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Nanostructure Formation During Electrosark Alloying

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Abstract. There have been considered the coating structure modification after electrosark alloying (ESA) by the Mo-electrode using a paste with carbon nanotubes. It was showed that the regimes and composition of the paste influenced the microstructure, continuity, roughness, and hardness of the coatings. The microstructures after the ESA of the Armco iron were studied. The use of higher discharge energy in ESA leads to an increase in surface roughness, coating thickness, and coating continuity. The use of higher discharge energy during ESA results in an increase in surface roughness, coating thickness, and coating continuity. The microstructures reveal nanoscale phases from 40 to 60 nm, which are evenly distributed in the coatings. The addition of ARKEMA nanotubes increases the hardness to 608 HV. The results of local X-ray diffraction analysis showed that carbon is concentrated on the surface, while molybdenum is distributed homogeneously in the coating. The addition of nanotubes to the paste before ESA improves hardness and continuity. The effect of nanotubes on the structure and properties of coatings is positive.

INTRODUCTION

The solution to the problems of improving reliability, efficiency, and service life of parts requires the use of materials capable of working in various conditions. Many parameters of the product determine the condition of the surface layer of the material from which it is made. It is obvious that the use of scarce and expensive structural materials in the entire volume of the product is inexpedient. It is known that the losses associated with the failure of machines and other equipment due to the wear of certain parts are great. There are numerous traditional methods of surface treatment, which have their own features, advantages, and disadvantages.

Most of the known methods of surface treatment are energy-consuming. For example, the most commonly used method of strengthening machine parts is chemical heat treatment (CHT). It involves saturating the surface of metals with various elements, such as carbon, nitrogen, chromium, etc. CHT has significant disadvantages. These include volumetric heating, which leads to changes in the structure of the part; deviation of the part from its original geometry (curvature and warping); bulky and expensive processing equipment; low productivity (cementation to a depth of 0.1 mm takes 1 hour); longer process time and using energy-consuming equipment, etc. In addition, the CHT resource is now exhausted.

In recent years, methods of surface modification by concentrated flows of energy and matter have become increasingly important, one of which is the method of electrosark alloying (ESA). Electrosark alloying of metal surfaces is a process of electrical erosion and polar transfer of anode material to the cathode during pulsed discharges in a gas environment [1]. Its advantages are the ability to process a local surface, low power

consumption, no deformation of the treated surface, strong adhesion of the deposited material to the substrate, the ability to automate the processing, etc. The ESA method allows for cementation, nitriding, and metallisation, i.e., processes similar to CHT, but with significantly lower costs.

Many studies have shown that the highest level of properties of materials when treated with concentrated flows of energy is achieved in the case of formation in the modified layer of sub-microcrystalline or nanocrystalline structure (average size of structural parameters is ≈ 100 nm). Dispersion of the structural elements of alloys in the nanostructural range leads to a number of unusual properties in alloys. The nature of the formation of such properties is associated with the extremely high surface development of nanoelemental structures, a large proportion of boundary regions per unit volume, and the peculiarities of the physical and chemical state of these regions. The creation and application of nanomaterials have now covered almost all high-tech fields of science and technology.

A promising area is the nanostructuring of the surface of products. First of all, this concerns the problems of friction and wear of contacting surfaces (micro- and nanotribology). formation of structures in the wearing surfaces, blocking the development of surface nanoflows of defects, which in some cases provides a twofold increase in the fatigue strength and wear resistance of materials, including when working in harsh conditions (aggressive environments, elevated temperatures).

Therefore, there is an interest and need to develop new surface-hardening technologies that are environmentally friendly and energy efficient. The use of ESA-based surface nanostructuring methods will increase the service life of parts.

ANALYSIS OF THE LATEST RESEARCH

One of the most promising areas of development of hardening technologies is the surface modification of working surfaces. The purpose of this treatment is to purposefully form a specified structural state of the material in the surface-modified layer, which causes a significant increase in performance properties. The type and optimal thickness of the layer, as well as the depth of penetration, depend on the modes of operation and the purposes of the parts and vary from parts of a micrometer to several millimeters. Strengthening the thin surface layers of parts made of ordinary structural materials allows for saving expensive alloy steels, non-ferrous metals, and other scarce materials, increasing the resource and reliability of mechanisms, reducing the energy consumption of production, successfully solving the problem of restorative repair in order to reuse worn parts, etc.

It is known that at performing the CTT process, having introduced due to diffusion, the particles of the substance are not able to penetrate too deeply into the material being processed and here to create high concentrations thereof on the surface of the part. In order to increase the depth of penetration of the particle, which is here penetrating into the surface of the part, it is necessary to provide that particle with high energy, and in order to obtain the large concentrations of the particles near the surface, it is necessary to maintain a low temperature of the part being processed.

