

OXIDATION OF TAYLOR CONE-SHAPED ASPERITIES BY APPLICATION OF PLASMA IN NORMAL CONDITION

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ABSTRACT: This paper refers on experimental investigations of the conductive surface treatment by applying electric discharges in impulse. We have particularly focused on the modifications of metal surface micro-geometry that leads to formation of Taylor cone shaped asperities. We have established and presented the best energetic conditions of extracting the latter from cylindrical and flat pieces made of W+10 % Re by electrical discharges in impulse. The process of extraction of cones was followed by formation of oxide nano-films. **Next** research is oriented on chemical analysis of oxide nano-films on the Taylor cone surfaces.

KEY WORDS: electrode spots, Taylor cones, chemical analysis, nano-oxide, plasma

1. INTRODUCTION

The modern stage of scientific and technical progress is characterized by a transit from macro- to micro- and nano-organization of matter. Obtained samples were studied by optical microscopy (XIM600 Optical Microscope) and SEM (Vega

TESCAN 5130 and QUANTA 200 FEI Fillips). In result, we observed that the Taylor cones are formed not only in the centre of the crater, but also at its peripheries, due to the development of electrohydrodynamic instability on the melted tungsten surface as a result of interaction between plasma channels and substance (Figure 1) [7, 9].

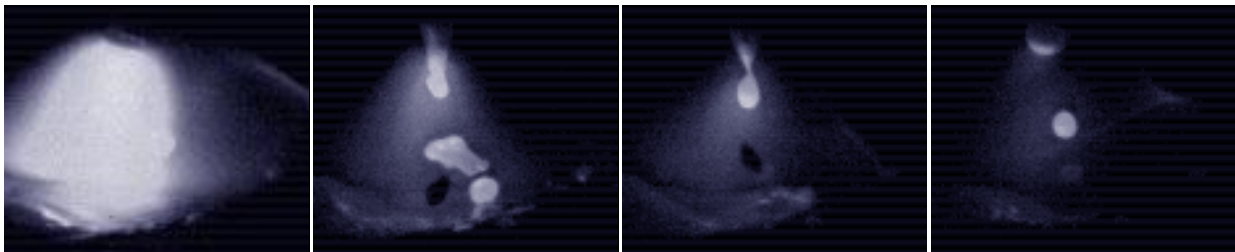


Figure 1. Interaction between plasma channel and the substance example

Varying the interstice size, it is possible to obtain almost any degree of oxidation of the working surfaces due to modification of the emission energy. Increasing the interstice size, we may obtain such states when the heating of the volume of the treated material is not essential and geometry of the surfaces does not change. This possibility may be applied in the development of new surface treating technologies by applying electric discharges in impulse.

Increasing of the active surface changes the character of interaction of surface with environment and with other active surfaces brought in contact.

The process of modification of metal surface micro-geometry was lead in normal conditions (air environment, room temperature, normal humidity)

and is followed by modification of chemical composition of examined surfaces and oxide nano-film formation.

2. METHODOLOGY OF EXPERIMENTAL INVESTIGATIONS

To obtain cone asperities, the tungsten surfaces were treated by electrical discharges in impulse by technology described in [4, 8]. For cone extraction pairs of cylindrical tungsten-rhenium electrodes were used. Their positioning is presented in Figure 2. The formation of conic asperities in the centre of craters is due to the perturbation of the liquid metal surface influenced by the electric fields of high strength, the force of superficial tension of the melted metal and less by the component of weight force [1, 2, 3].

