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SPECTRAL AND SEM ELECTRON MICROSCOPY ANALYSIS OF GRAPHITE FILM, DEPOSITED BY THE PROCEDURE OF ELECTRICAL DISCHARGES IN IMPULSE IN UNDER-EXCITATION REGIME, USING PYROLYTIC GRAPHITE CATHODE

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Abstract: An electric pulse generation device, pyrolytic graphite cathode electrode, and metal plate anode electrode were utilised in many attempts to deposit graphite films onto metal surfaces. The electrical pulse generating mechanism employed was underexcitation, characterised by low-energy electric pulses. These low-energy pulses caused the graphite on the cathode electrode to vaporise, but did not result in the melting of the metal surface on the anode. By implementing this method, we successfully produced graphite films that measured around 6 microns in thickness and exhibited excellent adhesion to the metal surface. The graphite film underwent spectral analysis using X-ray spectroscopy, EDX, and SEM electron microscopy to ascertain its structure.
Key words: graphite films, electric pulse, X-ray spectroscopy, EDX. Spectroscopy, SEM electron microscopy.

1. INTRODUCTION

This work seeks to elucidate, to establish the technological parameters and to determine as precisely as possible all the scientific aspects that determine the graphite film deposition process. For this, the graphite film was subjected to spectral analyses X-ray spectroscopy, EDX spectroscopy as well as SEM electron microscopy. The research works were carried out in the laboratories of the Technical University of Moldova Chisinau, the Alecu Russo State University in Balti - Republic of Moldova and in the laboratories of the National Institute of Research - Development for Chemistry and Petrochemistry - ICECHIM Bucharest Romania. The electric pulse generator as well as all the other necessary devices with which the graphite film was deposited on the metal surfaces were provided by the Alecu Russo State University - Balti, Republic of Moldova, the devices with which the spectral analyzes were performed were provided by the Technical University of Moldova Chisinau and the SEM electron microscope was provided by ICECHIM – Bucharest. The scientific literature mentions methods of depositing thin films of various chemical natures (metallic or non-metallic) on various surfaces - also metallic or non-metallic, The literature mentions deposition methods including carbon films - amorphous carbon, diamond, or graphite but it is not mentioned as a procedure of spreading pulsating electric arc. this procedure is experimentally applied by the research team for unconventional technologies from the Alecu Russo State University in Balti. The attribute of experimental gives to the process of depositing graphite films both degree of novelty and possibilities for improvement. The innovative idea that led to the start of research on the process of depositing graphite films through the process of pulsating electrical discharges was the next one: An electric discharge between a graphite cathode electrode and an anode - considering the particularly high temperature of the electric arc of 6000 degrees Celsius - vaporizes a microportion of the graphite from which the cathode electrode is made. The flow of electrons that flows from the cathode to the anode that makes up the electric discharge takes graphite vapors and transports them along the length of the anode-cathode interstice. When meeting the cold metal anode, the graphite vapors condense and settle on its surface, forming a thin film of around 2-4 microns that adheres to the surface of the anode.
Graphite films have practical applicability where other protective films either do not withstand technological conditions such as particularly corrosive acid or basic environments or where there are parts in dynamic contact to reduce the coefficient of friction between them.

2. METHODS AND MATERIALS

In the research works, the method of electrical impulse discharges (DEI) is used, in the underexcitation regime, with the use of "tool electrodes" made of pyrolytic graphite. As mentioned in a number of scientific papers, [1-4, 26], the graphite electrode being a non-metal, can be used in the discharge circuit with different polarities – anode, cathode and combined mode polarity. When using the "tool-electrode" made of graphite with anode polarity on the surface of the part, heat treatments take place with the diffusion of graphite in the surface layer, which leads to an increase in the microhardness by about 5 times compared to the base metal, [2]. In the case of using the combined "electrode-tool", [1], the micro-hardness increased 2-3 times compared to the base material, and a graphite film of micrometric dimensions is also obtained on the surface of the piece, the thickness of which depends on the processing regimes When using the cathode processing regime, the microhardness of the superficial layer can be increased by about 10 times compared to the basic one, [2]. The deposition of graphite films has been applied in industry, namely to the thermal treatment of the plungers of the glass molding molds, [5, 6]. For this technological operation, a series of metal samples were used, made from an embossing sheet format (type A3 – for deep embossing) obtained by lamination on an industrial scale, material OL 37.

