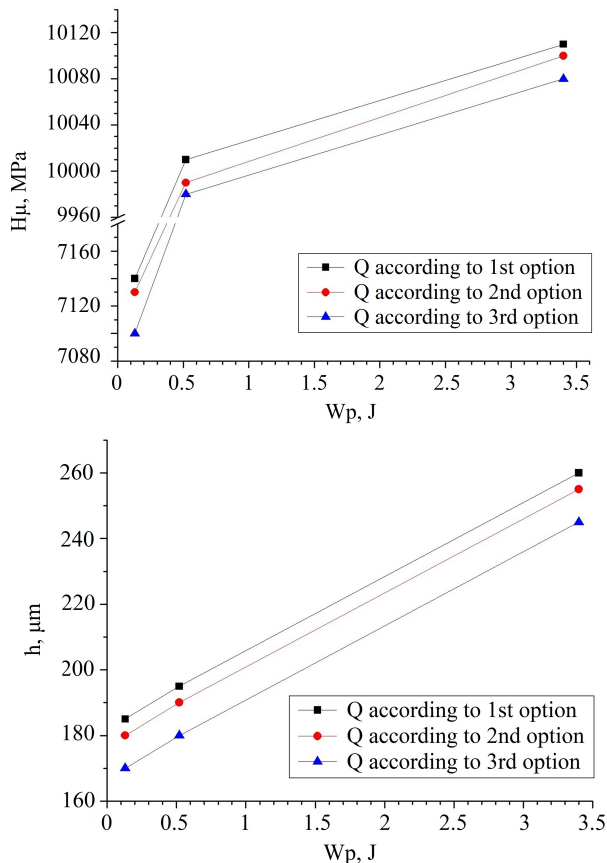


Fig. 5. Hardened layer parameters (H_{μ} – microhardness, h – hardened layer thickness) as a function of discharge energy (W_p) and ESA process productivity (Q) when nitriding C40 steel. The productivity values are shown in Table 2

of the hardened layer is 9980, 9990, and 10 110 MPa for the 1st, 2nd, and 3rd treatments, respectively.

It can be assumed that there is a limit value of productivity at which the quality parameters of the surface layer will not change. We obtained a similar character of dependence of the increase in the surface layer indicator – ESA process indicator when studying the effect of discharge energy on the hardness, layer thickness, and coating continuity during alitisation by the ESA method [24].

Thus, there is a limit value of the ESA process indicator, for example, discharge energy or ESA process performance, above which the coating is destroyed or has no positive effect. To determine the limit value of the ESA productivity for a given set of electrode materials and the composition of the saturation paste, additional research is needed.

5. SUMMARY AND CONCLUSION

The authors present their conclusions based on the analyses described in this manuscript as follows:

1. The influence of traditional ESA productivity values on nitrided coatings structure, phase composition, and microhardness was investigated. The coatings consist of a hardened "white"

layer on the surface, followed by a diffusion zone and then the substrate.

The coating obtained has a gradual reduction in hardness from the top layer to the substrate. The microhardness of the hardened layer is 6650 MPa and 9910 MPa, respectively, at a discharge energy of 0.13 and 0.52 J. The phase composition is represented by ferrite (BCC solid solution) and cubic iron nitride. Therefore, the ESA nitriding technology is effective in providing nitrogen saturation of the steel surface.

2. The effect of ESA productivity on modified structure and microhardness was investigated. Productivity decreased by a factor of two, three and four. Changing the productivity values does not affect the microstructural characteristics of the electrospark coatings, but it does affect the quality parameters. The thickness of the "white" layer and the diffusion zone grows with the energy regime. At the same discharge energy and reduced ESA productivity, the microhardness, geometric parameters of the formed structural zones, and the continuity of the top layer rise. At $W_p = 0.52$ J and a change in productivity, the microhardness of the hardened layer is 9980, 9990, 10 110 MPa for the 1st, 2nd, and 3rd treatments, respectively. The roughness of the surface is $R_a \sim 0.9 \mu\text{m}$ at low discharge energy $W_p = 0.13$ J and $R_a \sim 6 \mu\text{m}$ at $W_p = 3.4$ J.

3. Research has demonstrated that a reduction in productivity can have a positive effect on the quality parameters of ESA coatings. However, the degree of improvement in these parameters diminishes as the level of productivity reduction rises. The thickness of the hardened layer increases by $\sim 10\%$ with the 1st option, by $\sim 15\%$ with the 2nd option, and by $\sim 18\%$ with the 3rd option of productivity compared to the traditional values. The microhardness changes less. It can be assumed that there is a limit value of productivity at which the quality parameters of the surface layer will no more change.

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