

Micro and Nanotechnological Procedures of Materials Processing through the Application of PEDM

Pavel Topala^{1,2}, Alexandr Ojegov¹, Dorin Guzgan^{1,2}, Valeriu Creciun¹, Vitalie Besliu¹

¹Department of Physical and Engineering Sciences, Alecu Russo Balti State University, Balti, Republic of Moldova

²Department of Machine Construction Technology, National University of Science and Technology POLITEHNICA, Bucharest, Romania

Email: pavel.topala@gmail.com

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Abstract

Micro and nanometric material processing procedures are of great interest to both researchers and manufacturers. They are currently trying to solve the material and energy crisis that has affected the whole of humanity, on the one hand. On the other hand, it comes with new solutions in miniaturizing the technique and solving the problems faced by the compatibility of existing materials with living matter, that is, it comes with solutions to improve the quality of life. Thus, different procedures of material processing aimed at the micro and nanometric surface transformations are used: laser processing, ion beam processing, electroexplosive processing. All these procedures are high energy-consuming and have low efficiency. The authors of this paper propose pulsed electric discharge machining (PEDM) as concentrated source of energy that uses directly the accumulated electric energy for the surface processing by discharge pulses. It is low energy-consuming and has higher efficiency. The picture of electroerosion during PEDM is a complex one, it is produced under the action of strong heat and electric fields generated by “cold” and “hot” electrode spots. The removal of materials during electrical erosion is caused by the development of longitudinal and transverse capillary waves, but also by the bombardment of surfaces with particles from the work environment. Recent studies on the interaction of surface materials by PEDM plasma have shown that under its action the active surfaces of parts applied in machine building, electronics, chemical industry, food industry, medicine, etc., morphological, structural and chemical composition micro-changes can occur. On the surfaces of the machined parts, films of nanometric oxides and hydroxides can be formed in an amorphous state. PEDM allows for obtaining 3D carbon structures, fullerenes and single-walled carbon nanotubes. The authors demon-

strate the possibility of extracting Sn nanowires and welding metal wires for applications in electronics. The application of surface microgeometry modifications ensures the increase of the thermoelectric current of the electrons up to 10 times, the surface-active resistance by 10^7 times, the resistance to corrosion in the aggressive media from 2 to 100 times.

Keywords

Nanotechnology, Film, Corrosion, Microwelding, Microgeometry, Emission, Resistance

1. Introduction

At present, a vertiginous development has been known at the beginning of the field of micro, followed quickly by that of nanotechnologies. They come to solve a multitude of problems faced by all of humanity, among which the crisis of materials and energy can be highlighted.

Micro- and nanotechnology come with new solutions in the miniaturization of technology, thus solving the problem of the material crisis, and finally solving the problems faced by the compatibility of existing or artificially created materials with living matter, that is, it comes with solutions regarding the improvement of the quality of life. It applies a whole range of phenomena of physics, chemistry, biology and comes in turn with new knowledge and legalities obtained in the process of scientific research. Of particular interest are the physical methods of nanotechnology and especially those of plasma interaction with material surfaces or with material particles of different origins. Plasma as the fourth form of existence of matter is most often obtained by electrical discharges [1].

In this context, it is important to mention that the most widespread method of obtaining plasma is electrical discharges [1] [2]. In the case of electric discharges, plasma formations interact with the working environment and with the surfaces of the electrodes, causing important changes in composition, structure and, as a result, in properties.

It has been established that, depending on the conditions of the evolution of the electric discharge, at the contact of the plasma with the electrode surfaces, “cold” and “hot” electrode spots are born firstly [3]-[6], and then the plasma jet develops.

“Cold” spots are born at the beginning, immediately after striking the gap. They migrate very quickly (the erosive trace appears in the form of small separate craters [5], without obvious signs of melting), and “hot” electrode spots appear on the basis of the merging of the “cold” ones, have a lower “movement” speed and produce a substantially greater erosion than the first ones (at the points of their action, the liquid phase appears and the vaporization of the electrode material takes place [5] [7]).

The plasma formations for the case of pulsed electric discharges at gap values within the limits of 10 - 25 mm, for the charging voltage of the generator capacitor bank at values of 28 kV are presented in **Figure 1**.

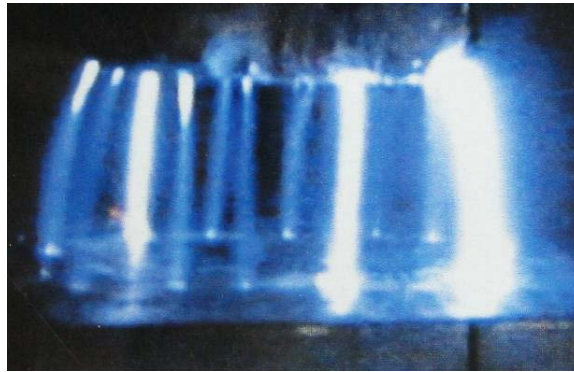


Figure 1. General view of electrode spots and plasma channels at the surface processing by applying PEDM.

It is observed from this figure that the spherical plasma formations arise at the electrode surfaces (obvious bright areas of spherical shape are observed) from which the plasma jet develops mainly from the cathode to the anode. It follows that the electrode spots generate not only the electrical and thermal effects that occur at the electrode surfaces, but also for the effects that occur in the gap (shock wave, luminosity, radiation, dissociation, synthesis, etc.).

Recently, a series of scientific papers have appeared that attest to the application of plasma in various nano-technological processes for obtaining nanoparticles and nano-films [6] [8]-[11]. In these papers, the interaction of the plasma with the processed surfaces is examined as a highly ionized gas, which is why a whole series of effects do not find their real explanation, but only remain, as experimental findings documented and applied in practice, without penetrating deeply into the essence things.

2. Research Methodology

For the research of the phenomena related to the electrical discharge in gases and its interaction with the surfaces of the electrodes, a construction made of two electrodes is used, placed at a certain gap in which the gaseous medium is located at different values of its pressure. The development of new technologies with the application of plasma as a concentrated source of energy is impossible without knowledge of the processes of interaction of the plasma with the surfaces of the processed parts (or the environment) in order to give them new permanent or temporary functional properties.

A set-up was used to carry out experimental research under the conditions of pulsed electric discharges, consisting of: a current pulse generator, a control unit and an initiation unit. The control unit allows fine adjustment of the discharge frequency within the limits of $1 \dots 10^5$ Hz, but also has a function of synchronizing the initiation pulses and power pulses. Initiation pulses ionize the gap and prepare the environment for the main discharge from the current generator.

Experimental research on the extraction conical asperities was carried out in air environment (under ordinary conditions) at solitary discharges. Wires made of

W (90%) with Re (10%) alloy, with a diameter of 0.2 mm and 0.25 mm, were applied in the construction of the cathodes intended for current emission. According to the methodology described in [11] [12], Taylor cone-shaped asperities were extracted from the cylindrical surfaces of the wires. The workpiece was connected in the discharge circuit as an anode, and the tool-electrode as a cathode.

Photographing the structures and the micro-geometry of the surfaces obtained as a result of the action of pulsed electrical discharges were carried out using the XJM600T metallographic microscope equipped with a digital information recording system.

The analysis of the morphology (SEM, Scanning electron microscopy) and the chemical composition (EDX, Energy dispersive X-ray analysis) of the surface layers of the samples subjected to processing were carried out with the help of the TESCAN type electron microscope equipped with the necessary analysis devices. The research was carried out in the National Center for Study and Testing of Materials, Technical University of Moldova, Chisinau, Republic of Moldova.