2.1. The experimental device for the generation and application of DEI

The pulse generator for the formation of graphite films on the metal surfaces of the parts with the application of electrical discharges in impulse, includes the following electrical blocks, figure 1: the power pulse generator, the priming block (intended for the initiation of electrical discharges) and the control block, whose role is to to synchronize power and priming pulses, it was concluded that, for the application of graphite films on the surfaces of conductive parts, RC generators with parallel priming can be successfully used, [27].

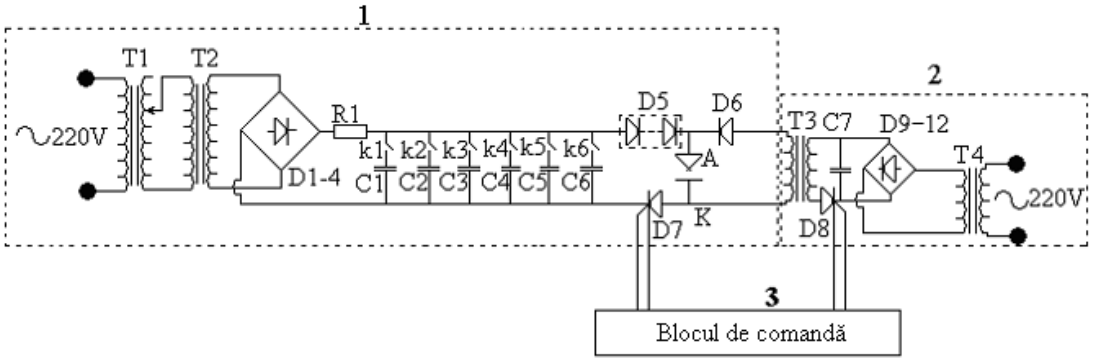


Fig. 1. Electrical diagram of the pulse generator intended for the formation of graphite films, [27]: 1 – power pulse generator; 2 – the priming block; 3 – the command block)

2.2. Graphite electrode

The electrodes - are graphite sticks with a thickness of Ø 3 mm and a length of approx. l = 50 mm (figure 2). they are obtained from high purity graphite - pyrolytic graphite with a low binder content. The end of the electrode that reaches the vicinity of the metal anode and that generates the electric discharge in a pulse is mechanically processed in the form of a hemisphere, [25].

The metal anodes were presented in the form of metal plates measuring 100x25x3 mm, figure 3.

Fig. 2. Graphite electrode

Fig. 3. Metal anodes

2.3. The working methode

The working method for obtaining the graphite films deposited by the DEI process on metal surfaces was as follows: The metal part is cut from an industrial format by cutting without heating in order not to change its

internal crystalline structure to a size suitable for the existing equipment. The pyrolytic graphite electrode having a cylindrical shape and the dimensions shown in figure 1 is positioned in the electrode holder which is coupled to the cathode pole. The anode/electrode gap is measured very precisely. From the experimental determinations, it was found that the optimal gap is 1.5 mm. The pulse generator is switched on and a number of 10 electrical discharges are applied to a point on the metal anode. The number of 10 downloads resulted from multiple attempts to identify it. The electrical impulses were obtained by discharging a capacitor with a capacity of 600 microfarads, the value of the capacity also determined by preliminary tests. The energy released at the gap was 4.8 J, and the regime was one of underexcitation. The formation of graphite films on a micrometric and nanometric scale on the surfaces of parts obtained from steel or other metal alloys is influenced by their diffusion in the surface layer accompanied by the formation of carbides with high hardness and, as a result, increases the wear resistance of this layer, [7-11]. The films formed can reach up to 7 microns in thickness and ensure at least a two-fold increase in the durability in operation of the components of the casting molds, due to the properties of solid lubricant and anti-refractory properties, [11 - 13]. The morphology of the treated surfaces was studied by the SEM method, and the EDX and XPS phase methods were used to study its chemical composition.