New possibilities for affecting the surface of metals have emerged, involving the use of concentrated flows. The use of these methods affects not only the surface layers of the metal but also the more deeply layered ones (10 to 20 times larger than during implantation). The complex physical and chemical processes involved (irradiation with a pulse duration of 10^{-6} to 10^{-8} s, thermal shock, micro-explosions, ultrafast gradient heating and cooling at speeds of 10^8 to 10^{11} K/s) modify metal surfaces, giving them unique properties due to deep (sub-surface) amorphization and creation of a nanocomposite structure.

The production of nanocoatings with controlled parameters is the basis for the application of the modification method. The established technologies for producing nanostructured coatings are CVD and PVD. These methods allow for control of the structure parameters and thickness of coatings. Promising methods for the formation of nanocoatings are laser deposition, magnetron sputtering, sol-gel method, and others [4-7]. These methods have been widely used and are sufficiently described in the literature. It should be noted that traditional nanocoating processes require special expensive equipment that does not always meet the requirements. There is a requirement to develop new methods of surface modification that should be environmentally friendly and energy efficient, as well as to be applicable to engineering companies. The ESA method is promising [8, 9].

In [10, 11] was shown that some ESA modes can form a nanostructured layer on the surface. The authors identified possible mechanisms for the formation of nanostructures in ESA coatings. At the same time, regardless of the electrode materials, a modified layer with a fine polycrystalline structure was formed on the treated surface. For example, in [12] the authors obtained an ESA coating on 41Cr4 steel with a bronze electrode. It was shown that a microstructure with nano-sized grains from 10 to 60 nm was formed in the "white layer".

In [13], the authors found that changing the regimes and conditions of processing by the ESA method affects the structure and dispersion of phases in modified layers. The formation nanostructure or subfine-grained structure depends on the processing parameters and conditions. These include the use of electrical modes with low energy and pulse duration, increasing the specific processing time, using cathode materials with low thermal conductivity, as well as anode materials with low heat capacity, and deposition of anode elements on the cathode in the solid state.

The analysis of the research results showed that the mechanisms of nanostructuring in ESA are the modification of the coating structure by grinding the blocks of fine structure and increasing the density of dislocations, as well as the use of an alloying electrode consisting of nanocomponents.

Thus, grain grinding and dispersion of structural elements of alloys to the nanoscale and sub-microcrystalline range can be obtained by using technologies based on the ESA method. However, the influence of ESA operating parameters and the choice of electrode materials and atmosphere are still unexplored. Therefore, the formation of technological principles for the development of surface nanostructuring processes by ESA is important.

Traditional anode materials for the ESA process are cast alloys and hard alloys. New materials have been developed in the form of composites produced by powder metallurgy technology. To form a hardened layer on the treated surface, ultrafine particles are added to the anode material [14], sometimes nanoparticles [15-18].

The authors of [19] studied the process of structure formation in coatings on C35 steel. The alloying electrode used was a material comprised of tungsten carbide with the addition of Al_2O_3 nanoparticles. Studies have shown that ordered nanoclusters up to 30 nm in size are formed on the surface of the substrate. The authors noted that the introduction of 1 % Al_2O_3 into the carbide anode material (WC ~ 92 %, Co ~ 8 %) increased the efficiency of layer formation by up to 3 times. The microhardness of the coating also increased 3-4 times compared to untreated C35 steel.

Papers [17-19] present the methods of formation and the results of studying the structural-phase state and properties of ESA coatings. It is shown that an effective way to provide a high level of physicomechanical properties and reliability of parts that are subject to surface treatment is to implement the ESA method using doping materials with components of nanometre scale. The recommended value of nano-additives in the composition is 1-5% by volume.

The literature presents a sufficient number of papers on the use of new electrode material compositions and technologies for their production. One of the new ones is the self-propagating high-temperature synthesis (SHS) method. The method is popularly used to create composite materials that are recommended for use as alloying electrodes in ESA. The essence of the SHS process is the propagation of a chemical reaction zone in media capable of forming valuable condensed products. Powders, including powders of chemical compounds or nanopowders, are used as basic materials. However, the raw materials and synthesis technology are difficult and expensive, which limits the use of synthesized materials in the industry. The authors of [20-22] proposed a new method of forming coatings using the ESA method. It consists of creating a special process medium (SPM) between the anode and the cathode containing the necessary alloying components. STM is a paste that is applied to the treated surface. STM can contain nanometre-sized particles. This method can be used to form one- and multi-component, multilayer, nanostructured, etc. coatings [23]. The positive effect of using STM lies in the fact that the technology is affordable and easy to implement and allows for a combination of the required physico-mechanical properties of the coating, reliability, and durability of critical machine parts.

Polymers are widely used as a base for SPMs. To obtain a homogeneous distribution of the filler ("doping component") in the polymer, mechanical mixing technology is used. However, when used as a modifier of nanotubes, this technology does not provide the desired homogeneity result [24]. Given that the most common nanofiller in composites is carbon nanotubes, the issue of dispersion has become particularly important. The literature presents a significant number of works on this issue. Ultrasonic dispersion (UD) is an effective method. The advantage of this technique is due to the increased purity of the composites and the high quality of nanoparticle distribution. This method allows for the homogeneous distribution of carbon nanofillers in various solvents, low-viscosity epoxy oligomers, and some polymers. Paper [25] investigated the regimes of ultrasonic processing on the homogeneity of the nanofiller distribution in epoxy resin. It was shown that this method is effective for epoxy resins, avoiding the formation of defects due to the integrity of the nanofiller structure.

