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# STRUCTURAL MODIFICATIONS – PROPERTIES OF SURFACE MICRO-STRATA WITH GRAPHITE DEPOSITIONS

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***Abstract:*** This paper presents the results of experimental investigations of the behavior of surface layers formed by graphite deposition under the action of electrical discharges in impulse (EDI). It is shown that the formed pellicle continuity constitutes at least 82%, the layer thickness does not exceed 7 μm, the diffusion depth is about 30 μm, the surface micro hardness increases by 2-8 times (for steels), the potential of corrosion decreases, the wear resistance increases by 3-4 times and the sticking effect during the functioning in mobile couplers at dry rubbing has not been attested. The use of graphite tool-electrode also improves the mechanical properties at the microscopic level, such as tensile strength. Test trials with the use of these three processing regimes have shown that the maximum tearing force for samples made of steel 3 coated with graphite increases compared to that of the raw sample.

***Key words:*** electric discharges in impulse, graphite pellicle, micro hardness, tensile strength, tearing force

## 1. INTRODUCTION

It is known from the studied literature that machinery pieces and assemblies wear out during the working process which leads to changing the geometric dimensions, shape and properties of surface layers and in some cases, cracks, curves, twisting, distortions and tearing appear. Currently, for thermal and thermo chemical treatment, for depositing resistant layers that prevent rapid wear of pieces both conventional and unconventional methods, among which we could mention thermal and thermo chemical traditional processing; processing via metallization; processing via plastic deformation; flame gas processing; processing via galvanizing; hardening via ion nitriding; via magnetic impulses; via laser radiation; via electrical discharges in impulse in the regime of electric contact and of sub excitation, etc may be used.

To annihilate the drawbacks mentioned in the paper we used the method of electric discharges in impulse in the regime of sub excitation. The deposition and hardening of the superficial layer via electrical discharges in impulse is based on the effect of electric erosion and on that of the tool electrode material polar transfer to the piece electrode (Mihaliuc, 2003; Bardac et al., 2005). Under the influence of the starting impulse a conductivity channel is formed through which, due to the application of the power impulse, the force discharge takes place accompanied by the formation of plasma channel that causes significant changes in the work piece surface, both macroscopically and microscopically, concerning the behavior of surface layers formed by graphite deposition influenced by electric discharges in impulse (EDI).

## 2. MATERIALS AND METHODS OF EXPERIMENTAL INVESTIGATIONS

The experimental investigations were done under ordinary conditions in the air medium, in the regime of sub excitation of EDI in which the piece or the electrode had the possibility to change its polarity.

Steel 3, copper M3 and cast were used as materials for pieces or samples subjected to processing. The tool electrode is a bar with a 2-3 mm diameter made of pyrolytic graphite. In this paper, we used two types of samples for microscopic study and for the study of traction samples. The samples used for the microscopic investigation of the surface layer made of steel, iron and copper have the form of a parallelepiped with the dimensions 10 × 10 × 3 mm, and the samples used for the study at traction have the form of a stepped bar the dimensions of which are shown in fig. 1. The tool - electrode made of graphite was connected in the discharge circuit in three ways: as cathode, anode or in a combined regime. We used the combined regime because, according to specialized literature and to our own experimental research (Beshliu, 2008; Topala and Beshliu, 2008), the tool-electrode made of graphite erodes more when it is a cathode than when it is an anode and as a result a bigger quantity of carbon is deposited on the piece surface which, due to the energy impulse, diffuses in the surface layer causing structural changes.

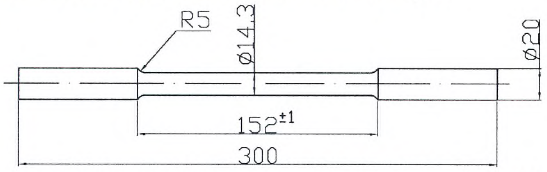



Fig. 1. Dimensions and form of samples for traction trial (tolerance of unindicated dimensions - 0,1mm)

When it is connected as anode, the deposits are smaller but the impulse power influences the modification of the superficial layer, the roughness decrease (Topala et al., 2006) and the oxidation of the processed surface takes place. When the combined regime of processing is used, as it was mentioned in some papers (Beshliu, 2008), due to the physico chemical processes that take place in the interstice and in the superficial layer of pieces, a more essential modification of hardness and durability of the pieces subjected to trial occurs than when monopolar electric discharges are used.