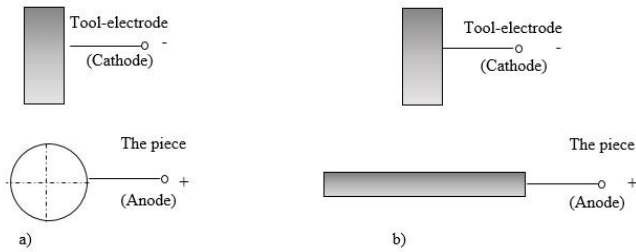


Figure 2. Schemes of electrode positioning used in the experimental investigations and their connection in the circuit of power impulse generator discharge:
 a) the case of cylindrical piece; b) the case of flat piece

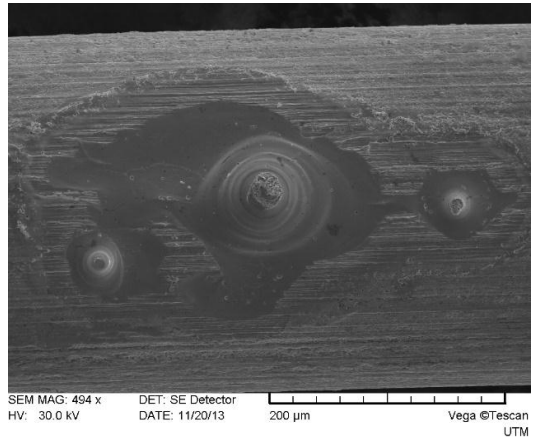


Figure 3. Taylor cone extraction on the main drop and on the drops flown away

Taylor cones are formed not only in the centre of the crater, but also at its peripheries, due to the development of electro-hydrodynamic instability on the melted tungsten surface, as a result of interaction of the plasma channels on the substance (Figure 3).

The aspects presented in Figure 3 confirm the fact that applying higher energy of the electrical discharge in impulse, the structure of the formed plasma jet in the gap is non-homogenous and poly-channel, and it is possible the simultaneous formation of more asperities of different sizes during a solitary discharge. Directed configuration of the plasma jet in the gap leads to the directed formation of asperities by shape, size and orientation direction.

It was practically stated that efficiency of cone forming on metal surfaces is significantly greater when piece is connected as anode [8]. It happens because in case of short electric discharges the strongest emission of energy occurs on the anode (Figure 4 left) for the same value of electric current that passes the interstice in a solitary electric discharge. In the same time decrease of cathode voltage is lower (as an absolute value) than the anodic one and, as a result, it cools faster and doesn't form the cone (Figure 4 right).

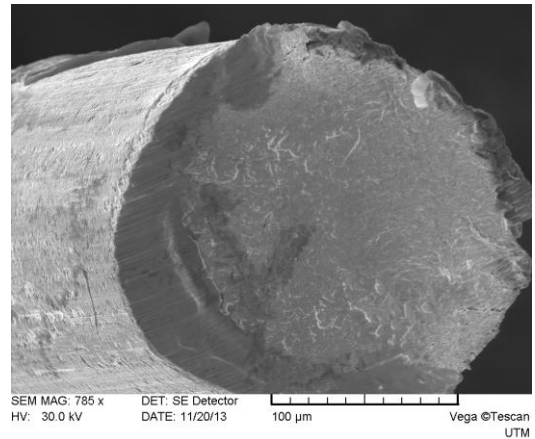
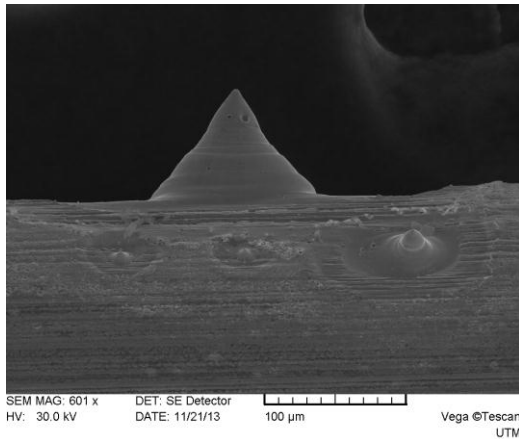


Figure 4. SEM images of tool-electrodes made of W+Re 10 %: left - the tool-electrode connected as anode; right - the tool-electrode connected as cathode

Also it was demonstrated that insufficient current density restrains formation of cone asperity with top angle near 90° .