3. Research Results and Their Interpretation

3.1. Extraction of Conical Asperities from Metal Surfaces

The authors of this work support the hypothesis that the contact between the plasma of electric discharges and the surfaces of the electrodes takes place through “cold” and “hot” electrode spots. In the case of the maintenance of electric discharges in impulse on “hot” electrode spots, the latter serve as sources of powerful heat ($T \sim 4 \cdot 10^4$ K) and are generators of strong electric fields ($E = 10^6 \div 10^8$ V/m). The authors of the most recent researches attribute to the electric field a special role in the process of heating and melting the material electrodes and explain the material removal from the electrode surfaces by the development of capillary waves caused by the electric field [13]. It is important to highlight that the pressure force of the electron beam (ions), the reaction force of the metal vapors, the force due to the static pressure of the metal vapors in the crater and of course the surface tension of the liquid metal also act on the liquid metal. Applying the scientific findings of the authors [14]-[17], the extraction of Taylor cone-type asperities from the processed surfaces was achieved, in order to confer new properties: increasing the active area, increasing the electronic thermos emission capacity, building micro-objects (cantilever for AFM).

Experimental research demonstrates that in order to achieve the technology of modifying the microgeometry of surfaces with the extraction of asperities in the form of Taylor cones by applying PEDM, it is necessary to satisfy the following conditions.

Ensuring the local melting of the workpiece surface [18] determined by the relation (1):

$$Q = \frac{4W}{\pi d_c^2 S} \geq Q_{melt} \quad (1)$$

where: Q is the amount of heat released in the plasma canal during PEDM; W is

the energy released in the plasma canal; d_c is the diameter of the crater with liquid phase expected to be obtained on the surface of the cathode; S is the gap between the electrodes; Q_{melt} is the volumetric melting density of the workpiece material and is determined according to the relationship $Q_{melt} = q_{melt} \cdot \rho$; q_{melt} , ρ are respectively the specific heat of melting and the density of the workpiece material.

In the experimental conditions, it was necessary to generate an electric field with an intensity of about 10^8 V/m [13] [19]. Here it was taken into account that the surface of the liquid metal is subject to the action of the electric field force created by the electrode spots, the surface tension force and the weight force (the last component was omitted by the authors [16] [17]). Taking into account the fact that at the beginning, the heating and melting of the surface take place with the disturbance of the surface under the action of the electric field caused by the electrode spots, for the calculation of the critical intensity of the critical electric field [16] it is necessary to apply the relation:

$$E_{cr} = \sqrt[4]{64\pi^2 \rho g \gamma \times 3 \cdot 10^4} \quad (2)$$

where: E_{cr} is the critical intensity of the electric field; ρ is the metal density; g is the free fall acceleration; γ is the superficial tension of the material in liquid state (the values calculated according to the last relation are: $16.1 \cdot 10^6$ V/m for Tungsten, $12.7 \cdot 10^6$ V/m for Fe, $8.6 \cdot 10^6$ V/m for Ti, $13.2 \cdot 10^6$ V/m for Mo).

At the same time, it is necessary to accumulate a critical charge density σ_{cr} on the surface of the electrode subjected to processing equal to [17] [20]:

$$\sigma_{cr} = \sqrt{\frac{\rho g \gamma}{4\pi^2}} \quad (3)$$

In order to form the liquid phase of the material on the processed surface, it is necessary to ensure a critical current density for its amplitude value in the gap, which can be calculated with the relation:

$$j = \frac{4I_{max}}{\pi d_c^2} \quad (4)$$

where: j is the current density in the gap during a singular discharge; I_{max} is the value of the current amplitude in the impulse; d_c is the diameter of the erosion crater that will form on the surface of the workpiece (experimentally it was determined that the value of the critical current density for Fe is 129.6 A/mm², for Ti 160.4 A/mm² and for Cu 369 A/mm²).

It was found that the height of the asperities (Figure 2) is a function of: the discharge energy, the discharge duration, the material of the electrodes, the intensity of the electric and magnetic fields applied to the gap [14].

In some of the published works [21] [22] it is mentioned that nanometric asperities are attested on the surface of the crystallization grains of the liquid phase resulting from the interaction of the PDEM plasma with the metal surface (Figure 2(a)). We believe, that in this case we would be entitled to support the hypothesis, that the PDEM is born on "cold" electrode spots which are the support for the

elementary conductivity channels, which attract according to Lorentz's law, and merge giving rise to the "hot" electrode spots, which cause melting, disturbance, extraction of conical asperities, rupture of particles or freezing of asperities, etc., but in the end, the discharge is completed by splitting the "hot" electrode spots into "cold" electrode spots, which interact with the still hot surface of the periphery of the processed surface on which it causes extracts asperities of nanometric dimensions (see **Figure 2(b)**).

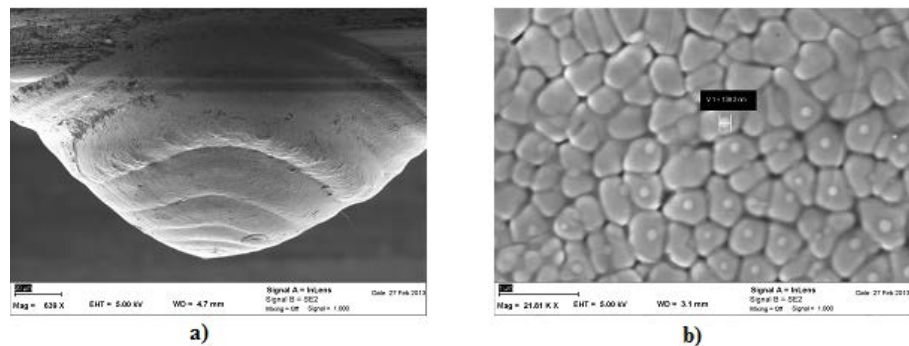


Figure 2. View of the conical roughness extracted from the anode executed from W + 10%Re ($U = 70$ V, $C = 200$ μ F, $S = 0,3$ mm, $d = 0.25$ mm, $n = 1$). Lateral surface of the roughness [18]: (a) The presence of concentric waves; (b) The presence of mosaic blocks on the lateral surface of the conical roughness.

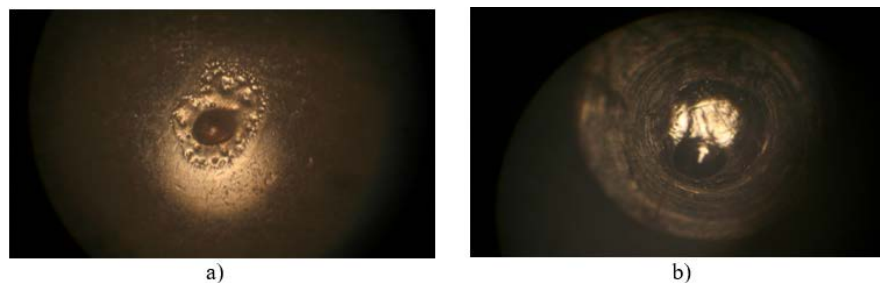


Figure 3. Meniscus extracted from powder particles: (a) $C = 500$ μ F, $U = 40$ V, $W = 0.4$ J, $S = 0.2$ mm, material - bearing steel 52100; (b) $C = 100$ μ F, $U = 89$ V, $W = 0.4$ J, $S = 0.2$ mm, material - niobium.

If most of the results analyzed in the paper were focused on such materials as tungsten and its alloys or titanium, in order to confirm that the above are not random things, or attempts were made to extract the menisci and on the surfaces of the parts made of steel and niobium, the results were confirmed and are presented in **Figure 3**.