Papers [26, 27] show the advantage of the ESA surface modification method from the point of view of nanostructuring. The ESA process has been studied using epoxy resin with a nanofiller (two-dimensional boron nitride and zero-dimensional silica). However, the modes of processing and the rationale for the choice of raw materials need to be improved and further studied.

The purpose of this work is to study the structure and properties of coatings obtained by the ESA method using a paste based on epoxy resin with a filler in the form of nanotubes.

RESEARCH METHODOLOGY

Samples of Armco iron and high strength cast iron EN-GJS-600-3 were prepared for the study. Molybdenum and T15K6 hard alloy were used as doping electrodes. The electrodes were used in wire form. Table 1 shows the source materials investigated.

TABLE 1. Composition of SPM and doping modes ESA

Substrate	Stages of doping	Composition of SPM	ESA modes, W_p , J
Armco iron	1) ESA by Mo electrode,	multiwalled carbon nanotubes ARKEMA (0.2% by weight), matrix – epoxy resin Epoxy 510 without hardener	0.13
	2) using SPM,		0.52
EN-GJS-600-3 cast iron	3) ESA by Mo electrode		0.13
	1) ESA by T15K6 electrode,		0.52
	2) using the SPM,		
	3) ESA by T15K6 electrode		

The carbon nanotubes were dispersed in epoxy resin using the ultrasonic dispersion method. This method is based on the technology described in [28]. The composition of the SPM was chosen on an empirical basis. The need to obtain a homogeneous distribution of nanoparticles in a non-metallic matrix determined the choice of modes.

Samples were processed in three steps: first, the surface was doped with a molybdenum electrode (or T15K6 carbide) with 0.13 or 0.52 J discharge energy. SPM containing nanoparticles was applied to the surface in the second step. The surface was doped without waiting for the SPM to dry. ESA was performed with a molybdenum electrode (or T15K6 carbide). The discharge energy was the same as in the first step.

The electrospark alloying process was carried out using an ESA Elitron-22A unit. Discharge energies of $W_p = 0.13$ and 0.52 J were used. Armco iron samples without SPM were studied after alloying with a molybdenum electrode at 0.13 or 0.52J for comparison.

Surface roughness was measured using a profilograph profilometer. Special software was used to process the measurement results.

Metallographic analysis methods were used to identify structural components. The manufacture of grinds, surface preparation, and metallographic studies were carried out according to known methods [29]. Special reagents were used to chemically etch the microgrit to increase optical contrast and selective coloring. The reagent used to chemically etch the Armco iron was a 3-5 % solution of nitric acid in ethyl alcohol.

A scanning electron microscope, model SEO-SEM Inspect S50-B, equipped with an AZtecOne energy dispersive spectrometer with an X-ray detector - model MaxN20 (manufactured by Oxford Instruments plc), was used to study the microstructural parameters of the coatings. The Vickers hardness distribution in the surface layer and in the depth of the cut from the surface was analyzed using a NOVA 330/360 hardness tester (manufactured by INNOVATEST Europe BV, The Netherlands) in accordance with ISO 6507.

RESEARCH RESULTS

The surface topography of the processed samples was examined using an electron microscope. Analyzing the surface topography of the samples tested, it can be concluded that, as the discharge energy increases, homogeneous continuous coating formation is observed, but with increased roughness (Table 2). Nanoscale inclusions were formed in the coatings during the ESA process at $W_p = 0.52$ J. These inclusions are homogeneously distributed in the coating surface layers (Figure 1, d).

Consider the characteristics of coating structure formation when the Armco iron was processed by ESA with a molybdenum electrode using SPM with nanotubes. The ESA process was carried out at regimes of 0.13 and 0.52 J. Figure 2 shows the microstructures of the coatings in the optical microscope and the distribution of the Vickers hardness.