In the case when the electric field intensity created by the electrode spots is sufficient to overcome the force of gravity and those of surface tension of the molten metal, the conical asperity is formed and frozen on the machined surface (Figure 5 left), but when the quantity of molten metal is high and the duration of electrode spot interaction with the sample surface is small, then there we observe a surface deformation with formation on it of an asperity with surface bordered by a spherical wavy

calotte (Figure 5 right). The last type of asperities may be of practical interest in the active surfaces geometry modifying to increase the active area with increasing the absorption capacity of different types of radiation, or to enhance the chemical reactions in specific processes of modern technologies. At the same time, the asperity shown in Figure 5 right shows an intermediate phase of transition from the initial surface to the conical asperity, and to further development of the technology of formation of these types of asperities it is necessary to establish the necessary and sufficient physical and technological conditions by theoretical analysis and the additional experimental research.

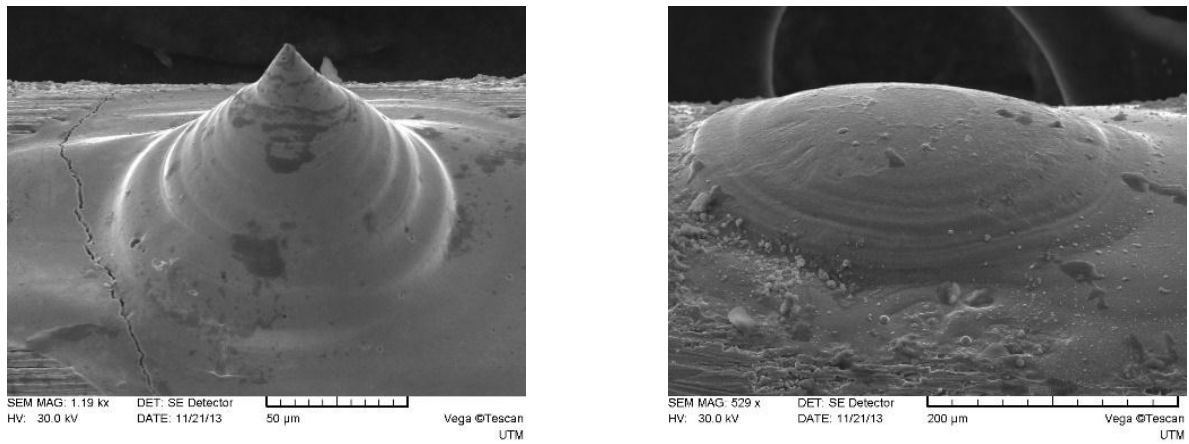


Figure 5. SEM images of Taylor cones that show growth of diameter of crater and of the base angle on reduction of current density. Power regimes: left - $U=60$ V, $C=200$ μ F, $d=0.2$ mm, $S=0.2$ mm, right - $U=70$ V, $C=200$ μ F, $d=0.25$ mm, $sc=0.2$ mm

It was observed that success of cone extraction and its height depend of the following conditions: discharge energy and current density, duration of discharge, material used in quality of electrode, strength of magnetic field applied on the interstice [6]. EDX analysis showed that all the cones after extraction are covered by oxide film with thickness of nanometers. Results of EDX investigations will be presented in the next chapter.

Behavior of the asperities formed on the active piece surfaces under the maintenance is determined by their size, the active area which they increase in all cases regardless of the geometric shape that they

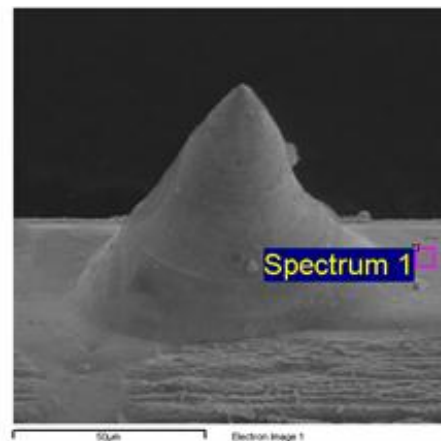
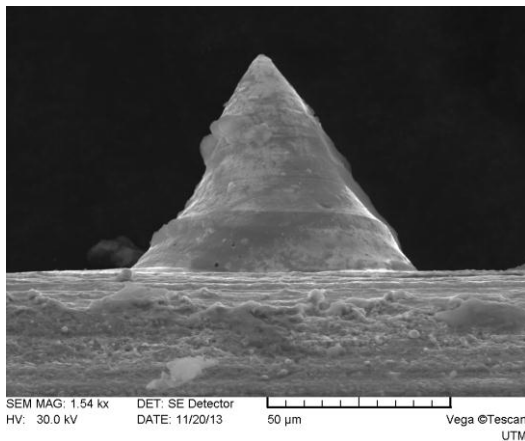


