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PROCESSING OF MICROSTRUCTURES OF METAL SURFACES WITH CHEMICAL-THERMAL TREATMENT IN LOW VOLTAGE ELECTROLYTIC PLASMA

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Abstract. The main reason for these researches is to solve the problem of intensification of the alloying process by electric sparks by increasing the energy of the electric pulses. In relation to this, the purpose and object of the research is the need to assume a new concept of organizing the alloying process in order to intensify the strengthening of metal surfaces, excluding the negative effects. Among the electrophysical methods of layer formation, a special place is occupied by alloying by electric sparks of metal surfaces, considering the multitude of its characteristic advantages, which are the following: high adhesion of the deposited coatings, the possibility of depositing coatings of any conductive materials, the simplicity of the process and the low consumption of energy for its implementation. The main results are that after the heating in the electrolyte, the cooling takes place in the air, then in the superficial layers of the samples treated in this way residual tensile stresses act. In conclusion, it is important to mention that through the combined processing the properties of the superficial layers and the exploitation characteristics can be controlled.

Keywords: *metals, electrolyte, materials processing, electrolytic plasma, pressing, chemical-thermal, process.*

Rezumat. Motivul principal al acestor cercetări este acela de a rezolva problema intensificării procesului de aliere prin scântei electrice prin creșterea energiei impulsurilor electrice. În raport de aceasta, scopul și obiectul cercetării este necesitatea asumării unui nou concept de organizare a procesului de aliere în vederea intensificării întăririi suprafețelor metalice, excluzând efectele negative. Dintre metodele electrofizice de formare a straturilor, un loc aparte îl ocupă alierea prin scântei electrice a suprafețelor metalice, având în vedere multitudinea avantajelor sale caracteristice, care sunt următoarele: aderență ridicată a învelișurilor depuse, posibilitatea depunerii de acoperiri a oricărui conductor conductor. materialelor, simplitatea procesului și consumul redus de energie pentru implementarea acestuia. Principalele rezultate sunt că după încălzirea în electrolit are loc răcirea în aer, apoi în straturile superficiale ale probelor astfel tratate acţionează tensiunile reziduale de tracţiune. În concluzie, este important de menționat că prin prelucrarea combinată se pot controla proprietățile straturilor superficiale și caracteristicile de exploatare.

Cuvinte cheie: *metale, electrolit, prelucrare materiale, plasmă electrolitică, presare, chimio-termic, proces.*

1. Introduction

The general name of "electrotechnologies" combines various technological processes, special features of which consist in the use of electricity to achieve technological transformation of materials (changes in condition, composition, structure, shape and operating properties, etc.) [1, 2].

Electrotechnologies as a separate field of materials technology has no strictly set limits, but in practice they can be classified into the following processes and methods:

1. Electric welding (with arc, contact, electric drill, etc.).
2. Galvanic deposits of metal coatings (electroplating, electroplating).
3. Induction heating, with high and high frequency currents (melting, soldering, heat treatments).
4. Methods of electron-ion technology (electrostatic painting electrophoresis, etc.).
5. Electrophysicochemical methods and combined processing of materials.

It is important to note that for a better acquisition of the theoretical material a special role is played by practical laboratory work and seminars in the research process have the ability to deepen their knowledge in the theoretical classes [3].

This guide proposes a cycle of works, which aims to familiarize students in theory and practice with laboratory techniques in the field of electrophysical methods of processing. It is addressed to future specialists, who want to deepen their knowledge in the field of high technologies.

The processing of metals and especially electrolytic plasma steels effectively solves complicated technological problems. Among the advantages of the method can be listed the following: obtaining high heating rates up to quite high temperatures, practically up to melting temperature, the possibility of a complete automation of the process in mass production, diversification of thermal and thermochemical processing operations, also soldering as well as making deposits by melting, etc., the possibility of locating and obtaining a strictly limited heating area; the ease of adjusting the thermal parameters by changing the electrical parameters; a specific energy consumption; non-aggressiveness of the work environment [4].