To do the experiments we used the supply source that possesses the following parameters: energy emitted in the interstice $W_S$=0 – 6 J, energy accumulated on the battery of condencers $W_c$= 0-12 J, voltage on the battery of condencers $U_c$=0-200 V, capacity C=100-600 μF with the step 100 μF, interstice S=0,05-2,5 mm, frequency of discharges f=0-50 Hz, impulse duration τ=0-250 μs. Due to these parameters that the supply source possesses we may ensure the functioning of EDI in the regime of „warm" electrode spots (with the melting of the processed surface) and in the regime of „cool" electrode spots (without the melting of the processed surface though at the level of nanometers this happens). To do this kind of experiments it is necessary to respect the following condition:

$$Q = \frac{4W_S}{\pi d_c^2 S} < Q_{melt} \qquad (1)$$

where $Q$ is the heat emitted on the surface of electrodes per volume unit, J/m$^3$; $W_S = \int_0^{\tau} u(t)i(t)dt$ is the energy emitted in the interstice, J; $u(t)$ is the voltage on the interstice at the discharge, V; $i(t)$ is the instantaneous value of the current in the interstice, A; $\tau$ is the duration of the discharge impulse, s; $d_c$ is the diameter of the plasma canal, m; $S$ is the distance between the electrodes (the interstice), m; $Q_{melt}=q_{melt}\cdot\rho_{melt}$ is the volumetric melting heat of the processed piece, J/m$^3$; $q_{melt}$ is the specific melting heat of the processed piece, J/kg; $\rho_{melt}$ is the material density at the temperature of melting, kg/m$^3$.

The scanning electron microscopy (SEM) was used to investigate the surface morphology; the photoelectron spectroscopy XPS was used to determine the chemical composition of the superficial layer.

## 3. THE RESULTS OF INVESTIGATION EXPERIMENTS AND THEIR ANALYSIS

As it has been mentioned above, the electric discharges in impulse were executed on samples made of copper, cast and steel. The parameters of electric discharges in impulse are selected depending on the parameters of current impulse generator, the value of the processed interstice and electrode polarity. When the processing is done with the tool-electrode as cathode on all sample surfaces there are carbon deposits which, due to the applied energy impulse crystalizes on the processed surface under the form of graphite pellicle and partially diffuses there causing structural and chemical changes. As experimental data show the graphite pellicle contains about 74-78% of carbon that confirms again the previously done experiments on cast and presented in the paper (Topala et al., 2010). The data concerning the chemical composition were achieved by XPS analyses and are presented in Table 1 where we can see that the graphite pellicles for samples made of steel 3 contain 73,8 % of carbon, for those made of copper - 77,5 % and for cast - 77,6%. As the processing takes place in the air, there is an accumulation of oxigen of about 17-19 % and respectively 3-4 % of nitrogen on the piece surface. These components allow the durification of the superficial layer, by about 2-8 times (Beshliu, 2008) in comparison with the basic material, through its diffusion inside it and the thermal treatment due to rapid cycling warming and cooling of the superficial layer.

Table 1. Chemical composition of the superficial layer

| No. | Sample material | C1s (%) | N1s (%) | O1s (%) | Fe2p (%) | Cu2p (%) |
|---|---|---|---|---|---|---|
| 1 | Steel 3 | 73.8 | 4.3 | 19.8 | 2.1 | |
| 2 | Copper M3 | 77.5 | 4 | 17 | | 1.5 |
| 3 | Cast | 77.6 | 2.7 | 17.5 | 2.1 | |

Having removed one layer of carbon from the surface we again investigated the chemical composition of the steel sample surface (Table 2). This time the quantity of carbon has decreased and constitutes 57%. This confirms the idea that a great deal of carbon has not only deposited on the surface but has also diffused in the superficial layer of the sample thus modifying the crystalline net.