Figure 6. The same Taylor cone before (left) and after (right) application of discharge with increased electrode distance

as anode and once treated by EDI, samples connected as anode and twice treated by EDI.

The oxide patterns can act as robust masks for dry or wet etching and allow to realize electronic nanodevices.

Clean electrode contains from 0 % up to 27.9 at% of oxygen (Figure 7).

The oxidation was found to proceed through the motion of both oxygen and metal ions and/or their vacancies under the electric field applied across the oxide [10].

3. EXPERIMENTAL RESULTS

Anode once treated by EDI (Figure 8) shows increase of oxygen concentration from the bottom moving to top of the cone. We assume that while cone is being extracted, congelation starts from crater and drop is remaining liquid on the top. This liquid drop contacts with air maximum period of time comparatively to others regions of the cone. This fact determines maximum concentration of oxygen on the top of cone.

have, and also by the chemical composition of the surface layer that separates it from environment.

For these reasons, in the present research we investigated the chemical composition of the asperities surface in different areas depending on the gap size and the way of vertical positioning (top or bottom) of the surface of the sample (Figure 6).

Increasing the interstice size, we may obtain such states when the heating of material volume is not essential and the geometry of the sample surfaces does not change. However it results in deoxidation of treated surfaces.

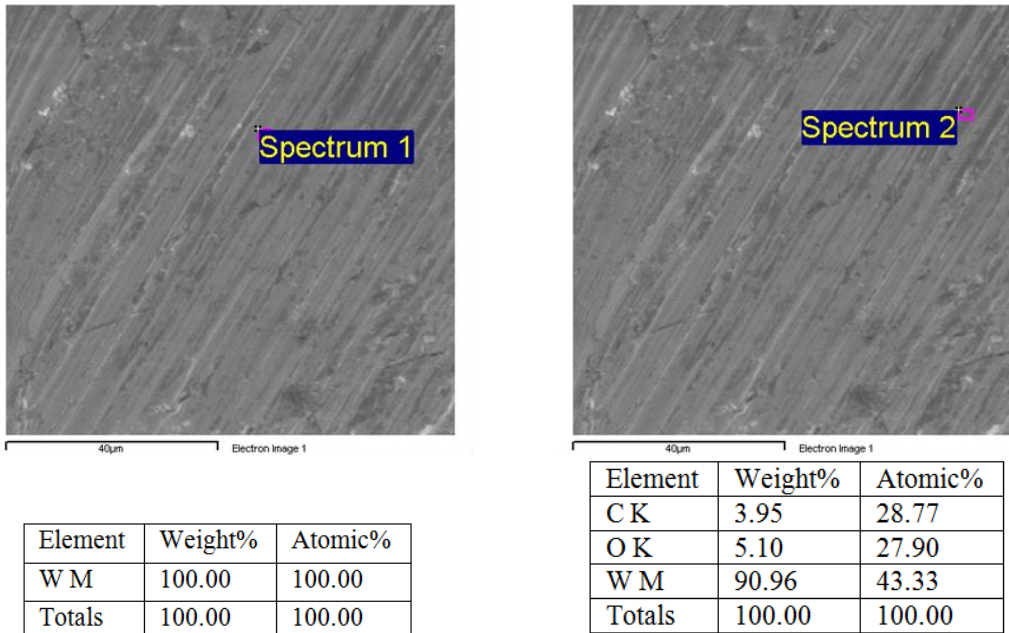
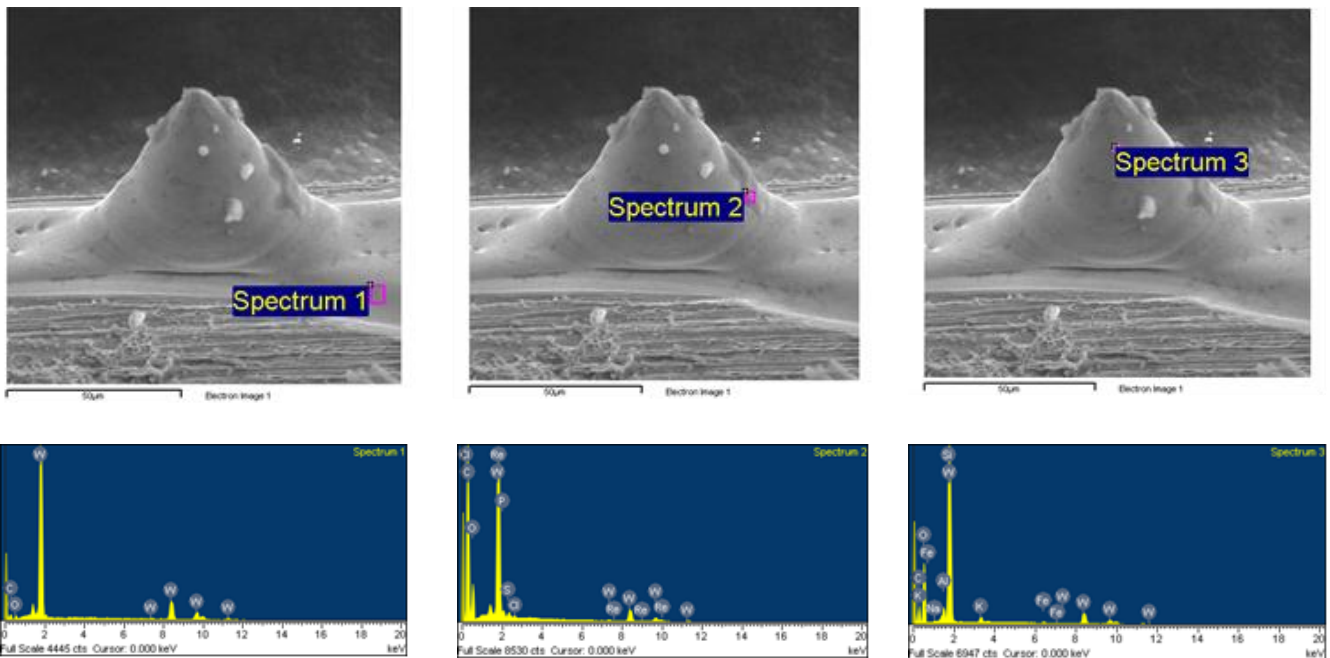


Figure 7. Results of EDX investigation of unprocessed plane sample made of W+10 % Re



h	at%	w%
1	49,79	32,42
2	16,54	14,24
3	17,28	3,62

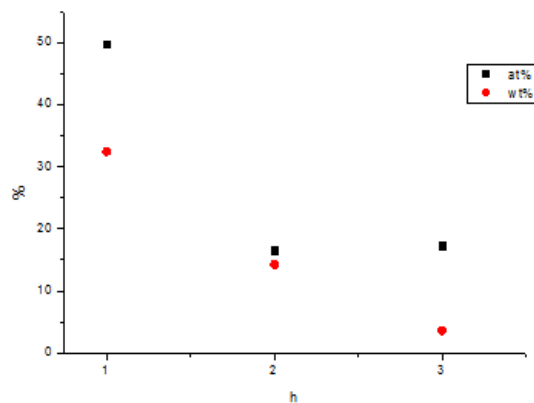


Figure 8. Results of EDX investigation of Taylor cones extracted on anode sample with power regime: $U=60V$, $C=200\mu F$, $d=0,2mm$, $S=0.2 mm$, where h1 – top, h2 – lateral surface, h3 – crater. On the graph, it is shown atomic and weight oxygen concentration in different zones of the cone