3. SPECTRAL AND SEM ANALYSIS OF GRAPHITE FILM DEPOSITED BY PULSED ELECTRICAL DISCHARGES ON STEEL SUPPORT

The experimental research with the formation of graphite films on the metal surface have demonstrated: the analysis of the surface morphology subjected to processing confirmed that the formations on the surface do not exceed micrometric sizes; apart from the initial components of the processed material, a considerable amount of carbon (about 80%) in atomic content is attested, [19], figure 4. It cannot exist in a free state and it forms bonds in the metallic structure forming carbides or in separate structures in the form of graphite, [16].

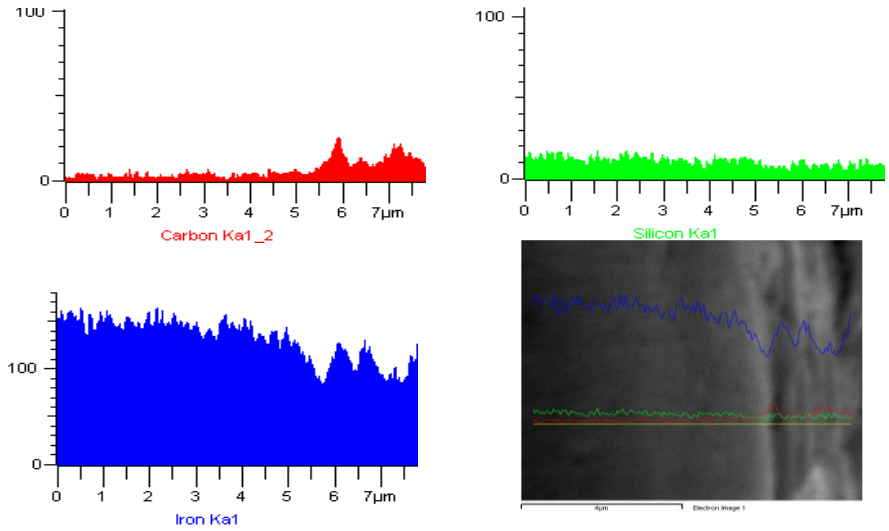


Fig. 4. Carbon distribution in the machined surface, [16 - 18]

Under the conditions of interaction with the DEI plasma containing carbon atoms in an excited state, metallic carbides of the MeC type can be synthesized in the form of particles, fibers and films, figure 5.

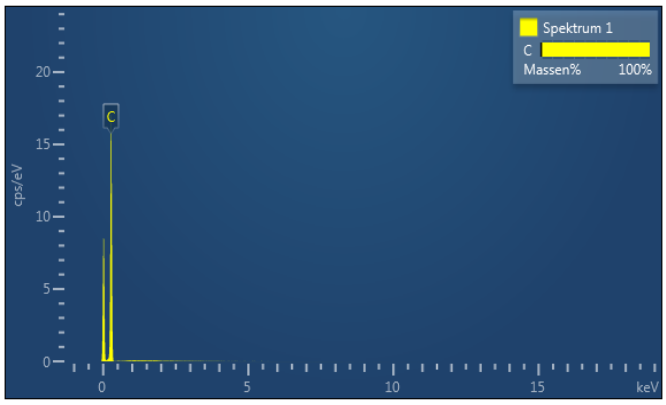


Fig. 5. The chemical composition, determined by the EDX method

In order to ensure the energetic conditions for the formation of carbon deposits on a metallic support, it is necessary to satisfy the condition, equation (1):

$$Q=\frac{4W_s}{\pi d_c^2 S} \approx Q_{top}, \quad (1)$$

where: Q is the volumetric amount of heat released in the interstitium, J/m^3; dc - diameter of the interaction spot of the plasma channel with the processed surface, S - size of the interstitium,m; Qtop = qtop·ρtop is the volumetric specific heat of melting of the material J/m^3; qtop – specific heat of the part material, J/kg; ρtop is the density of the sample material, kg/m^3. The energy released in the gap differs from that accumulated on the capacitor bank of the current pulse generator is determined by the efficiency of the installation η and can be determined with the relation (2):

$$W=\frac{CU^2}{2}\eta \quad (2)$$

where: C is the capacity of the capacitor bank, F; U-its charging voltage, V. The heat generated in the interstitium can be calculated with the relation (3):

$$q = JE \quad (3)$$

where: J and E are respectively the current density and the intensity of the electric field in the gap. Carrying out SEM and EDX analyzes on the samples processed by the method of electrical impulse discharges, it was observed that the weight or mass (Weight) of carbon on the surface of the electrode-part in certain points varies within limits of 50-70%, and the atomic part varies in limits 70-85% (see figure 6 and figure 7), [16].