Table 2. Chemical composition of the steel sample surface

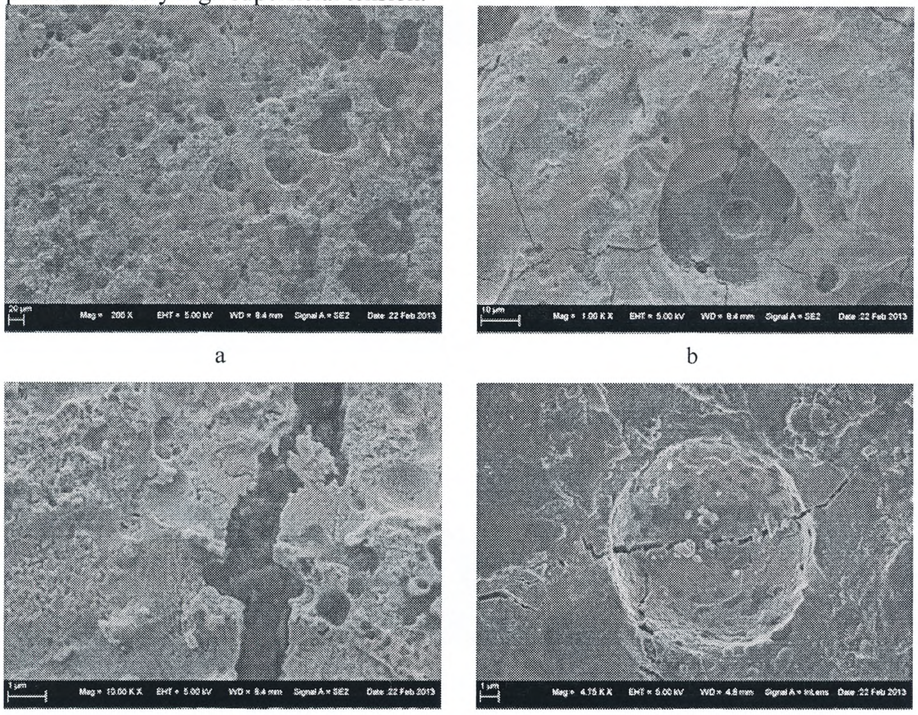
| Sample material | C1s (%) | N1s (%) | O1s (%) | Fe2p (%) |
|---|---|---|---|---|
| Steel 3 | 57 | 1.8 | 37 | 4.2 |

The analysis of the sample superficial layer morphology was done alongside with the study of the chemical composition. From the analysis of the obtained figures we stated that the graphite pellicle is not ideally continuous as it has cracks, hollows and craters. The cracks on the surface, as can be seen in Figure 2, c, d, are up to 1 μm wide. It probably happens due to the very high temperatures during the surface processing. This, however, cannot influence the exploitation properties of the piece under conditions of glass moulding. Semiliquid glass possesses a very high superficial tension.

Simultaneously the cracks play a positive role for depositing, creating possibilities to diminish internal tensions, thus removing the danger of the deposit self distruction.

Craters of irregular forms also appear; we have an example in Fig. 2, b inside which there is a crater that is almost identical with the form of a spherical calotte whose diameter is approximately 13 μm. The formation of diamond-type structures is possible in such craters which contain carbon.

The basis of this hypothesis is zonal heating at $10^4$ K temperatures (graphite melting is possible), high pressures caused by this and sudden cooling based on increased thermal conductivity of the processed metal surface.



a b c d

Fig. 2. Morphology of the surface layer after the application of electric discharges in impulse for the steel 3 sample

The experimental investigations done on cast (Fig. 3) prove that carbon possesses the ability to diffuse in the superficial layer at a depth of approximately 3-7 μm (Topala et al., 2010; Topala et al., 2011). The application of this method in industry, namely in the functioning in the technological cycle of glass moulding form poansons, the latter being processed via electric discharges in impulse, may increase their durability (Topala et al., 2011) compared to the unprocessed ones as the formed graphite pellicle serves as a solid ointment that decreases the sticking of glass to the active side of the piece and the presence of oxigen may offer the piece a higher level of resistance to wearing out due to the formation of oxides. The paper (Krasilinikov et al., 2009) demonstrates that during depositing via electric discharges in impulse in the electric contact using the tool – electrode made of metal carbide T15K6 alongside with the increase of the superficial layer microdurity the limit of resistance to breaking of the processed piece (417MPa) compared to the unprocessed one (526MPa) decreases. When two layers of deposits of metal carbide and graphite are used, the limit of resistance increases a little compared to the deposit of one depositing layer. Based on these considerations, it is logical to suppose that the graphite cover, when the superficial layer microdurity is increased, does not negatively influence the resistance limit of the piece. Thus the investigation is futher devoted to determining the influence of deposits via electric discharges in impulse with the graphite electrode on the resistance limit value to piece breaking and to determining the dependence of the resistance limit value to piece breaking compared to the parameters of the processing regime.