A simpler process for reducing the roughness is the subsequent sparking of the alloyed surface with a graphite electrode at a lower regime, than during the formation of the coating itself [6-8]. In this case, due to the substantial specific resistance of graphite compared to metals, in the places where the electrode contracts with the micro-irregularities, as a result of the release of Joule heat at the moment of the short circuit, the melting of these micro-irregularities takes place. However, since in the alloying by electric sparks with a graphite electrode as a result of the transfer of the electrode material to the cathode, there is no increase in the coverage, but only the diffusion of carbon atoms in the alloy surface, with a multiple action of electric pulses the roughness of this surface decreases. Thus, as a result of the sparking of the coatings with the use of the graphite electrode, not only the reduction of the surface roughness is obtained, but also the synthesis of different carbon compounds, which in the end, improve the physico-chemical and exploitation characteristics of the superficial layers [5].

However, these preliminary results did not allow for an in-depth knowledge of the laws of the processes that take place in the space between the electrodes and in the superficial layers of the cathode during the alloying by electric sparks during the simultaneous action of the magnetic field superimposed on the working area and the direct passage of the current electrically through anode and cathode. These new knowledge can only be obtained in the case of systematic studies of all aspects of this new variant of the alloying process by electric sparks. Systematic studies on the influence of additional energy sources on the ASE process are being carried out for the first time [6]. Under these conditions, the opportunity for research is evident, and the objectives of the doctoral thesis refer to the following aspects:

- analysis and presentation of the results of worldwide research in the field of alloying by electric sparks;
- the development of the research method and the creation of specialized devices and installations for conducting experimental investigations and for the application of technology.
- establishing the legality of the formation of coatings on the surface of the cathode - part under different conditions of the action of continuous and pulsating electric current, as well as of the magnetic field;
- establishing the redistribution of elements and the transformations of structure and phase in the superficial layers subjected to AES in the magnetic field and when the electric current passes through the electrodes;
- evaluation of the structural, substructural, tensional, physical-mechanical and operational characteristics (microhardness, residual stresses, wear resistance, electroconductivity) of the layers obtained during complex processing:

a) alloying by electric sparks with the additional action on the AES of the magnetic field and the electric current;

b) alloying by electric sparks with subsequent plasma chemical treatment in electrolytes;

- the establishment of complex technologies with industrial applications for obtaining coatings on construction alloys [7, 8].

2. Materials and Methods

When carrying out the experimental part, certain technological parameters of processing were taken into account. Thus the voltage on the electrolytes must be adjusted between 0-300 V, the current density up to 50 A/cm^3 in the transaction mode, and up to 2 A/cm^3 in the heating mode and ensuring the fuselage of the electrolyte, the temperature of which in the processing process does not must exceed 30 °C. Steel 45 (0.45% C) was used to carry out the work and cutting knives were made directly to the right with the following construction elements [9].

Testing of 45 steel hardened electrolytic plasma hardened and Y13 heat-treated knives was performed on the 1K62 lathe on a 20 mm cylindrical rolled 45 steel semi-finished product at the following cutting speeds:

- cutting speed- $v = 20$ m/min;
- cutting depth $t = 1$ mm;
- federate $S = 0.1$ mm/rot.

The amount of wear was determined with the help of a laboratory microscope with a magnification of 25 times.

2.1. Theoretical-practical methods and variants of materials processing

At the continuous increase of the voltage on the electrodes of the bath, at the beginning, the normal electrolysis process takes place in this region, the dependence between current and voltage has a linear character and is described by Ohm's law. A further increase in voltage leads to the beginning of the first phase intensive formation of gases on the surface of the cathode - is manifested by oscillations of the liquid in the cathode region, the appearance of spark discharges, and with characteristic cracks. The further increase of the voltage leads to the increase of the number of unit discharges and finally on the surface of the cathode the formation of a luminous coating takes place, the intensity of the current again increases. The second phase of the process begins, through which the heating of the cathode surface takes place. It is not recommended that the total voltage (e.g. 220 V) be applied to the electrodes, in which case the process will proceed abnormally in accordance with the first phase [10].