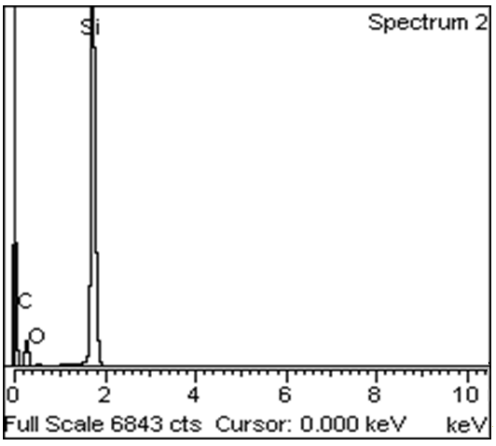


Fig. 6. EDX analysis of metallic piece after DEI processing, [16-18]

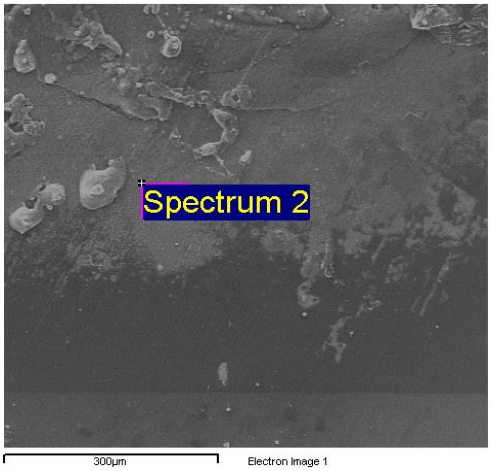
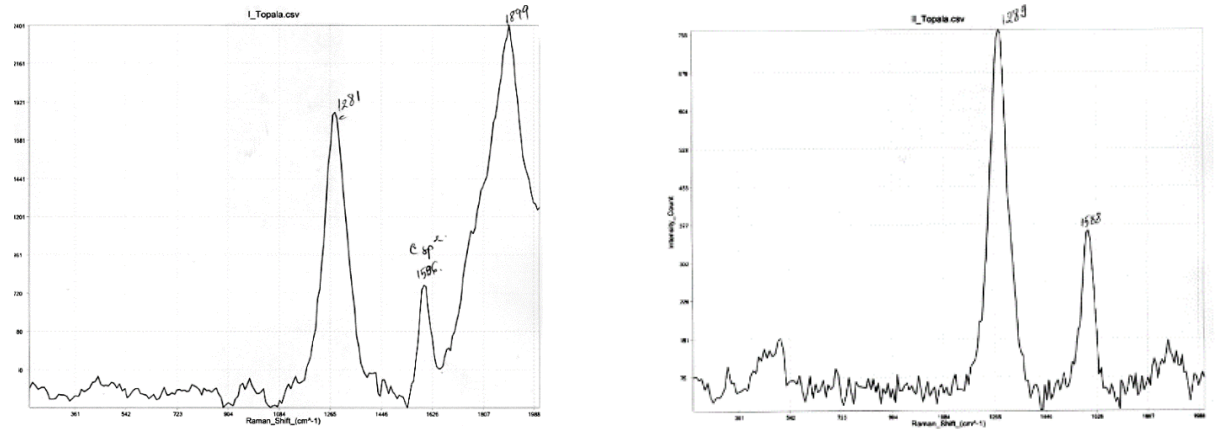


Fig. 7. SEM analysis of the Metallic piece after DEI processing, [16-18]

Table 1. Chemical composition on silicon surfaces, [16,17, 24]