Anode twice treated by EDI (Figure 9), for the first time with interstice size 0.2 mm and the second - with increased interstice to 2 mm. And here there was observed decrease of oxygen concentration from bottom to top. The second discharge has by 10 times smaller energy. In this case, cone had been extracted and the plasma jet was focusing on the top

of it. Therefore, the second discharge heated just top region and only there have changed oxygen concentration. Now material hasn't liquid phase and doesn't contact with air, plasma jet isolates top region from the ambient and evaporates oxygen from surface.

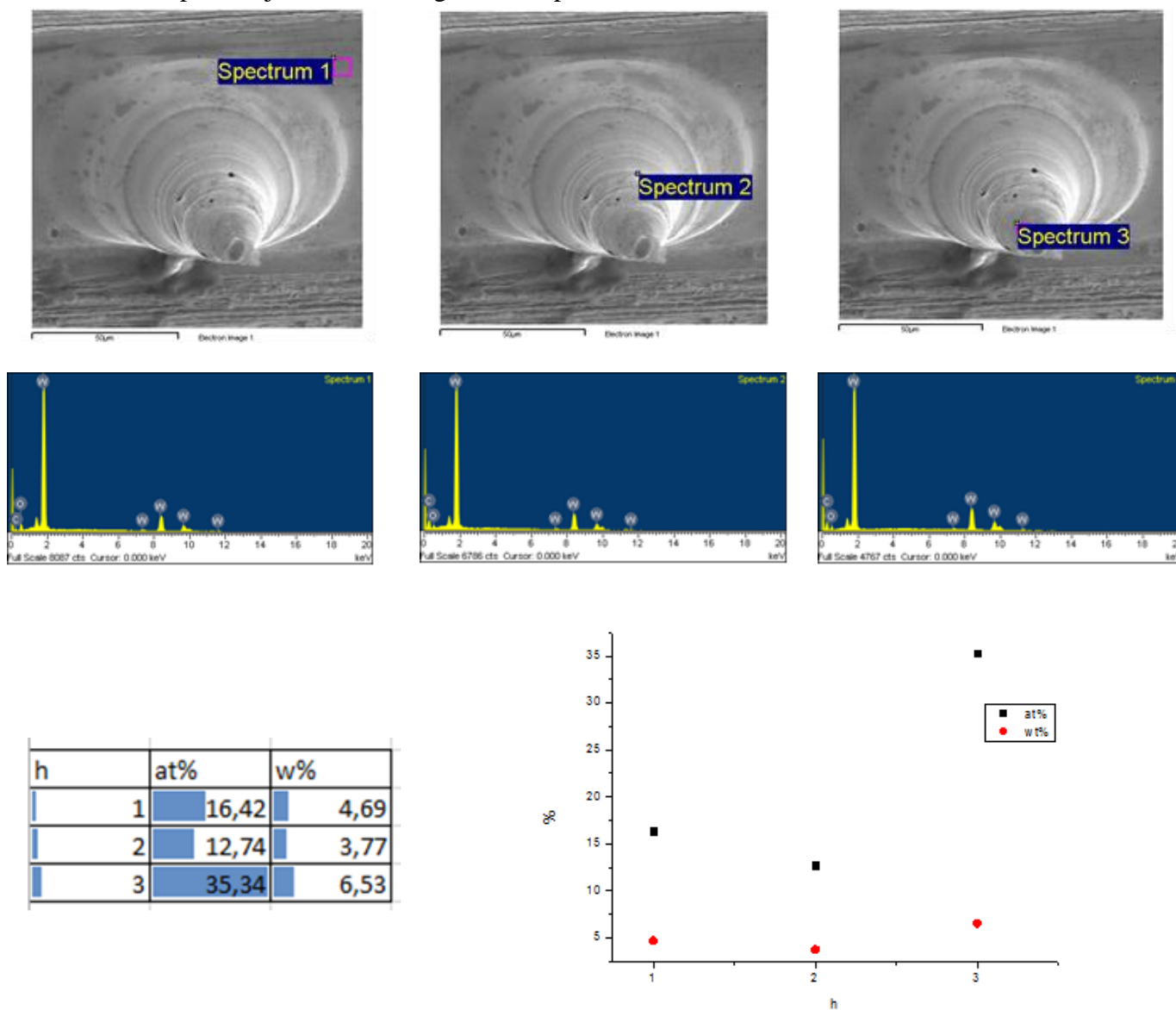


Figure 9. Results of EDX investigation of Taylor cones extracted on anode sample with power regime: $U=60$ V, $C=200$ μ F, $d=0.2$ mm, $S=0,2$ mm for the first discharge and $S=2$ mm for the second discharge, where $h1$ – top, $h2$ – lateral surface, $h3$ – crater.

On the graph it is shown atomic and weight oxygen concentration in different zones of the cone

Depending on the working environment in the interstice in the asperity surface, we can synthesize phases containing oxygen, nitrogen, hydrogen and carbon, which will modify the functional properties of cathodes applied at electronic thermal emission [9].

4. CONCLUSIONS

- The amount of oxygen dissolved in the surface layer of the metal pieces under the action of the plasma channel of electric discharges in impulse depends on the chemical content of the used alloy of sample and of the processing power regime.

- The influence of electric discharges in impulse on the micro geometry of treated surface is stronger when it is connected in the discharge circuit as anode.
- Cones extracted on anodes by EDI at start are covered with oxide nano-films. Thickness of oxide films on anode samples is less than on cathode samples.
- At EDI treatment of micro objects, plasma jet can as oxidize such as deoxidize metal surfaces.