Figure 4 presents images of samples with semicircular active surfaces from which conical asperities were extracted following PEDM at different processing energy regimes.

It can be seen from **Figure 4(a)** that the shape of the extracted meniscus is more elongated than the meniscus from **Figure 4(b)** because in the working process, the gravitational attraction force component acts.

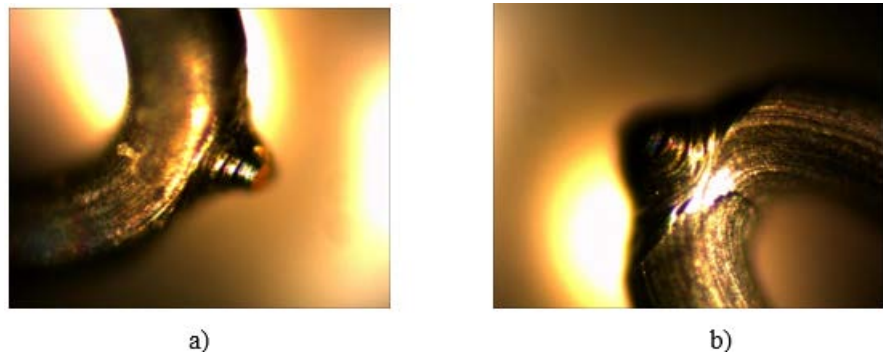


Figure 4. General view of the meniscus extracted from the active surface of the cathode for electron emission: sample and tool-electrode material W+10%Re; $U = 80$ V, $C = 600$ μ F, $S = 0.3$ mm, $n = 1$, $\varnothing_{\text{tool}} = \varnothing_{\text{sample}} = 0.2$ mm; (a) The piece was positioned at the top as anode; (b) The piece was positioned at the bottom as anode.

Of course, if conical asperities are formed under air conditions, they oxidize and their application in the field of electronic thermo-emission is less efficient. In the case of the use of cathodes processed with PEDM in the argon medium, a completely different character of the volt-ampere characteristics was obtained for the roughness sample and for the smooth cylindrical surface (see **Figure 5**).

If we analyze the facts presented in **Figure 5**, we note that in the case of thermocathodes whose surface contains conical meniscuses, under the same operating conditions as those of cathodes with smooth surfaces, we observe that the intensity of the emission current (in saturation mode) is approximately 9 - 10 times higher [11].

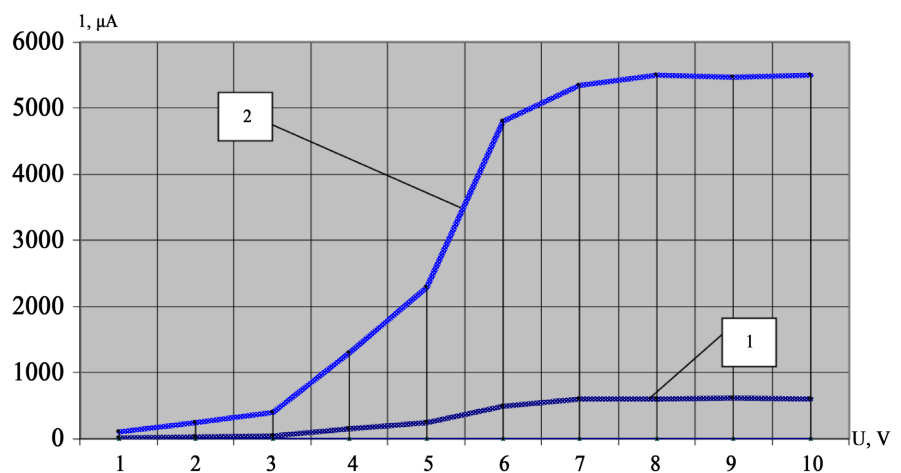


Figure 5. Emission volt-ampere characteristic obtained under express conditions, in which the potential difference between the cathodes remained a constant value [11].

The tests on increasing the thermo-electronic emission capacity of cathodes made of W + 10%Re alloys [21] [22] demonstrated that for the same operating regime, in the case of applying only a single conical asperity on its active surface, the intensity of the emission current increases by about 10 times.

3.2. Results Regarding the Microwelding of Cylindrical Tungsten Wires

Microwelding of the electrodes and short-circuiting of the electrical circuit was performed by the authors of the paper [11] at the gap values smaller than 0.15 mm. This possibility of microwelding offers new opportunities for obtaining junctions between wires of different origins. At the base of this process [23] is placed the action of the electric fields and the strong thermal fields generated by the “hot” electrode spots. As we can see from **Figure 6**, the weld site has a smaller cross-section, and in the case of electric current passing through this area in various circuits, it can perform various functions, from the area of active resistance, the construction of electrical voltage dividers in electronics, to the thermocouple function in electrical measuring devices.

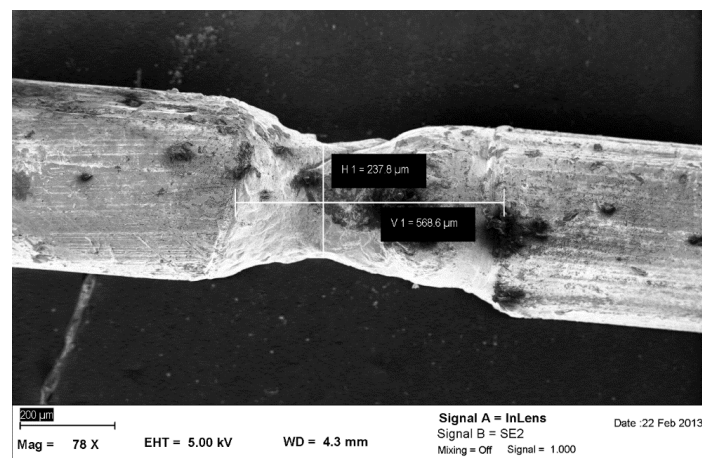


Figure 6. Microwelding of W + 20%Re wires with diameters of 0.4 mm.

The ones presented in **Figure 7** demonstrate that through the PEDM method several wires can be welded, and practically, such constructions can generate new applications or possibilities for exploiting the process.

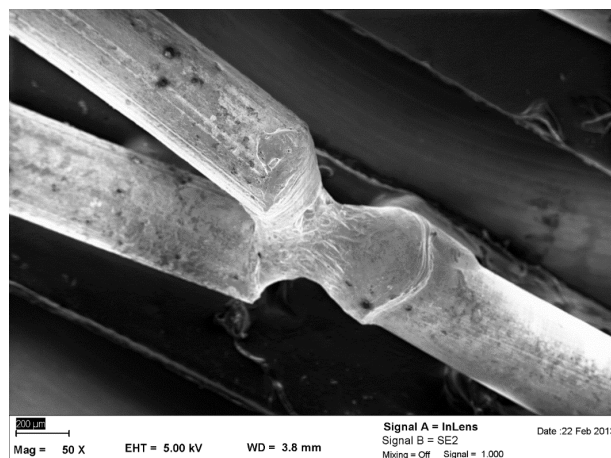


Figure 7. Microwelding of three wires made of W + 20%Re with diameters of 0.4 mm.

Microwelding of three wires of various natures, could lead to the manufacture of transistor products for microelectronics applications.

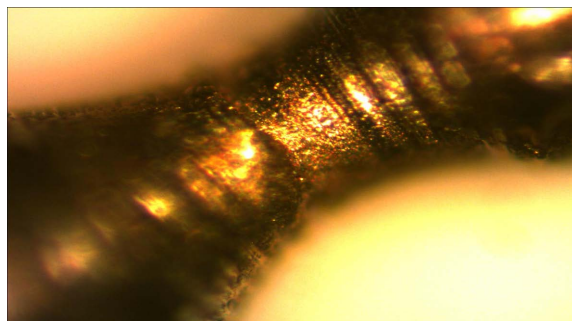


Figure 8. Microwelding of Ni and Cr wires.