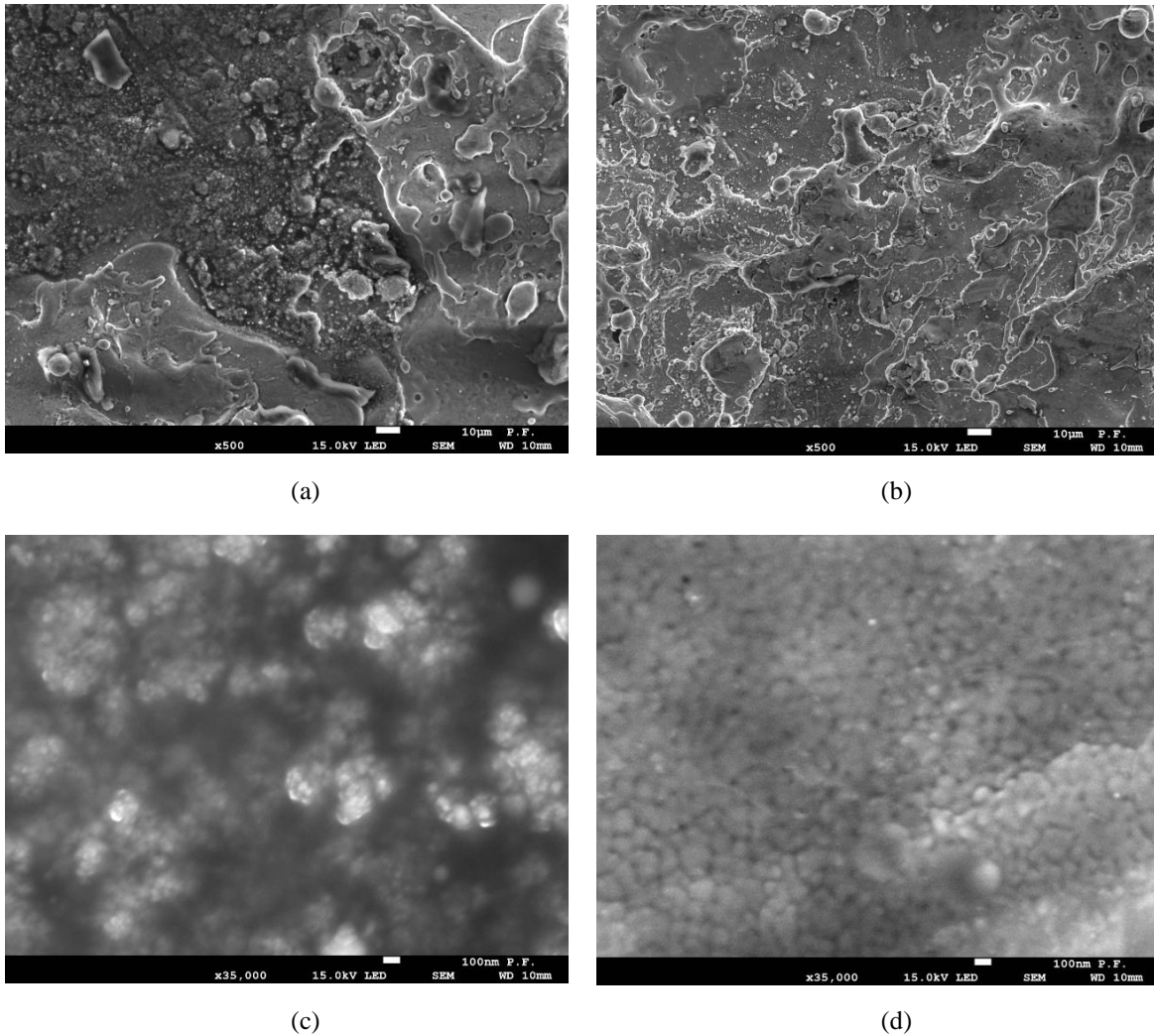


FIGURE 1. Results of topographic studies of Armco iron samples after the ESA according to the Mo-SPM-Mo scheme at different magnifications. Mo electrode treatment at discharge energy: a, c – 0.13 J; b, d – 0.52 J.

TABLE 2. Summary of research results

Electrode materials, ESA technology	SPM composition	Wp, J	The thickness of the "white" layer, µm	Micro hardness of "white" layer, HV	Continuity of "white" layer, %	Surface roughness, Ra, µm
Substrate – Armco iron, electrode -Mo	Without SPM	0.13	20-30	446	50	0.98
Substrate – Armco iron, electrode – Mo.	With SPM	0.13	30-40	608	50	1.26
Scheme of ESA:						
1) ESA by T15K6 electrode,						
2) using the SPM,	0.52	30-40	1300	70	2.67	
3) ESA by T15K6 electrode)						