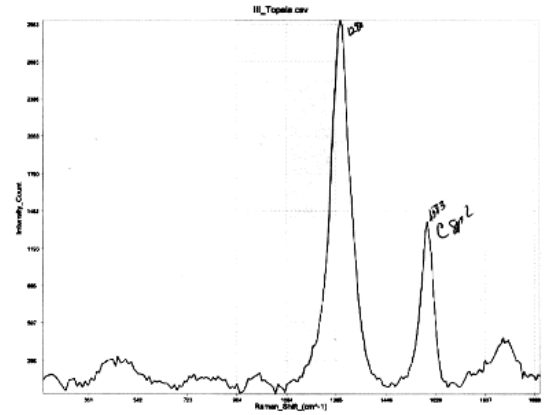
Element	*Weight%*	*Atomic%*
C K	53.25	71.73
O K	3.07	3.10
Me K	43.68	25.17
Totals	100.00	

In order to have a plausible explanation of the results obtained, it is not sufficient to interpret them in terms of the formation of graphite deposits or deposits containing carbon black, both of which serve as solid lubricants and which, are heat resistant. If we consider that the deposits are graphite formations, then knowing the constancy of the crystalline network and assuming that the formations break layer by layer and knowing the thickness of the deposit as the maximum and minimum value, we would determine the maximum number of cycles it can withstand, however, the behavior of the deposit does not correspond to the usual wear of graphite, which is why Raman analyzes of the carbonaceous deposits were performed.



(a)

(b)



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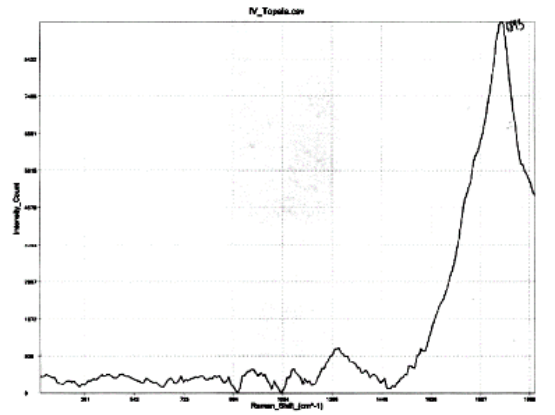

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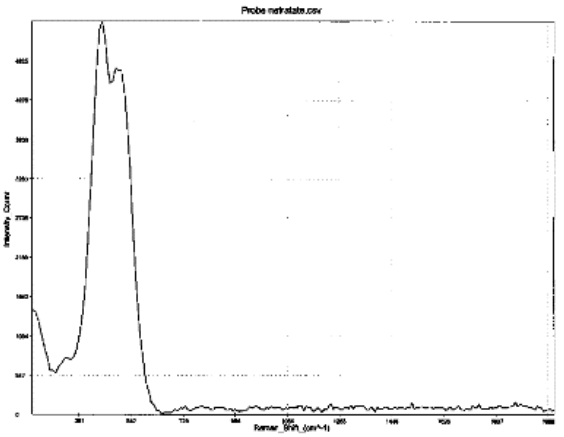
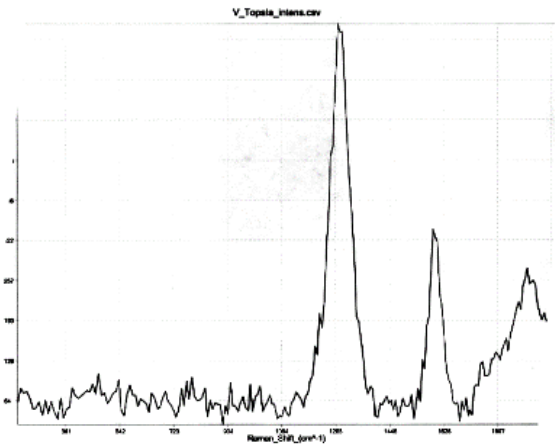
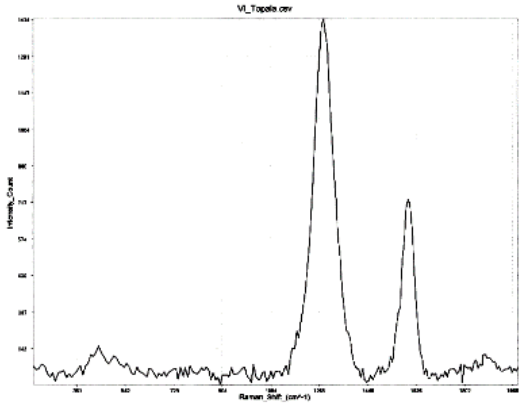