The method has its drawbacks, such as the need to make a special machine and certain devices for it: complicated machine to ensure a safe operation due to high voltages; difficulties in directly controlling the temperature of the parts to be processed; some difficulties in processing long profiled parts; a yield not exceeding (40-45 %).

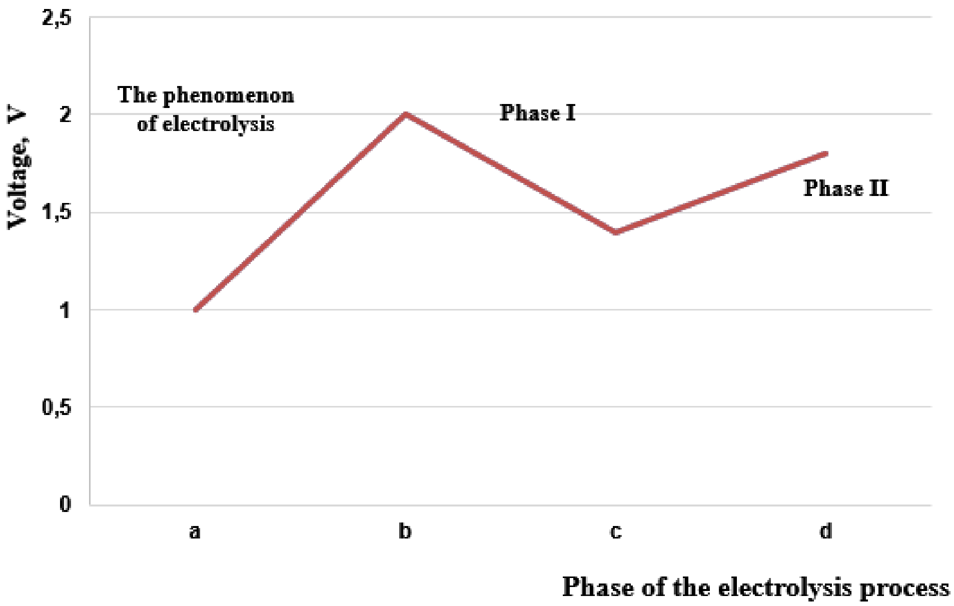


Figure 1. The typical volt-ampere characteristic of the phases of the plasma electrolytic processing process [10].

The typical volt-ampere characteristic of the phases of the electrolytic plasma processing process is shown in Fig.1. At the continuous increase of the voltage on the electrodes of the bath, at the beginning, the normal electrolysis process (a-b) takes place in this region, the dependence between current and voltage has a linear character.

A further increase in voltage leads to the beginning of the first phase (b-c) intensive formation of gases on the surface of the cathode - is manifested by oscillations of the liquid in the cathode region, the appearance of spark discharges, and with characteristic cracks. The further increase of the voltage leads to the increase of the number of unit discharges and finally on the surface of the cathode the formation of a luminous coating takes place, the intensity of the current again increases.

The second phase of the process (c-d) begins, through which the heating of the cathode surface takes place. It is not recommended that the total voltage (e.g. 220 V) be applied to the electrodes, in which case the process will proceed abnormally in accordance with the first phase [12].

2.2 Tempering the parts by the free end heating method

This method hardens the valve heads, bolt heads, screws, pushers, regulator clutch heads, etc. The high precision of the length of the end subjected to heating is maintained by ensuring the constant immersion depth of the part which is the cathode in the electrolyte. The metal bath connects to the positive pole of the DC circuit.