Microwelding of Ni and Cr wires that can be applied directly to obtain thermocouplings are presented in **Figure 8**. The welding process occurs for solitary discharge energy with value of 1.2 J, at the gap $S = 0.3$ mm. This result was observed by repeating the discharges at a frequency of 5 Hz. The authors substantiate the process by the development of capillary waves, which cause the extraction of Taylor cone-type asperities and their fusion into a liquid state at the end of the pulse of the electrical discharge. The capillary waves develop on the surfaces of both the cathode and the anode, and finally when the asperities merge and the discharges are repeated, the microwelding point is strengthened.

3.3. Extraction of Nanowires from the Surfaces of Parts Made of Al-Sn Alloys

The authors of the papers [24]-[28] have experimentally verified the possibility of extracting nanowires for the case when the electrode manufacturing alloy subjected to PEDM processing presents in a solid state a mechanical mixture of the crystals of the chemical elements in the composition.

Thus, the authors of the papers [24]-[27] obtained nanowires of SnO_2 on the surface of the sample made of the Al-Sn binary alloy under the action of the PEDM plasma in air conditions at atmospheric pressure and room temperature (see **Figure 9**). After the SEM/EDX measurements, the chemical composition of these nano-wires was determined (see **Table 1**).

Table 1. Chemical composition of nanowires.

Chemical element	Al	Sn	O	N	C
Mass, %	19.1	61.5	11.5	4.9	3.0

It can easily be seen that the concentration of Sn is considerable. The analysis of the composition of the nanowires proves that it is basically tin dioxide. It is natural to form oxides given the fact that the processing takes place in the atmosphere under ordinary conditions, and the components of the electrode execution

alloy have an avidity for oxygen.

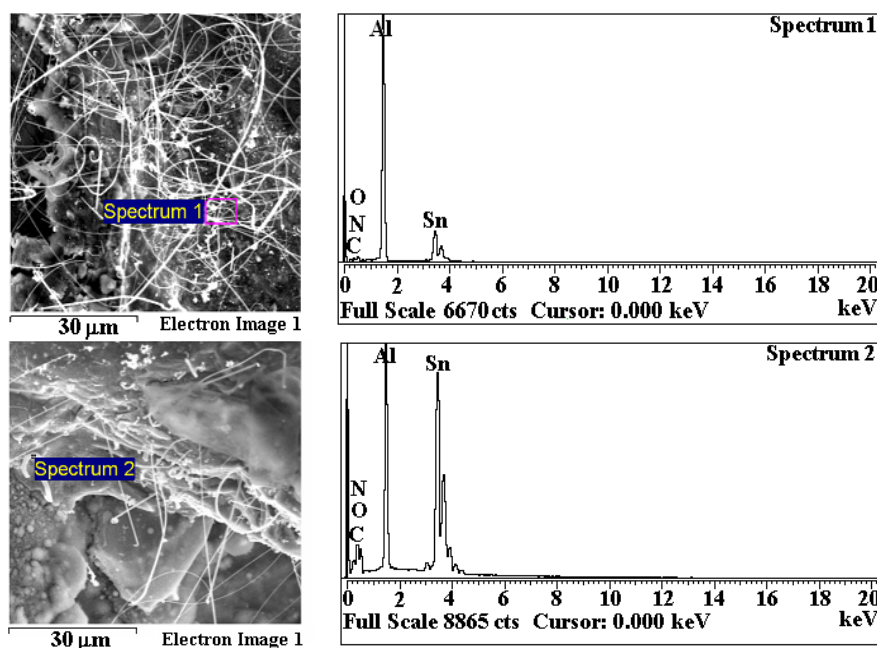


Figure 9. Morphology and EDX spectra of the electrode-anode and workpiece-cathode surfaces after processing.

The explanation of the physics of the formation of nano-wires interpreted by the authors [27] [28] is not a plausible one, given the fact that they put forward the hypothesis only emerging from those “as the alloy from which the part is made covers an aluminum matrix in which the tin is dissipated, which melts being thrown into the gap in the form of a drop, and it is deformed under the action of the electromagnetic field, up to the state of nanowires”.

From our point of view, things are not like that. If we take into account the fact that the melting takes place under the action of the electrode spots, which according to [29], present concentrated sources of both heat and strong electric fields ($10^6 \div 10^8$ V/m), these being distributed according to the radius vectors of the point source, we could have the argument that the obtained wires have a cylindrical shape. If we refer to their length, then based on the fact that the surface of the liquid metal is electrified, and the “hot” electrode spot moves (migrates) at a speed of about 30 m/s and entrains the molten and electrified metal, we could obtain nanowires of considerable lengths as shown in **Figure 9**. The distribution of the wires on the processed surface attests to this, having a configuration characteristic of the trajectory of the displacement of the electrode spots! The length of the wires can be obtained only due to the movement of the electrode spots ($V = 30$ m/s) and in no case of the electrodes, which for the deposition formation installations is $V = 10^{-2}$ m/s and the duration of a discharge is the order of 10^2 μ s.

Of course, in this situation, the fact that Sn melting takes place at considerable depths accompanied by the expansion of the solid aluminum matrix, which facil-

itates even more the action of the electric fields generated by the electrode spots.

The electrical discharges occur at the gaps of $S = 0.03$ mm, the energies of 0.3 J and the durations of 15 microseconds. The extraction and solidification of nano Sn nanowires occurs in an air environment at normal pressure and room temperature. Tin being very greedy for oxygen, excessive oxidation of the surface of the nanowires occurs.

The nanowires generated by this method are relatively cheap and could solve the problem of electrical conductors for contemporary electronics.

3.4. Generation of Fullerenes and Carbon Nanotubes

The formation of graphite films at micrometric and nanometric scales on the surfaces of alloys-made parts causes their diffusion in the surface layer accompanied by the formation of high hardness carbides, and, as a result, the wear resistance of this layer increases [30]-[33].

The process of formation of graphite films, in all cases, leads to a decrease in surface roughness of the processed surface. According to the results obtained by the authors [34]-[36], the application of films on the surfaces of the components that work in cinematic couples, leads to a decrease of the friction coefficient of at least 3 times.

Many research and SEM attempts were done to unravel the phenomena occurring at the surface of the processed piece and at the surface of the tool-electrode.

Thus, in **Figure 10** and **Figure 11**, the morphology, as well as the chemical composition of the deposition electrode tool is presented. We can observe the mosaic block surface of the electrode-tool, magnified on micrometric scale and applied for depositing accomplishment, its chemical composition consisting of 100% carbon.

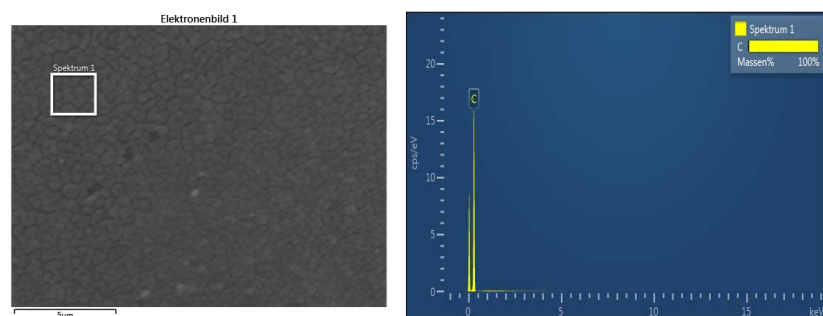


Figure 10. General view of the tool-electrode surface and its chemical composition.