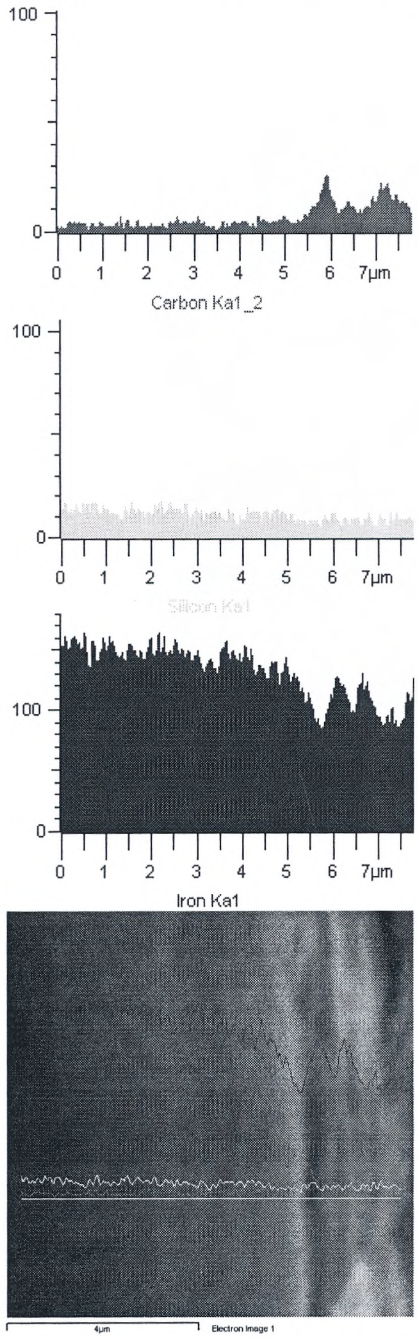



Fig. 3. Distribution of elements in the superficial layers

Three processing regimes were investigated. At regime No1, the graphite tool-electrode is a cathode, the latter eroding strongly, its material depositing on the piece surface and forming a graphite pellicle as it has been mentioned before. At regime No2, the graphite tool-electrode is ande and it erodes just a little while on its surface, processes of diffusion, thermal and thermo chemical, occur. Regime No3 presents a combination of the first twp processing regimes, namely, at first, the sample surface is processed in regime No1; this is followed by superposition with impulses characteristic of regime No2. Five pieces were processed in each regime.The parameters of the processing regimes are: the voltage at the clema of the condencer battery ($U_c$); the capacity of the condencer battery (C); the frequency of electric discharges (f); discharge time ($\tau$); the interstice between electrodes (S); the speed of tool displacement compared to the piece (V); the tool and piece polarity are indicated in Table 3.

Table 3. Parameters of the processing regime

| Processing regime | | $U_c$, V | C, µF | f, Hz | $\tau$, µs | S, mm | V, mm/s | Polarity | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | Tool | Piece |
| 1 | | 200 | 600 | 3 | 220 | 1 | 0,455 | – | + |
| 2 | | 180 | 600 | 3 | 220 | 0,9 | 0,455 | + | – |
| 3 | 3.1 | 200 | 600 | 3 | 220 | 1 | 0,455 | – | + |
| | 3.2 | 180 | 600 | 3 | 220 | 0,9 | 0,455 | + | – |

The processed and unprocessed pieces (Table 4) were subjected to traction trials.