The depth of immersion of the part is determined by means of the fixing device. The possibilities of using the process are determined by the section, shape and length of the end of the part to be heated. When increasing the section and the length of the end subjected to processing, the current density is distributed unevenly. Such a condition leads to melting of the end and first of all of the parts that are sharp. Basically, this process is only used for machining the cylindrical and spherical ends of parts with a diameter of up to 15 mm and a length of 5-10 mm [11].

2.3 Hardening and annealing of the heads by shielding them

Expanding the region of the end use of heating contributes to its shielding and the extremities. To reduce the current density on the ends based on the sharp ones (edges), the parts are fixed on an electrically insulating refractory material. Using screens (screens) it is possible to evenly heat parts with a diameter of 30-35 mm when immersing in electrolyte up to 40 mm.

2.4 Processing of materials by pressing

The principles of metalworking in electrolyte before forging, stamping, bending, discharge are the same as in other cases. The only difference is that before pressing, the metal is heated to full volume, but not only on the surface, as in hardening. Such processing is carried out by the appropriate selection of heating regions and occurs in installations used for tempering. Semi-finished products with a diameter of 100 mm may be subjected to such processing.

When the constant conditions regarding the composition, concentration and temperature of the electrolyte are observed, the heating regimes are regulated only by changing the voltage of the current source and the heating time [12].

The advantages of the electrolyte heating method are manifested in the process of performing concrete operations in a certain way. Gluing excludes the need to use fluxes, reduce the use of soldering material, significantly increase productivity, the possibility of simple automation of lines in the torrent. Local or general heating, volume or surface heat treatment or mechanical heat treatment - this technology allows easy adjustment of heating rate, no surface oxidation, preservation of the initial roughness and accuracy of the parts to be processed, use for any electroconductive materials, simplicity of automation and simple operation. In the case of white incandescent annealing of wires, thin pipes, flexible shafts, etc. - high production quality and simplification of operations are achieved. In the process of baking and hot pressing of parts made by the powder metallurgy method - there is a correlation of the joining of the grains between them, with pressing in an operation, - simplification of technology, increase of quality of parts, articles. The basic conditions for obtaining a uniform and economical heating consist in creating a necessary current density on the cathode and maintaining a correlated ratio between the surface of the cathode and the anode [12].

The surface of the anode must exceed the surface of the cathode not less than 10 times. The uniformity of the heating of the surface of the piece of complex shape is carried out by changing the distance between the different sectors of the anode and the cathode to obtain on the whole surface of the cathode approximately the same current density. The speed and temperature directly depend on the size and duration of the current flow, but in each case the absolute values of these indices are different depending on the type of items subjected to heating [12].

2.5 Wear resistance

The tribological tests of the superficial layers obtained from the additional energetic action on the alloying by electric sparks process were carried out in two stages. First of all, the wear resistance of the coatings was evaluated using the express method at the installation, the principle scheme of which is presented in Figure 2 [13].