A significant line of modifications of the surface morphology occurs as the result of electric discharges in impulse under plasma action and bombarding ions of plasma generate gas. The globular formations with nanometric size are confirmed on this surface (**Figure 11**). The globular formations, observed on the electrode tool surface, may be explained as the result of two concurrent phenomena, that have simultaneous manifestation: heating, melting and graphite recrystalliza-

tion under the influence of the plasma energy, on one side, and ions bombarding generating plasma gas, on the other side.

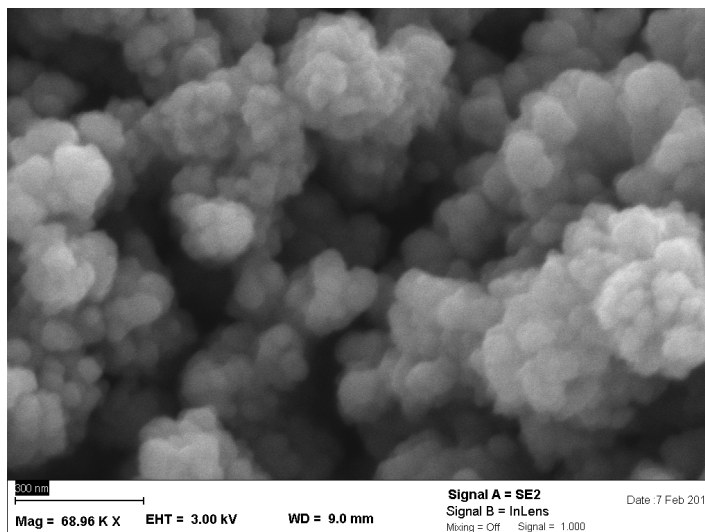


Figure 11. Surface morphology of cathode pyrolytic graphite tool-electrode after the PEDM plasma influence.

SEM of pyrolytic graphite, investigated on the tool-electrode, shows the formation of macro formations (**Figure 10**) on their surface, assembled by the discovered nanostructures (**Figure 11**). This leads to the conclusion that a chain of specific erosion phenomena occurs.

If we try to give an explanation to the phenomenon of fullerenes and carbon nanotube growth in the case of the application of electrodes made of pyrolytic graphite [6] [37], then we could start from the shape they have. We could say with certainty that in the beginning fullerenes are formed, and then, of course not in all cases, under the influence of temperature fields and powerful electric ones, their development takes place in single-walled nanotubes.

Thus, the SEM analysis at the beginning made it possible to establish the formation of spherical formations on the surfaces of the electrodes (**Figure 11**).

In the first view, the authors [38] [39] admitted that graphite deposits are formed on metal surfaces and especially on the surfaces of samples made of steel C45. If we consider that the deposits are graphite formations, then knowing the constant of its crystal lattice and considering the fact that when two parts of a couple interact, their surface is worn by breaking the deposited formations layer by layer. Taking into account the thickness of the deposition as the maximum and minimum value, knowing the constant of the crystal lattice of graphite, we would determine the maximum number of cycles it can withstand, but the behavior of the deposition does not correspond to the usual wear of graphite, as demonstrated by the results obtained by the authors [37] [40]-[42] reason from which Raman analyzes of the carbonic deposits were performed, in order to elucidate what are in reality the carbonic structures that are formed during the vaporization of pyrolytic graphite

in PEDM plasma in the presence of iron atoms from the vaporization of the target that in this situation served as the anode.

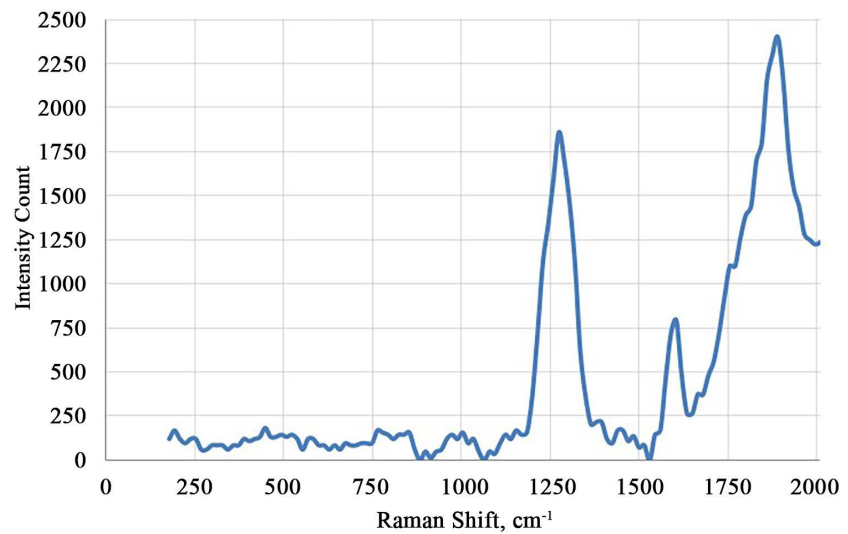


Figure 12. RAMAN spectrum of the carbon film ($S = 0.8$ mm, $W = 3.2$ J, $f = 50$ Hz).

Thus, if we compare the Raman bands of the carbon depositions on the samples' surfaces shown in **Figure 12** and **Figure 13**, then corresponding maxima “1281”, “1596” and “1899” are attested on them, followed by “1280” and “1843”, and these correspond to the characteristic structures of fullerenes and single-walled carbon nanotubes.

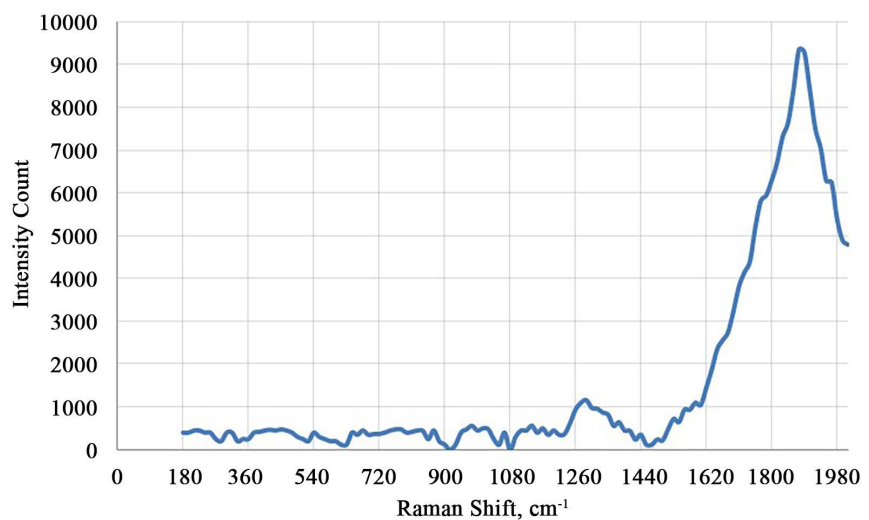


Figure 13. RAMAN spectrum of the carbon film ($S = 2$ mm, $W = 5.23$ J, $f = 50$ Hz).

The functional properties of the graphite deposits [37] [42], their chemical composition and morphology indicate the fact that they present 3D structures and especially of carbon nanotubes. The presence of iron on the target, the purity of the graphite in the electrode, as well as the energetic conditions in the plasma of

the pulse electric discharges ensure sufficient and necessary conditions for the synthesis of carbon nanotubes.