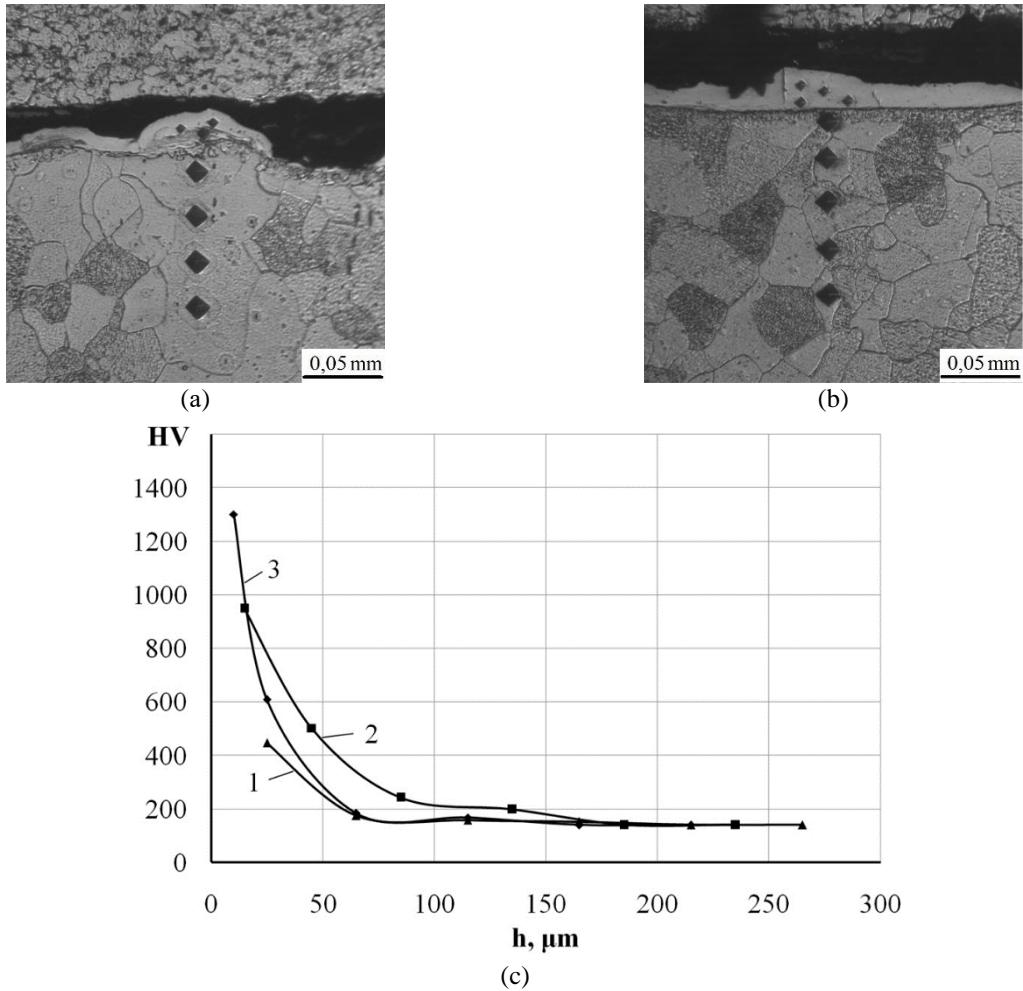


FIGURE 2. Optical microscope microstructures (a-b) and microhardness distribution (c) of coatings on Armco iron samples after the ESA. Surface treatment was performed at discharge energies: a – 0.13 J; b – 0.52 J; in figure (c): 1 – without SPM, discharge energy 0.13 J; 2 – 0.13 J; 3 – 0.52 J

The surface microstructure of ESA samples consists of three sections. The "white" layer is the top layer. It is known that the white layer is formed by the transfer of the electrode material to the substrate and by the chemical interaction of the electrode materials with each other and with the environment. The white layer is very difficult to etch due to its particular state of structure. Beneath the outer layer is a diffusion zone in which the elements of the anode material diffuse in the solid phase. The sample material (substrate), Armco iron, has a ferrite structure. The energy processing parameters have an effect on the characteristics of the coating. The thickness of the "white" layer and its continuity increase as the discharge energy increases. At the same time there is also an increase in the hardness value. At discharge energy of 0.13 J, the hardness value is 608 HV, and at 0.52 J it is 1300 HV. The hardness indicators are influenced by the composition of the SPM. The addition of nanotubes contributes to an increase in the hardness of the coatings under the same ESA conditions. Coatings without SPM have a hardness of 446 HV, while those with nanotubes have a hardness of 608 HV when treated with a discharge energy of 0.13 J. The thickness of the "white" layer and the continuity of the coating are also slightly improved by the use of SPM (Table 2). Obviously, the formation of a crushed micro- and nanocrystalline structure in the "white" layer, as well as the formation of nanostructural elements, are related to the results obtained from the metallographic analysis and hardness tests of the samples studied (Figs. 3, 4). In the electron microscopic structures, nanoscale phases ranging in size from 40 to 60 nm were found, which are homogeneously distributed in the coatings.

Maps of the elemental distribution in ESA coatings obtained with nanotube SPM at $W_p = 0.13$ J and $W_p = 0.52$ J are shown in Figs. 5 and 6. The results of local X-ray diffraction analysis showed that carbon is concentrated on the surface, while molybdenum is distributed homogeneously in the coating.