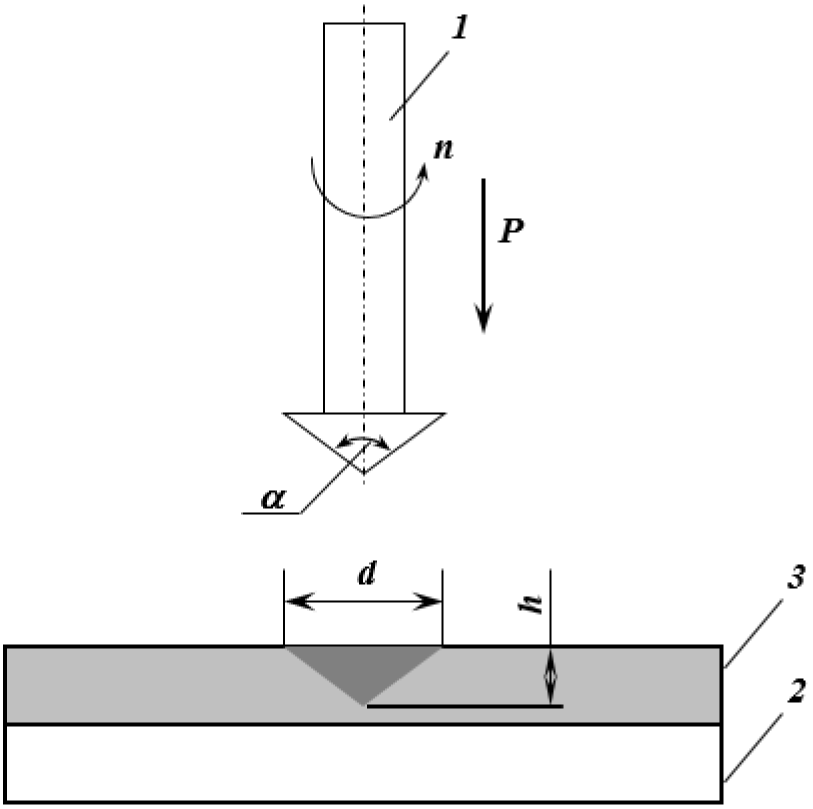


Figure 2. Scheme of the wear resistance measurement procedure: 1 - centering device with penetrator; 2 - sample; 3 - cover; *d* - diameter of the footprint; *h* - penetration depth of the penetrator: *n* - rotation frequency of the penetrator; P - force, applied to the penetrator; *α* - the angle at the tip of the penetrator (*α* = 90 - 1500) [14].

Depending on the thickness of the coating and the microhardness of the layer subjected to the test at different depths, the parameters that determined the wear of the layer were measured. The essence of the method of measuring the wear resistance of the material using this procedure consists in determining the diameter and depth of the imprint when rotating the diamond penetrator with an eccentricity of no more than 0.1 μm. The measurement accuracy is ~1.0 μm. The dimensions of the fingerprint on the sample-standard coated by the traditional method of spark alloying were taken as 100%, and the ratio of the dimensions of the fingerprint on the investigated samples with coatings obtained by alloying by electric sparks, in the magnetic field compared to the standard was chosen as the unit of wear resistance for the given test procedure [14].

The samples for the tests were made at optimal regimes of alloying by electric sparks in the magnetic field, for which conditions were created to obtain coatings with greater thicknesses and higher quality for the given pair of materials of the anode and the cathode. For example, when depositing silver coatings, the discharge energy (Wd) and magnetic field induction (B), superimposed on the processing area, were chosen, starting from the condition of minimal irrecoverable losses of silver and obtaining a uniform and continuous coating. These parameters were: Wd = 0.1 J and B = 0.06 T. When establishing these parameters in the case of alloying by electric sparks with Ni, Cr, Al, graphite electrodes, the discharge energy was 10 times higher and was 0.9-1.0 J for magnetic field induction 0.074 T.

In the second stage, the samples with similar coatings were tested for wear on the MI-1 and CMT-1 standard rubbing machines.

Under the conditions of friction without grease, the load was 140 N/cm^2 at the sliding speed of 0.3 m/s, and at the semi-liquid friction, respectively 700-750 N/cm^2 and the sliding speed 1.3 m/s. Such a regime was chosen in order to create conditions close to those of exploitation.

Industrial oil -20A was chosen as the grease, which was introduced into the friction area at the rate of 3-4 drops per minute. The coatings by electric spark alloying were deposited on the cylindrical generator of the 50 mm diameter roller. The contact surface of the track with the roller was approximately 100 mm, which made it possible to substantially save the running-in time.