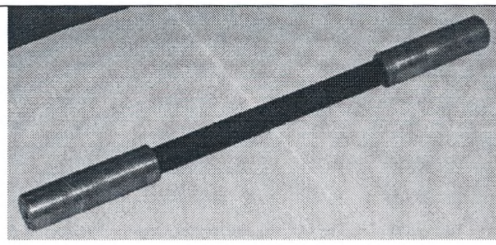
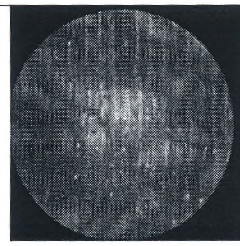
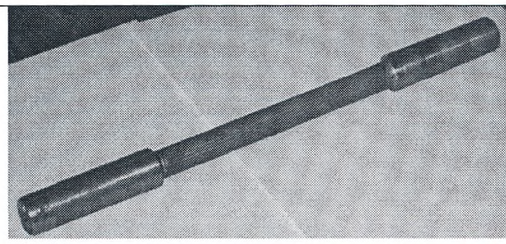
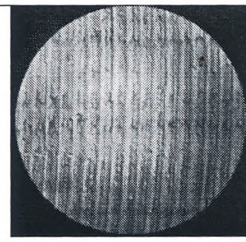
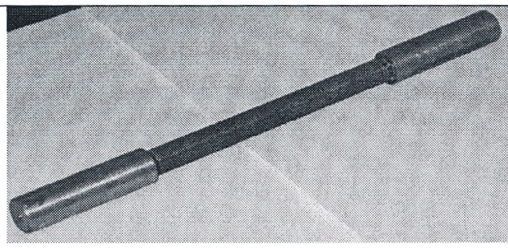
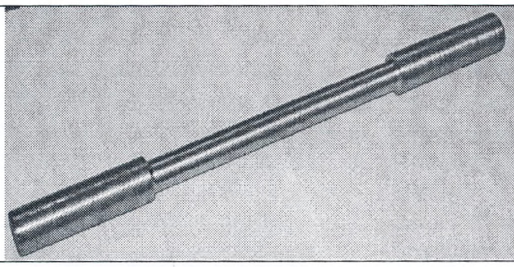
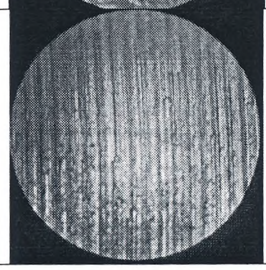
Having measured the pieces subjected to traction, we got the following parameters: initial piece diameter ($d_o$); initial piece length ($l_o$); highest breaking force ($F_{max}$); piece final diameter (of the narrowest part of the tool) ($d_f$); final length of the piece ($l_f$); initial area of the cross section ($A_o$) and the final one ($A_f$); resistance limit to breaking ($\sigma_r$); absolute elongation ($\Delta l$); relative elongation ($\delta$); absolute narrowing ($\Delta A$); relative narrowing($\psi$). The values of these parameters are presented in Table 5.

The histograms on Fig. 4 and Fig. 5 illustrate the comparison between the medium values of the highest breaking force and the resistance limit to piece breaking in different regimes of processing.

The presented analysis shows that the maximum breaking force of processed pieces in all regimes is 50 kgF higher than in those that were not processed. Respectively, the resistance limit to breaking ($\sigma_r$) of processed pieces in all regimes is 3 MPa higher than that of unprocessed pieces. It has been experimentally demonstrated that the maximum breaking force ($F_{max}$) and the resistance limit to breaking ($\sigma_r$) of processed pieces are equal for all used processing regimes.

The absolute elongation ($\Delta l$) is (1-3)mm smaller and the relative elongation of pieces ($\delta$) is (1.53–0.91)% smaller.