The presence of the sample made of steel containing an important amount of iron stimulates the formation on its surface of 3D type formations (fullerenes or nanotubes). These goals are confirmed by the behavior of depositions at thermogravimetric actions (stating mass increase under the influence of nitrogen at the temperature of 222.99°C, 476.12°C and 614.73°C) and by solubility tests in different environments: it varies from 51 for chlorine-naphthalene down to 0.006 for tetrahydrofuran).

Carbon films formed by applying electrical discharges in impulse possess a set of beneficial functional properties [37] [43] [44], such as: decrease the surface adherence by $3 \div 4$ times, decrease the wear coefficient from $1.5 \div 2$ down to $7 \div 10$, increase the wear resistance of piece components of glass molding forms, condition the increase of corrosion resistance in chemically aggressive media by 1.5 times.

A series of exploitation tests were carried out at the State Enterprise “Glass Factory” from Chisinau, for the constituent parts of the glass casting molds. Thus, if we analyze those presented in **Figure 14(a)-(b)** we can see that the film is formed by clusters of nanometric formations. Between these clusters and respectively between the formations that constitute them, there are gaps (pores), which can explain a series of properties that they possess.

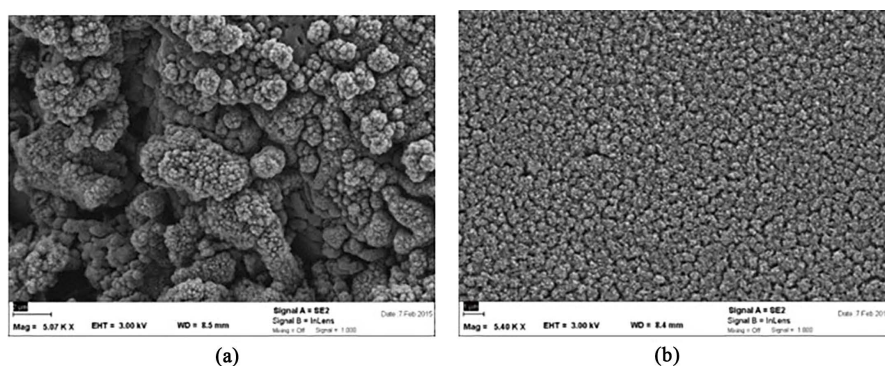


Figure 14. SEM image of the surfaces of hardened metal parts by applying PEDM with a graphite tool-electrode: (a) Low frequency generator; (b) High frequency generator.

As a result of the formation of the graphite film on the surface of the part, the effective diameter of the part increased on average by approximately 14 μm compared to the initial diameter, *i.e.* as a result we have graphite deposits of the respective size on the surface in the form of a continuous film.

Thus, the application of the films on the surfaces of the constituent parts of the glass casting molds allowed to establish their very efficient functionality. The parts were provided with a durability of at least 2 times greater than the parts coming from the factory. This fact can also be explained by the fact that graphite has an ointment in the solid state and prevents the adhesion of the glass to the surface of the part, and respectively its wear by adhesion, as well as by the fact that the

graphite film possesses anti-refractory properties and serves as a thermal insulator between the surface of the metal part and the liquid glass.



Figure 15. General view of the punch surface of the plunger of the glass casting molds: (a) Initially; (b) After machining [37].

The above is also confirmed by the results obtained by the authors of the paper [42] who tested the plungers in real operating conditions (Figure 15). As a result, it was established that the plungers of the casting molds on the active surface of which graphite films were formed functioned at 57600 cycles without changing their shape and dimensions. In this regard, in order to compare the wear of the plungers of the glass casting molds, experimental research was carried out in the technological cycle [45]-[47].

3.5. Oxide Pellicle Formation

As was already mentioned in the papers [48], the interaction of the electrode surfaces with the plasma of PEDM takes place by means of “cold” and “hot” electrode spots. “Cold” electrode spots cause minor transformations in the electrode surfaces and cause their enrichment with the elements of the environment in the plasma gas. In their research, the authors [49] discovered that in air conditions at atmospheric pressure and room temperature, upon the repeated action of electrical discharges on a portion of the surface, of the cathode electrode, it changes its color from that of the actual metal to that of freshly picked metal.

Analyzing the results obtained by the author of the work [18], it was established that in order to obtain the first type of interaction with the plasma canal on the surfaces of the parts, it is necessary that the energy density on the processed surface be lower than the specific heat of melting of the material and the latter can be estimated with the relation [48]-[50]:

$$Q = \frac{4W}{\pi d_c^2 S} < Q_{melt} \quad (5)$$

The parameters in the relation (5) are the same as in the relation (1).

The detailed investigations [35] of the surfaces processed in the regime of maintenance of electric discharges on “cold” spots by Mossbauer spectroscopy of steel samples (Figure 16) indicate both spectra and represent an elusive superposition of doublets of iron oxides and hydroxides and confirm the presence of the γ -Fe phase for $S = 0.5$ mm.

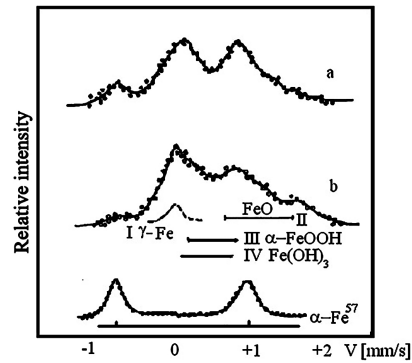


Figure 16. Mossbauer spectra of steel A284Gr.D samples processed by PEDM: $W = 4.5$ J; $S = 2$ (a); $S = 0.5$ mm (b).

The distribution of iron, oxygen, carbon and nitrogen is presented in **Figure 17(a)**, from which it can be observed that carbon agglomerates on the surface. Its concentration decreases quickly with depth, and the oxygen concentration reaches up to 50% at.

This increased oxygen concentration can be achieved by the formation of hydroxides in the surface layer of the sample. At a substrate depth of up to 300 nanometers, the nitrogen concentration is up to 5% - 6% at.

At the gap value of $S = 2$ mm, the basic components of the surface are iron and oxygen, and the secondary ones are, respectively, carbon and nitrogen (**Figure 17(b)**). The oxygen concentration reaches 60% at the surface and decreases a lot in the depth of the sample, the variation that can be explained by the fact that hydroxides are formed on the surface and metastable oxides are formed at depth, according to roentgenograms at the surface of the sample lacks FeO (the only paramagnetic iron oxide at room temperature).

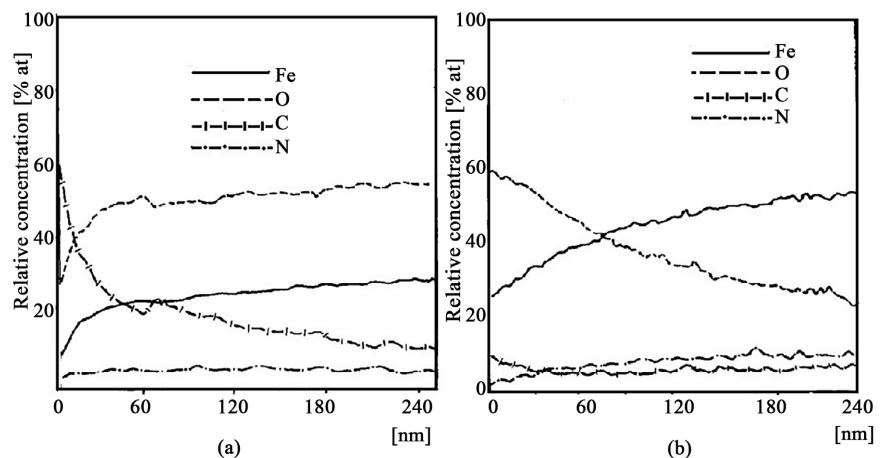


Figure 17. Concentration distribution of elements (Fe, O, C, N) in steel A284Gr.D after processing by PEDM for: $S = 0.5$ mm (a); $S = 2$ mm (b).