In a series of cases alloying by electric sparks nickel, silver and chromium coatings were smoothed with a 12 mm diameter hard ball of 15 steel with a spring smoothing device at a load of 300 N. The rotation speed of the part (roller) and the advance of the support are established in accordance with the recommendations of D.D. Papsev and the support of the advance constitute 50-100 m/min and the rotation speed of the piece is 0.15-0.2 mm/rot. The hard layers, obtained by electric spark alloying with tungsten carbide-based hard alloys, were first smoothed with a hard ball, and then with a diamond grinder with a radius of 1.5 mm at a load of 15-20 kF and respectively the advance is 0.05 mm to obtain the roughness 3.0...0.32 µm is recommended for the surfaces of the friction pairs [15].

The results of the tests, in which the wear of the coating over time is determined, is presented on Figure 3.

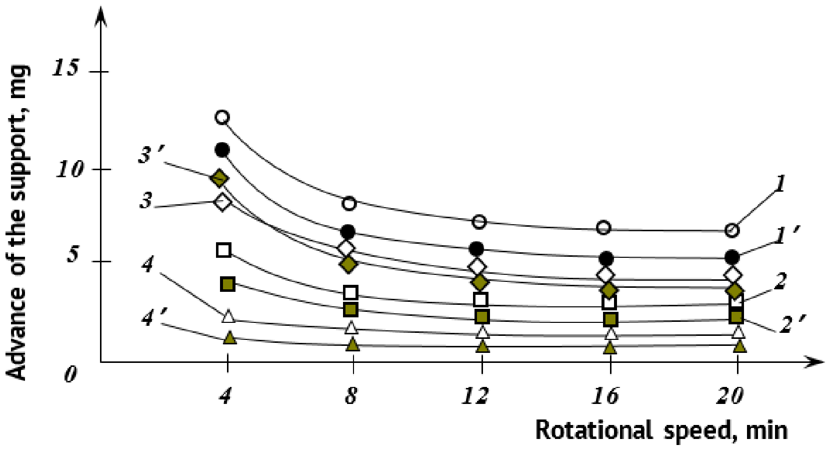


Figure 3. Wear over time of alloying process by electric sparks coatings in traditional airing: 1 - Al; 2 - Cr; 3 - Ni; 4 – graphite and at alloying by electric sparks in magnetic field: 1' - Al; 2' - Cr; 3' - Ni; 4' – graphite [15].

3. Results and Discussion

The obtained results show us that if, after heating in the electrolyte, the cooling takes place in the air, then in the superficial layers of the samples treated in this way, residual tensile stresses act. Tempering in the electrolyte, however, leads to the formation in them of residual compressive stresses with maximum values in the depth of the superficial layer.

Based on this, the conclusion can be drawn, that with the combined processing, possibilities are created to control the properties of the superficial layers, and therefore, the exploitation characteristics [17].

The chemical-thermal treatment is more fully performed under the regime of heating in low voltage electrolytic plasma.

The results of the calculations are shown in Table 1.

Table 1

Wearing the face of the lathe knives

The path taken by the cutting tool, m	The size of the back surface wear, mm
100	0.1-0.4
200	0.2-0.4
300	0.2-0.6
400	0.3-1.0
500	0.7-2.0

Considering the limit wear for knives h_s = 0.5 mm, the length of was obtained cutting for 45 steel spindles, processed into voltage electrolytic plasma low equal to 465 m, and for heat-treated U13 steels equal to 260 m.

A considerable number of scientific papers have been written about the influence of the magnetic field on the mechanism and kinetics of transformations in the heat treatment process of steels, whose results are analyzed in the specialized literature [17].

In the previous chapter, the influence of the magnetic field on the initial and intermediate phases of alloying process by electric sparks was studied in detail: the erosion of the anode and the transfer of the eroded material to the cathode. But it is necessary to appreciate the influence of the magnetic field on the final phase of this process, that is, the formation of the superficial layer on the cathode.