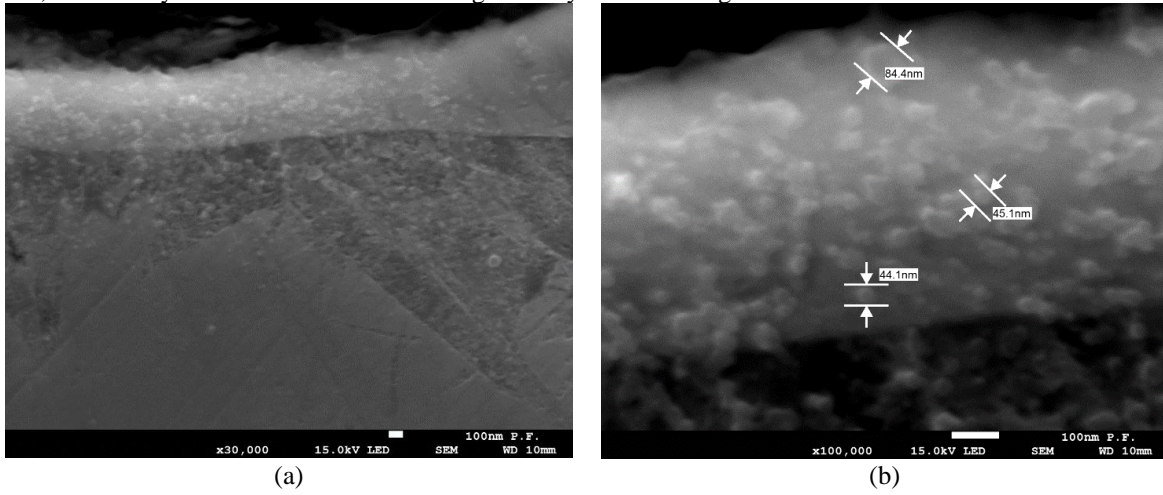


FIGURE 3. SEM microstructure of coatings after ESA at various magnifications. Coating was obtained at discharge energy 0.13 J.

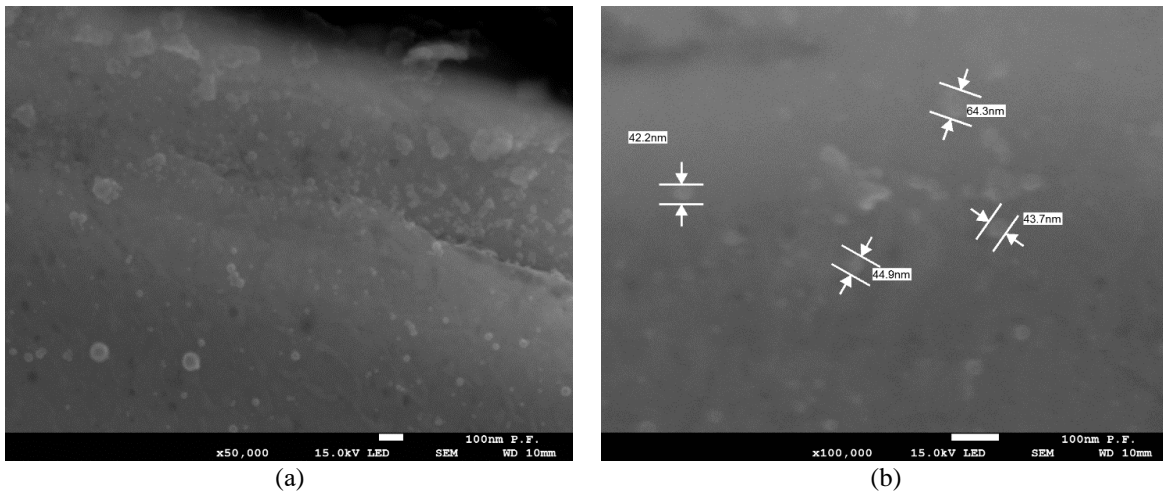
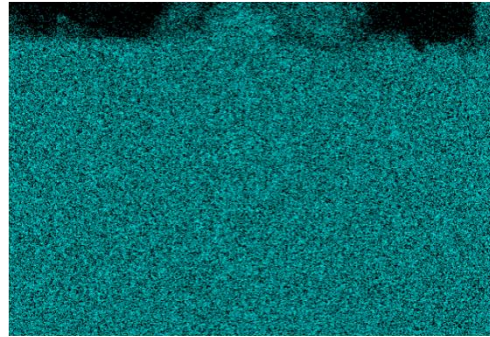
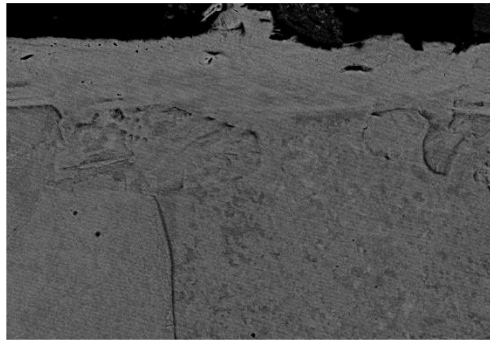


FIGURE 4. SEM microstructure of coatings after ESA at various magnifications. Coating was obtained at discharge energy 0.52 J.