The metastability of very thin layers of Fe_2O_3 and Fe_3O can be also admitted, a fact that is well known [49]. The possibility of forming iron hydroxides in an

amorphous state is also not excluded [49]. The complexity of Mossbauer spectra shows that the univocal identification of amorphous hydro-oxides is very difficult.

Thus, the analysis of the diffractograms demonstrated that among the elements of the processing regimes, the most important ones that act on the phase component of the surface are, respectively, the value of the gap and the time of interaction with the plasma (number of passes).

The penetration depth [49] of these elements in the surface layer of the part is a function of both the energy of the impulse and the size of the gap and can be expressed with the relation:

$$H = kW/AS \quad (6)$$

where: k is a coefficient dependent on the properties of the processed material; W is the energy released in the gap; A is the area of the thermally influenced surface and S is the gap size.

The phase composition determined by the authors [49] [50] established the presence of: a) OH-component; b) $-O^{2-}$ component and c) O-C and O-C = O structures.

The chemical analysis showed the concentration of each component which is C(a):C(b):C(c) = 0.89:1.00:0.50, which means that the film is mostly made up of oxides and hydroxides the base metal of the sample. The additional study of the superficial layer highlighted the possibility of the existence of the fourth oxygen component of the O-H₂ type, but the value of the relative concentration of this component does not exceed 0.15.

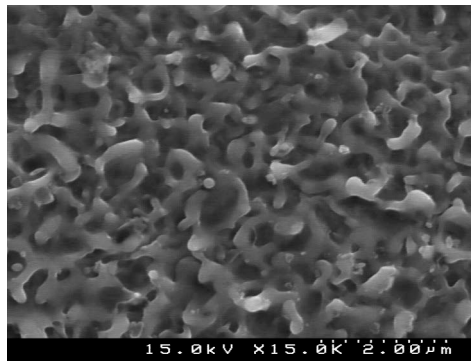


Figure 18. Electronic microscopy TESCAN of the surface layer made of steel C45 processed by applying PEDM.

If we analyze **Figure 18**, which represents the morphology of the processed surface layer, we can see that the quality of the surface with oxide film ($R_a \approx 0.1 \div 0.2 \mu\text{m}$) has decreased in relation to the initial one mechanically prepared ($R_a \approx 0.63 \mu\text{m}$). This phenomenon can be explained by the fact that the sharp points left after the mechanical processing are rounded following the action of the EDM plasma.

The results of the SEM and EDX [46]-[53] analyses of the samples processed at the optimal regime established are presented in **Figures 19-22** and in **Table 2**. Thus, it was observed abnormal dissolving of oxygen up to 60% in the steel surface.

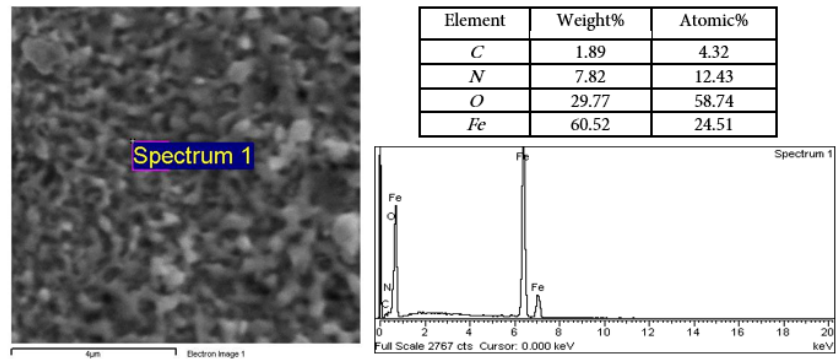


Figure 19. SEM and EDX analyses of steel C45 samples processed by PEDM.

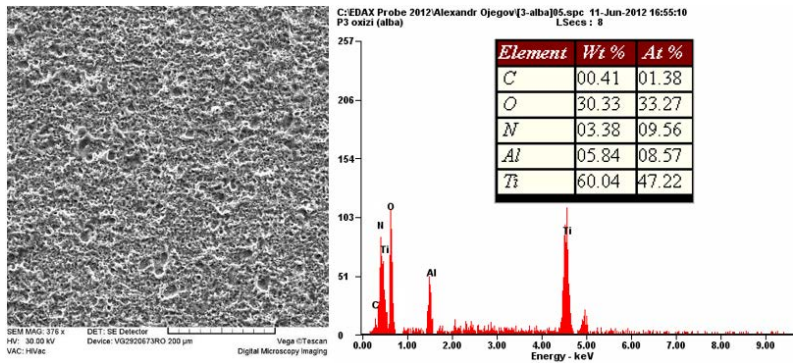


Figure 20. SEM and EDX analyses of VT8 (TiAl₆Mo₄) titanium alloy samples processed by PEDM.

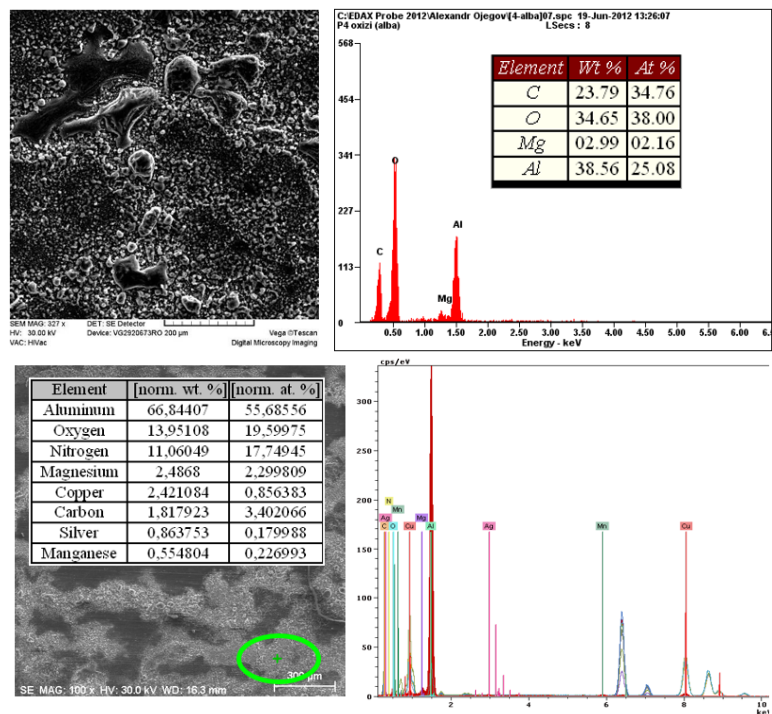


Figure 21. SEM and EDX analyses of aluminum alloy D16 (its analogue is AlCuMg₂) samples processed by PEDM.