Because in the process of alloying with electric sparks on the electrodes, in particular, in the superficial layers of the cathode as a result of the thermal action of the impulse discharges, micrometallurgical processes occur with phase and structure transformations, with diffusion phenomena, etc. The constant magnetic field on the alloying zone with electric sparks is applied in such a way that in one case it ensures the parallelism of the vectors of the magnetic induction and the discharge current, when they coincide ($\vec{B}\uparrow\uparrow\vec{I}_d$) or have opposite directions ($\vec{B}\downarrow\uparrow\vec{I}_d$), and in another case - their perpendicularity ($\vec{B}\perp\vec{I}_d$).

It should be mentioned that for the proposed energy and time parameters of the alloy with electric sparks, no displacements of the reflexes were observed on the radiographs, that is, they were not fixed to the voltage of the second degree. This was verified at AES with ammonium anodes of copper and iron samples. On fig. 4. the concentration curves of chromium distribution in St. 3 are presented in the case of the presence of the field and in its absence.

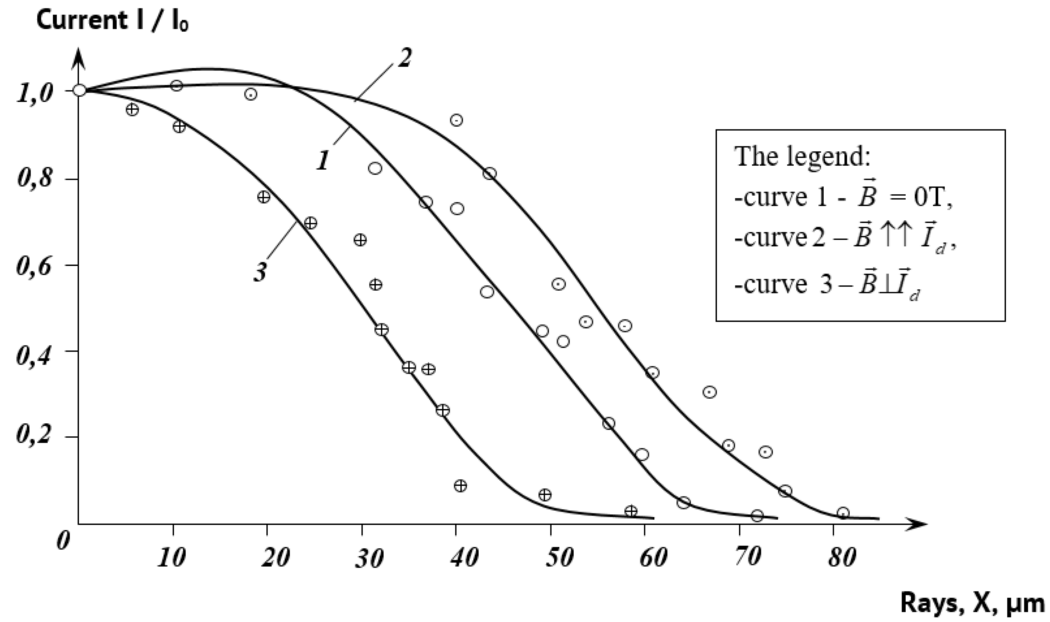
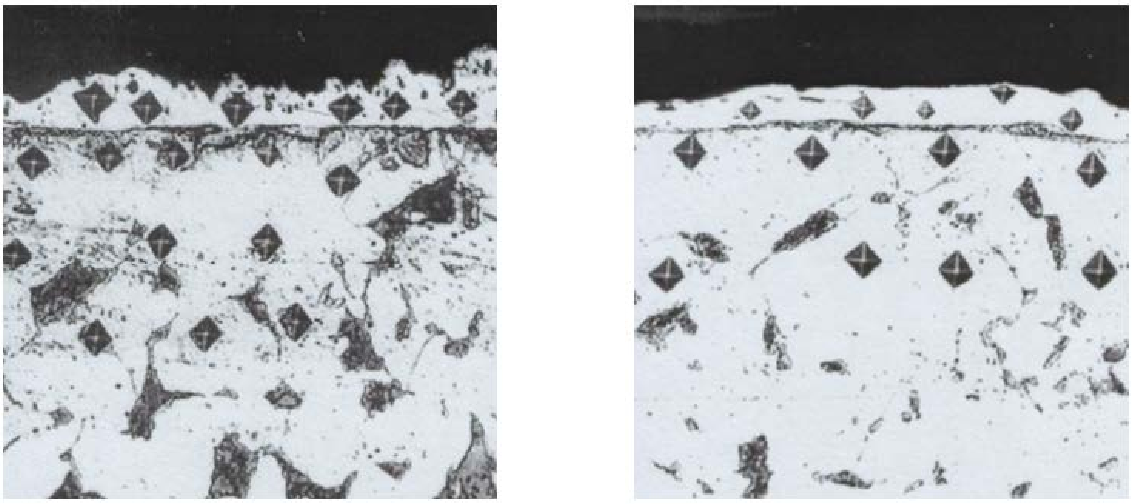