(e)



(f)



(g)

Fig. 8. RAMAN analysis of the film of graphite: (a) no. 1; (b) no. 2; (c) no. 3; (d) no. 4; (e) no. 5; (f) no. 6; (g) no. 7

If we consider that the deposits are graphite formations, then knowing the constancy of the crystal network and assuming that the formations break layer by layer and knowing the thickness of the deposit as the maximum value and Thus, if we compare the Raman bands of the untreated sample (see figure 8(e), sample no. 5 characteristic for graphite) with those shown in figure 8 (the sample marked with no. 1 - 4 and 6 - 7), then on these the corresponding maximums "1281, 1596 and 1899", "1289 and 1588", "1280 and 1583", " 1843", "1289 and 1583", "1283 and 1588", and these correspond to the structures characteristic of monoparental carbon nanotubes and other spatial formations.

XPS analysis of the surface in the case of the samples analyzed in the present study, were performed using a PHI 500 VersaProbe equipment (manufactured by PHI-ULVAC Inc. USA) which uses a hemispherical photoelectron kinetic energy analyzer (resolution 0.50 eV). The equipment uses a source of monochromatized X-rays (hυ=1486.7 eV) and analyzes electrons emitted under an angle of 45º to the surface (take-off angle). Thus, the typical probing depth was 5 nm. The analyzed surface of the samples was previously subjected to a bombardment with a flow of Ar+ ions with an energy of 1.5 keV, (3x1015 electrons/mm2), in order to reduce surface contaminants, [16]. XPS spectra (survey) were recorded in the entire range of bond energies of interest (0 – 750 eV), with a step of 0.50 eV, as well as high-resolution spectra of oxygen (O 1s orbital), carbon (C 1s), iron (2p1/2 and 2p3/2) and silicon (Si 2p and 2s). A typical survey spectrum of the investigated surfaces, presented in figure 8(f), reveals the presence of the above-mentioned elements, the elemental composition of the surface region being: carbon 60.7%, oxygen 22.9%, iron 6.2%, silicon 6.2% and nitrogen 2.7%, other elements 1.3%. The high value of the atomic concentration of carbon is noted. The deconvolution of the carbon peak showed that it is predominantly present (approx. 80%) as an element adsorbed on the surface (BE=285.3 eV) and to a lesser extent (approx. 20%), in C-O type structures (BE =286.6 eV), O-C=O (BE=288.9 eV). We present, as an example, the high-resolution XPS spectrum of oxygen (curve 1 in figure 8(e), in which, in the graph, some experimental points have been ignored, for reasons of visibility. The spectra were acquired using the PHI SUMMIT XPS software package, and their processing – using the PHI MultiPack 8.2C package, followed the standard procedure, described in detail in ref., [20, 21, 22], based on the reference data of the VersaProbe5000 equipment, [23].

The analysis of the O 1s signal revealed the presence of oxygen in 3 types of chemical bonds (called - hereafter - components). These are: (a) the O2- component (comprising the oxygen atoms involved in the oxides of the metallic elements in the sample and denoted by 3 in figure 8(f), with the specific binding energy of 529.6 eV; (b) the OH- component with the bond energy 531.5 eV (marked with 2 in figure 8 (f) the component due to O-C and O-C=O bonds (BE=533.4 eV marked with 4 in figure 8 (f).

The chemical analysis demonstrated that the concentration ratios of the 3 components C(a):C(b):C(c) are 0.89:1.00:0.50. Additional tests showed that there could also be a 4th component of oxygen, which enters the O-H bond, but its relative concentration does not exceed the value of 0.15.