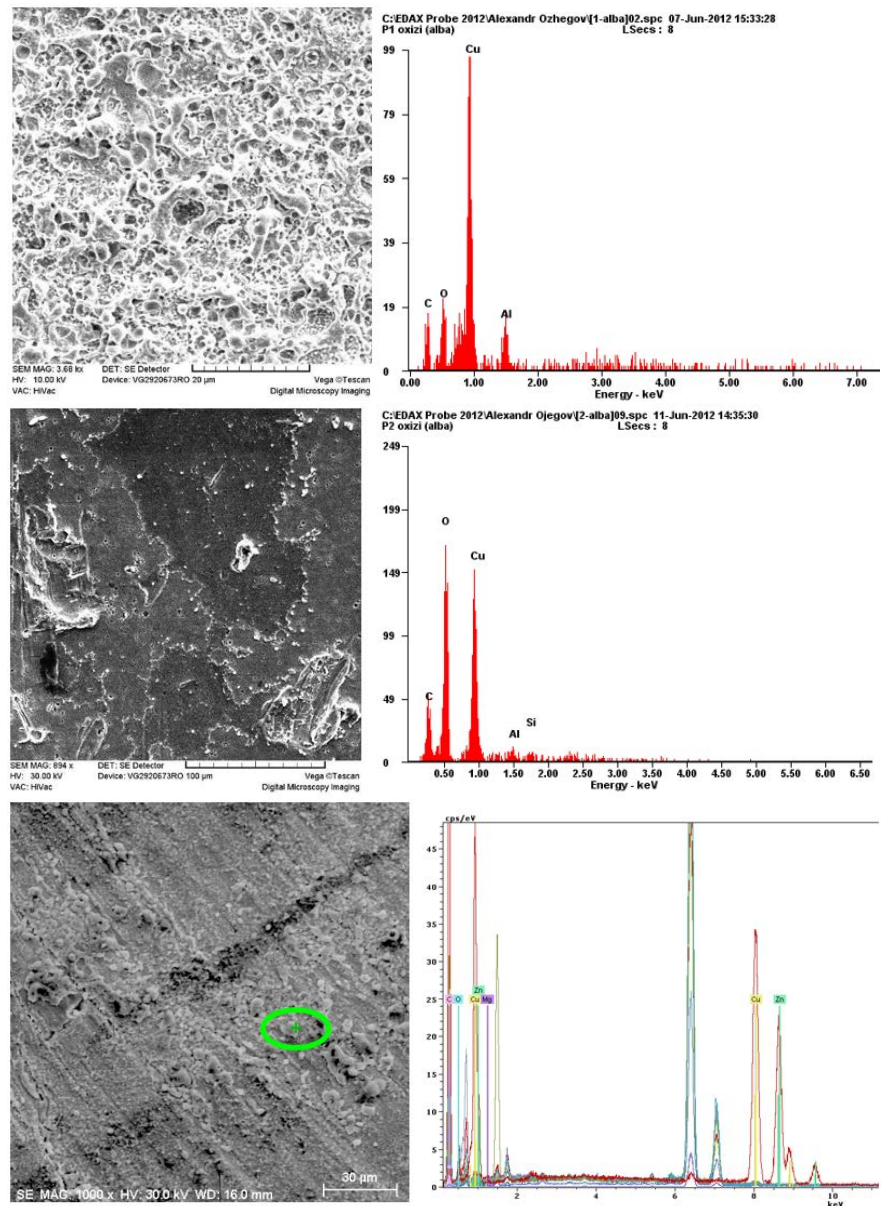


Figure 22. SEM and EDX analyses of copper alloys (M0 technically pure copper, BrA5 (CuAl₅) bronze and L63 (CuZn36) brass) samples processed by PEDM.

Thus, it was found that the dissolution of oxygen during the processing of construction steel samples reaches 60% at., from titanium alloys 30% - 35% at., those made from aluminum alloys up to 20% at., and those from copper alloys 50% at. It is found that in alloys with a high copper content, the dissolution of oxygen is significant and proportional to the amount of copper in them. In the case of aluminum and titanium alloys, the amount of oxygen is lower, due to the fact that they undergo the process of volatilization and erosion. The superficial layer of the surface of the samples processed from steel and titanium alloys besides oxygen also includes nitrogen. The different dissolution of oxygen in metals is explained by their different avidity towards oxygen and the electro-physical and chemical

properties of the constituent elements.

Table 2. EDX analysis of machined surfaces for parts made of different alloys.

Base material of the sample	EDX chemical content of the processed surface		
	Element	[norm. wt.%]	[norm. at.%]
Steel C45	Carbon	1.89	4.32
	Nitrogen	7.82	12.43
	Oxygen	29.77	58.74
	Iron	60.52	24.51
Titanium alloy VT8	Carbon	00.41	01.38
	Oxygen	30.33	33.27
	Nitrogen	03.38	09.56
	Aluminum	05.84	08.57
	Titanium	60.04	47.22
Aluminum alloy D16	Aluminum	66.84	55.68
	Oxygen	13.95	19.60
	Magnesium	2.49	2.30
	Carbon	1.82	3.40
	Copper	2.42	0.86
	Manganese	0.55	0.23
	Silver	0.86	0.18
Technically pure copper M0	Copper	59.5	25.55
	Oxygen	29.53	50.50
	Carbon	10.22	23.20
	Aluminum	0.75	0.76
Bronze BrA5	Copper	52.97	21.18
	Oxygen	26.66	42.34
	Carbon	14.83	31.38
	Aluminum	02.66	02.50
	Silicate	02.87	02.60
Brass L63	Copper	42.30	23.25
	Zinc	32.89	17.57
	Oxygen	16.72	36.50
	Carbon	7.52	21.86
	Magnesium	0.57	0.82

Corrosion resistance tests of oxide and hydro-oxide films applied to the surfaces of parts made of iron alloys have shown that the corrosion potential increases by more than one volt, and the corrosion resistance can increase by up to 100 times in the aggressive media.

In the case of surface oxidation of parts made of C45 Steel, exceptional results were also attested regarding the increase in active surface resistance (see **Table 3**). Initial measurements showed that for samples made of C45 Steel, the active surface resistance is only 0.05 Ω . These results confirm that the plasma interaction process at PDEM is complex and of an electro-physical-chemical nature and accompanied by the effect of cathodic dissipation, as the authors mentioned. At the same time, we could admit that the ions with positive charge in the plasma prevail about three times over those with negative charge. From the results presented in

Table 3, we could admit the possibility of producing elements for electronics with very small dimensions, given the fact that an increase in the active surface resistance of at least 10^7 times is attested.

Table 3. Surface electric resistance of oxide films for C45 steel samples.

Sample	Surface electric resistance of samples with oxide films, $\times 10^6 \Omega$				Average value
	Experimental data				
Cathode	0.88	0.92	1.46	0.91	1.03
	0.97	1.52	0.86	0.92	
	0.99	1.09	0.86	0.83	
	1.33	1.10	1.04	0.85	
	1.07	0.88	1.21	0.89	
Anode	0.81	0.26	0.46	0.31	0.33
	0.11	0.14	0.56	0.34	
	0.29	0.11	0.62	0.38	
	0.87	0.38	0.11	0.15	
	0.12	0.13	0.27	0.17	

4. Conclusions

From the analysis presented above, we can conclude that:

- 1) Knowledge of the phenomenology of plasma in various environments, the legitimate behavior, and of course its application in material processing technologies could provide solutions to the crises that humanity is currently facing;
- 2) The interaction of the plasma with the surfaces of the samples made of various materials causes thermal, chemical-thermal, morphological, structural and phase changes in them;
- 3) The extraction of Taylor-type conical asperities on the cathodes' surfaces ensures a 100-fold increase in the intensity of the thermoemission current under the same operating conditions of the cathode;
- 4) Micro welding of metal wires is possible for microelectronics applications;
- 5) The application of carbon or oxide films on the workpiece surfaces ensures the decrease of surface adherence by $3 \div 4$ times, the decrease of wear coefficient between two couples, the increase of the wear resistance, the increase of active surface resistance up by 10^7 times, the increase of resistance to corrosion in aggressive environments from 2 to 100 times.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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