Table 4. Overview of samples according to the processing regimes

| Piece | Overview | Increased by 32 times |
|---|---|---|
| Regime 1 |  |  |
| Regime 2 |  |  |
| Regime 3 |  |  |
| Not processed |  |  |

These effects may be explained by the fact that considerable modification occurs at 5-7 μm depth and it cannot considerably modify the piece properties along the whole section; however relaxation phenomena, due to the cumulative process of heat accumulation in a considerable value of the piece material, also occur in the material (thousands of electric discharges in impulse occur on a relatively small surface). The sample processing was performed on necks with a relatively small diameter (d=14.3mm), because of which the processes of cooling worsen and transformations are produced in the material. Though there is considerable growth of hardening (Beshliu, 2008; Topala et al., 2006) microscopically the processed piece does not become fragile, on the contrary, we may notice a small growth of the resistance limit to breaking and, as it has been demonstrated, the resistance limit value to breaking depends little on the parameters of the used processing regime; it fully depends on the covering material and, probably, on the working medium. The newly formed layer (the white layer, the zone of diffusion and that of thermal influence) possesses special properties, hardness and high resistance to breaking though its thickness is at the level of microns; it is able to positively modify the resistance limit to piece breaking, in our case the diameter of the piece is 14.3mm. Probably much more positive results could be obtained for pieces of smaller dimensions; this is to become the investigating domain to be studied in the nearest future. These results could be used to obtain layers from durable materials in which the graphite pellicle would play the role of substratum that would allow to get a relatively higher resistance to breaking.

Table 5. The results of traction trial

| Regime of processing | №1 | №2 | №3 | Without processing |
|---|---|---|---|---|
| $d_o$, mm | 14,3 | 14,3 | 14,3 | 14,3 |
| $A_o$, $mm^2$ | 160,61 | 160,61 | 160,61 | 160,61 |
| $l_o$, mm | 152,5 | 153 | 153 | 151 |
| $F_{max}$, kgF | 8100 | 8100 | 8100 | 8050 |
| $d_f$, mm | 8,7 | 8,7 | 8,8 | 8,8 |
| $A_f$, $mm^2$ | 59,45 | 59,45 | 60,82 | 60,82 |
| $l_f$, mm | 190 | 191 | 190,5 | 190 |
| $\sigma_r$, MPa | 494,25 | 494,25 | 494,25 | 491,20 |
| $\Delta l$, mm | 37,5 | 38 | 37,5 | 39 |
| $\delta_{med}$, % | 24,30 | 24,92 | 24,55 | 25,83 |
| $\Delta A$, $mm^2$ | 101,16 | 101,16 | 99,79 | 99,79 |
| $\psi_{med}$, % | 62,56 | 62,56 | 62,56 | 62,56 |

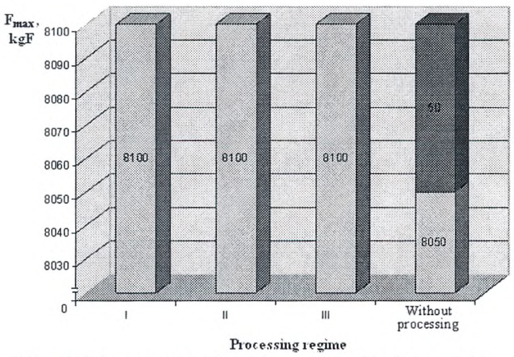



Fig. 4. The comparative histogram of values of the breaking highest force ($F_{max}$) in different processing regimes

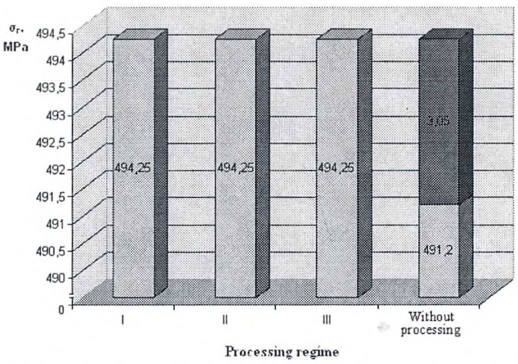



Fig. 5. The comparative histogram of values of resistance limits to breaking ($\sigma_r$) in different processing regimes

## 4. CONCLUSIONS

Using the tool-electrode made of graphite with the cathode polarity we can obtain graphite pellicles on such materials as steel, cast and copper with a content of about 74-78% carbon.

The diffusion depth of carbon in the superficial layer constitutes about 3-7 μm that positively influences the properties of the pieces subjected to processing.

The trial for breaking of samples made of steel demonstrates that the graphite pellicle deposited at different polarities increases the maximum breaking force with 50 kgF and the resistance to breaking with 3MPa; this makes it possible to use the graphite pellicle in depositing polycomponental materials.

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