An interesting phenomenon of mass addition revealed following the thermogravimetric analyzes performed on the material extracted from the graphite films deposited by electrical impulse discharges, determined the research team to propose and support a new series of analyses.

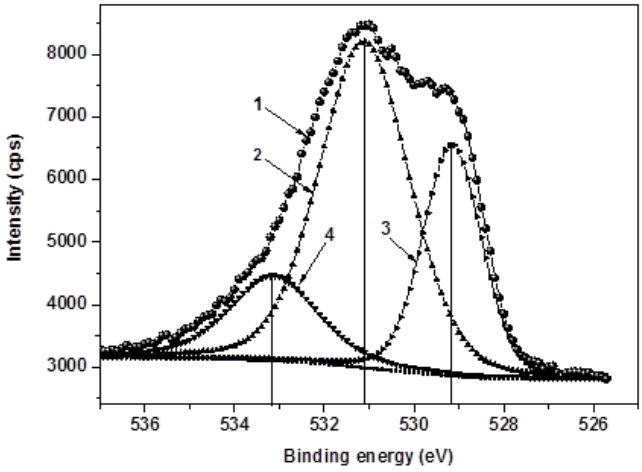


Fig. 9. The presence of oxygen in 3 types of chemical bonds, [16, 17]

Thus, the graphite film from another series of samples obtained under the same conditions was subjected to electron microscopy analysis through the SEM (Scanning electronic microscope) process. Thus, following the analyses, a series of particularly interesting images were obtained. The most interesting and relevant images were

obtained on the samples which also presented the most important percentages of water absorption and mass additions. The images were taken with the electron microscope Type Quanta FEI, supplied by Phillips.
The analysis of the morphology of the surface processed by pulse electric discharges with graphite "tool-electrodes" demonstrated that the physico-chemical changes on the surface do not exceed micrometric sizes. Apart from the initial components of the processed material, a considerable amount of carbon (about 80%) in atomic content is attested. The analysis of the microstructure of the transverse micro-grinds demonstrates that the vast majority of the carbon transferred to the surface of the part is found at depths of the order of micrometers, which allows us to conclude that it is possible to form the carbide and graphite phases separately at the interface of the metal part and the submission formed with the application of the DEI. If we are to analyze those presented in figure 10(b), we can see that the film consists of clusters of nanometric formations. In order to obtain a series of more conclusive images and to avoid electromagnetic interference, a series of samples of graphite films were made by electrical impulse discharges on the silicon chips used as anodes. In order not to excessively thicken the graphite layer and not to generate melting points, a single pulsed electric discharge positioned tangentially to the anode was used for each sample. The images were obtained at magnifications of 1000X – 20000X and are presented in figure 10, [24].