Electron Image 5

Fe K α 1

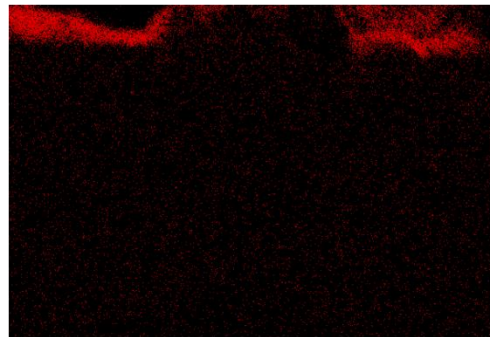
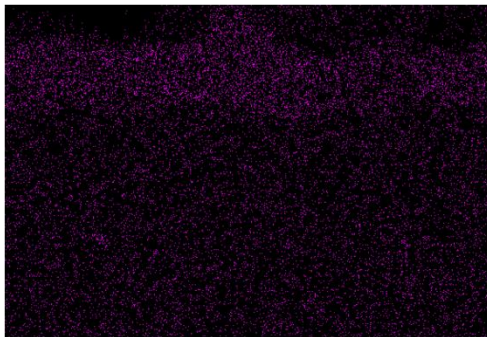


10 μ m

10 μ m

Mo L α 1

C K α 1_2



10 μ m

10 μ m

FIGURE 5. Elemental distribution maps of coatings produced at discharge energy 0.13 J.

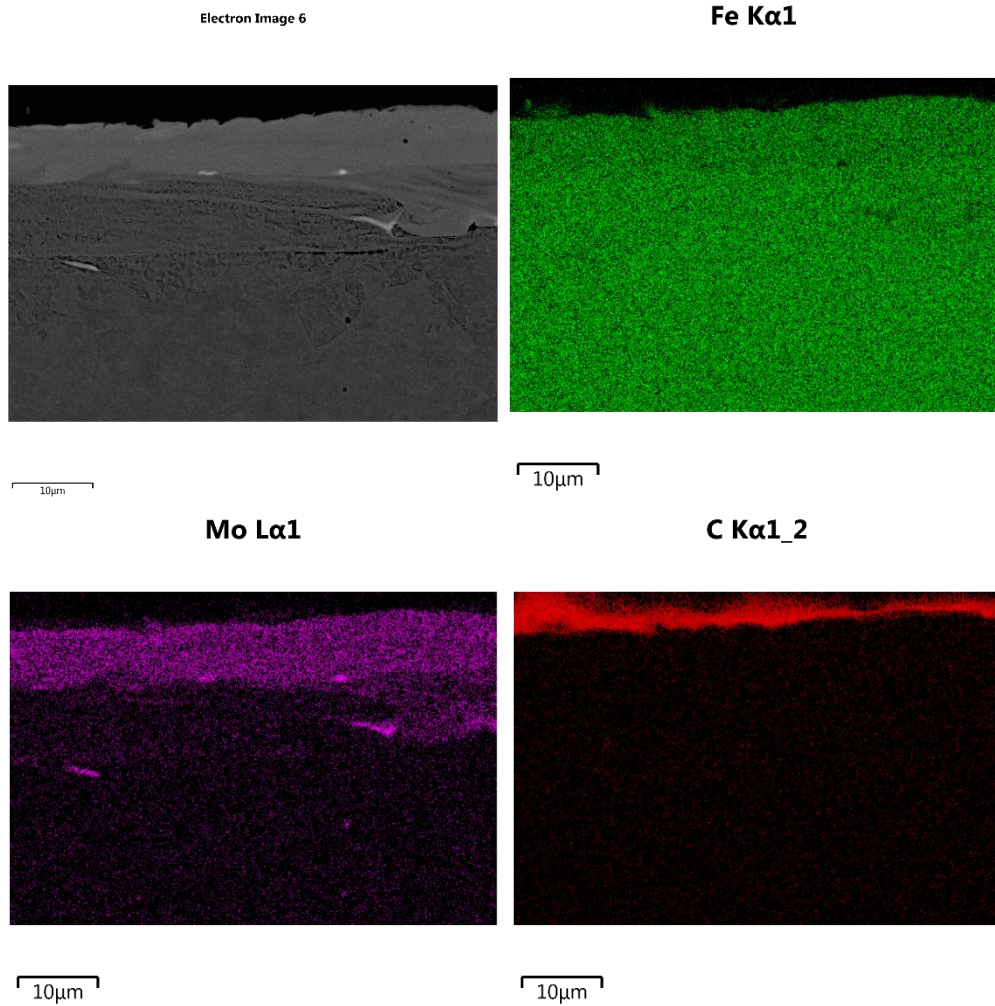


FIGURE 6. Elemental distribution maps of coatings produced at discharge energy $W_p = 0.52$ J.

Figure 7 shows the microstructure of the coating on EN-GJS-600-3 cast iron. Like steel, the microstructure consists of several areas: "white" layer, diffusion zone and substrate. The distribution of hardness in the coating is shown in Figure 2, c. The surface hardness increases as the discharge energy increases. Coatings produced by ESA using SPM containing nanotubes have a positive effect on increasing the surface hardness on both steel and cast iron.