Figure 4. Chromium distribution curves in the coating at alloying process by electric sparks in a magnetic field.

From the analysis of fig. 4. it turns out that the application of the magnetic field parallel to the vector of the discharge current ($\vec{B}\uparrow\uparrow\vec{I}_d$) (curve 2), contributes to increasing the depth of chromium penetration. But when changing the direction of the magnetic field by 90^0 ($\vec{B}\perp\vec{I}_d$) (curve 3), the penetration depth of the chromium atoms decreases substantially.



a) b)

Figure 5. The microstructure of the sample from St. 3, subjected to AES with nickel under normal conditions (a) and in a magnetic field (b); discharge energy 0.3 J, specific alloying time – 1 min/ cm^2; x 500 (Made by the author).

The superimposition of the magnetic field also affects the solubility of the alloying element in the matrix. Thus, the parameter of the steel network, which in its initial state was 0.28771 nm, after alloying with electric sparks with chromium for $\gamma = 0$ decreases by 0.0006 nm as a result of the dissolution of chromium in it, and under the action of the magnetic field

- by 0.0010 nm, which speaks of the content in the solid solution of a larger amount of chromium [18].

Through the metallographic analysis and measurement of the surface roughness, it is confirmed that the magnetic field, superimposed on the processing area, influences the intensification of the mixing of materials, contributing to the increase of homogenization and the formation of more uniform coatings by thickness (fig. 5).

When alloying St.3 steel with electric sparks in a magnetic field with graphite anodes, the phase composition of the superficial layers changes less than in the usual alloying conditions. For example, the change of network parameters of the iron solid solution constituted 8×10^{-3} nm and 2×10^{-3} nm, respectively for the case of alloying with electric sparks in the absence of the field and in its presence. At the same time, the content of cementite and austenite in the diffusion zone is practically the same in both cases.

4. Conclusions

In conclusion, it should be noted that:

1. The dependences of the mass transfer and the intensity of the deposition of the hardened layer on the size and nature of the electric current passing through the electrodes as well as on the parameters of the magnetic field superimposed on the area of alloying by sparks were established.

2. The phenomenon of quasi-regular oscillation of the intensity of the anode material transfer in the process of pulse discharge and structure and phase transformations in the superficial layers of the cathode under the action of the magnetic field superimposed on the alloy area was discovered.

3. It was established that when the direct current with the density of 1 – 3 A/mm^2 passes through the anode, a maximum amount of material of the anode and respectively of the addition of the cathode was obtained, and the variation of the induction of the magnetic field within the limit of 0.01 – 0.1 T allows directing in a wide range the processes of physicochemical transformation in the surface layers of the cathode.

4. The dependence of the intensity of the coating formation on the surface of the cathode on the magnetic properties of the processing electrode and the matrix was established. Under equal conditions in the magnetic field of the St.3 matrix with a nickel electrode, the intensity of the eroded mass transfer in a unit of time is 3.5 - 4.0 higher than when processing with a silver electrode.

Conflicts of Interest: The author declares no conflict of interest.

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