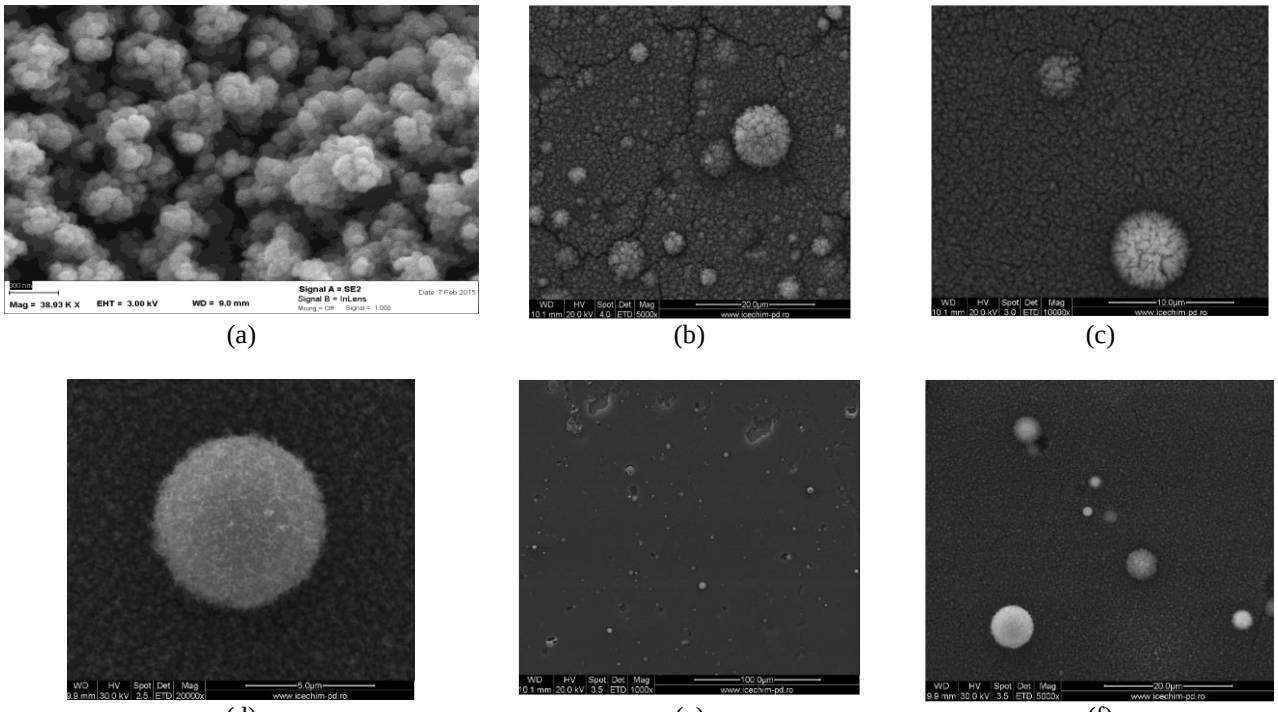


Fig. 10. SEM morphology of graphite film deposited on Steel 45 support, for different magnifications [16]: (a) X 300; (b) X 5000; (c) X 10000; (d) X 20000; (e) X 1000; (f) X 10000;

Further consulting the specialized literature, we identified images with spatial formations made of fullerene-type carbon atoms [14, 15]. These are shown in figure 11.

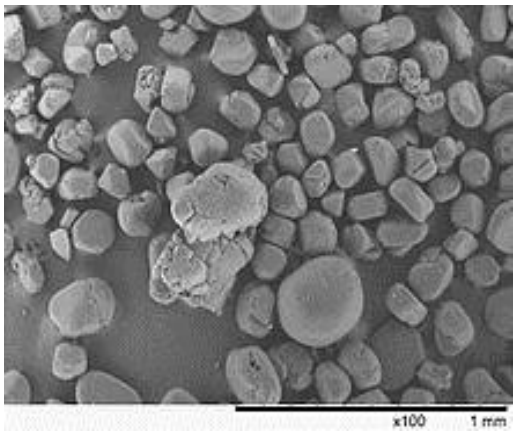


Fig. 11. Fullerene type spatial structures (SEM images), [14, 15]

These formations, shown in figure 11, are similar in shape, and the dimensions are comparable to those of the globular formations identified from the images taken of the graphite films.

4. CONCLUSION

This article presents the results of the research works of a complex working group made up of researchers and teachers from Romania and the Republic of Moldova. The team of researchers belong to the National Research and Development Institute for Chemistry and Petrochemistry ICECHIM Bucharest, the university professors belong to the Polytechnic University of Bucharest and the Alecu Russo State University in Balti. This particularly competent work group contributed equally to the realization of this research, which had as its object the elucidation of the technological aspects of the process of depositing graphite films through the process of electrical impulse discharges. The working regime was chosen in such a way that the electric discharge does not damage the metal surface of the anode. This working regime was named "underexcitation working regime". The graphite film was subjected to spectral analyzes - X-rays EDX, RAMAN as well as SEM electron microscopy analyzes to define its structure. following these analyses, especially of RAMAN spectroscopy and SEM electron microscopy, a series of spherical formations made up of carbon atoms of the fullerene type were identified. the discovery of these spatial formations made up of 60-100 carbon atoms that were obtained following the process of electrical impulse discharges in the underexcitation regime - even if it is a collateral result compared to the subject of the doctoral thesis, it also represents a scientific novelty for which the issuance of an invention patent was also requested. This patent was granted under Law 64/1991 and has the number 133558.

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