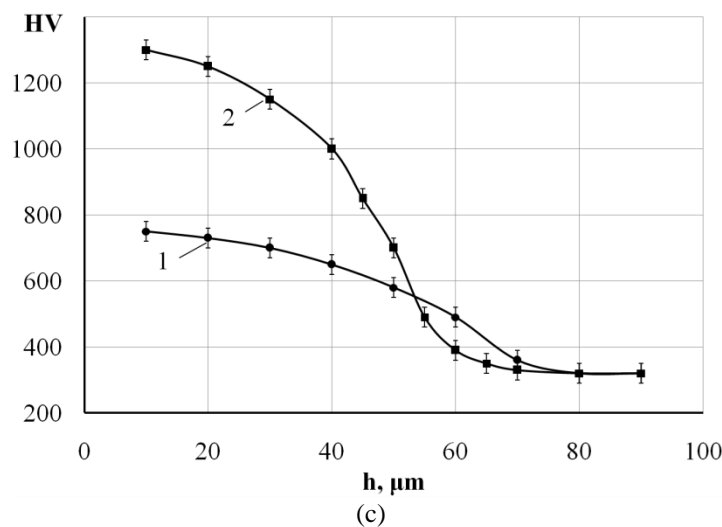
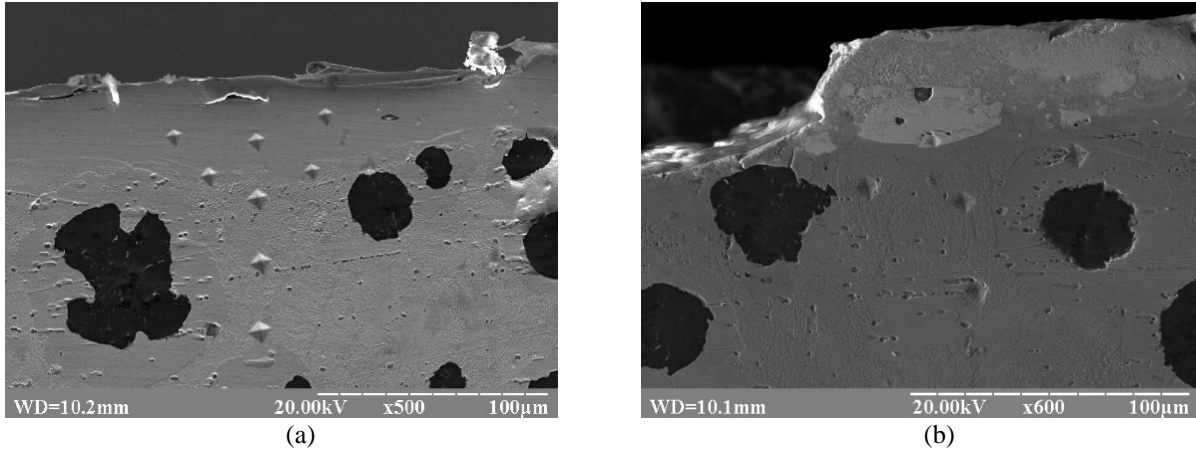


FIGURE 7. SEM microstructures (a-b) and microhardness distribution (c) of coatings on EN-GJS-600-3 cast iron samples after the ESA. Surface treatment was performed at discharge energies: a – 0.13 J; b – 0.52 J; in figure (c), discharge energy: 1 – 0.13 J, 2 – 0.52 J.

CONCLUSIONS

1. Topographic analysis of the surface of the samples after their treatment by electrospark alloying using a molybdenum electrode with the participation of STM, which included nanotubes with uniform distribution in epoxy resin, showed that evenly coatings are formed as a result of ESA, but with increased roughness. Furthermore, nanoscale inclusions are found on the surface, homogeneously distributed in surface.

2. The peculiarities of coating structure formation are presented. There are three sections to the surface microstructure of ESA samples. The "white" layer is the top layer. Under the outer layer is a diffusion zone in which the elements of the anode material diffuse in the solid phase. The sample material (substrate), which is Armco iron, has a ferrite structure.

3. The energy processing parameters have an effect on the characteristics of the coating. The thickness of the "white" layer and its continuity increase as the discharge energy increases. At the same time there is also an increase in the hardness value. At a discharge energy of 0.13 J, the hardness value is 608 HV, and at 0.52 J it is 1300 HV.

4. The addition of nanotubes contributes to an increase in the hardness of the coatings under the same ESA conditions. Coatings without SPM have a hardness of 446 HV, while those with nanotubes have a hardness of 608 HV when treated with a discharge energy of 0.13 J. The thickness of the "white" layer and the continuity of the coating are also slightly improved by the use of SPM. The positive effect of nanotubes on the quality and hardness of coatings is associated with the formation of nanostructures. SEM structures shows nanoscale phases from 40 to

60 nm, which are evenly distributed in the coatings. The results of local X-ray diffraction analysis showed that carbon is concentrated on the surface, while molybdenum is distributed homogeneously in the coating.

5. A coating was obtained on EN-GJS-600-3 cast iron using SPM, which consisted of multilayer carbon nanotubes ARKEMA (0.2%). With increasing discharge energy, the surface hardness increases from 750 HV to 1320 HV.

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