5. ACKNOWLEDGEMENTS

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6. REFERENCES

1. Grigoriev, A. I., Shiriaeva, S. O., Belonjko, D. F., Klimov, A. V., *On the form of the Taylor cone and the characteristic time of its growth*, ZOM, no. 4, Kishinev, pp. 34-40, (2004).
2. Stavitskii, I. V., *High precision spark material processing. Scientific basis of precise materials of surface forming*, ZOM, no. 6, Kishinev, pp. 5-32, (2001).
3. Gabovich, M. D., *Liquid metal ion emitters*, Success of physical science. Vol. 140, no. 1, pp. 137-151, (1983).
4. Topala, P., Olaru, I., Rusnac, V., *New sequences on the physical picture of electrical erosion*, Collection of scientific papers, The Modern Technologies, Quality, Restructuring, Vol. 2, Kishinev, (2005).
5. Topala, P., Stoicev, P., *Processing technologies of conductive materials by electrical discharges in impulse*, Edition „Tehnica-INFO”, Kishinev, 265 p., (2008).
6. Topala, P., Dushenko, V., Gitlevichi, A., *On the conditions of melt formation on the surface of the cathode-piece during electro-spark alloying on installations of Razread type*, Kishinev, „Electronic processing of materials”, no. 6, pp.17-18, (1990).
7. Luban, R. B., Pekker, L. S., Galinov, I. V., *On possible mechanism of transfer of the material from the anode to the cathode on electro-spark doping of metals*. Electron spark discharge treatment of materials, no. 5, pp. 13-14, (1990).
8. Topala, P., Guzman, D., Rusnac, V., *Tehnologii de formare a suprafețelor pentru emisie electronică*, Vol. 38, Simpozion Științific Internațional, Chișinău, pp. 196-201, (2013).
9. Topală, Pavel. *Electrical charges as measure for removed metal mass the electrical discharge machining*, Iasi, Editura PIM, Nonconventional Tehnologies Review, no. 4. pp. 103-108, (2007).
10. Vu, Quoc Ho, Takuo, Sugano, *Selective Anodic Oxidation of Silicon in Oxygen Plasma*, IEEE journal of solid-state circuits, vol. sc-15, no. 4, pp. 501-508, (1980).