The application of the modified Blumlein scheme for electrodischarge obtainment of nanopowders in liquids

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Abstract. The formation of metal-carbon structures in electro-impulse discharge is investigated. The method of decrease of discharge voltage is proposed.

1. Introduction
Electro-impulse technology allows to obtain nanoparticles of the electrode material at low-powered electric impulse discharges in water, gasoline, ethanol, liquid nitrogen. Discharge impulse method has some essential advantages: cleanliness and purity, isomerism and preservation of crystal planes of the resulting product that enable to select the material effectively and to obtain new surface, physical and chemical and technological properties.

2. Experimental set-up
This work considers the electro-impulse method based on the so-called Blumlein scheme but modified to some extent and electrodes with the geometric pattern “needle-plane”. Such geometric configuration apparently seems to be the most appropriate for voltage decrease at the discharge gap in connection with the local electric field enhancement near the needle. The effective mechanism to obtain powder materials in such discharge is the impulse plasma in the discharge gap, the formation of which is accompanied by strong heating of the discharge channel and by hydraulic impact.
In this scheme electric capacity C1 is charged from high-voltage transformer Tr through ohmic resistance of the discharge gap between electrodes 3,4 which results in voltage increase on commutator P. When the critical voltage on commutator P is reached, capacity C1 turns out to be connected to the discharge gap and leads to plasma ignition.

3. Results and Discussion

The obtained powders were examined by X-ray analysis and scanning microscopy. The method of ACM-analysis is described below. First of all, the series of surface images of the samples in the area ≈ 3 μm / 3 μm were obtained. Then the most perspective sample of the smaller size was chosen out of them. It was scanned at a higher resolution once again. After that the spatial resolution of the microscope was increased and the images were obtained in the area ≈ 1 nm / 1 nm. Next, the analysis of the obtained images including the estimation of the sizes of the detected fractions, the statistical data processing and comparison of the given results with the results of other types of microscopic researches.

In all images not only separate particles but also multiple groups are easily determined. The smallest particles have the sizes about 150 nm, height 20 nm. The average size of particles is 200-300 nm, height 40-50 nm. In figure 2 the profile of a typical particles is given, its dimensions are in accordance with the average ones.

It should be mentioned that one can notice bigger particles as well. As a rule, they have the stretched forms. But the thorough analysis of this profile showed that it is multiple groups of smaller particles. Such groups of particles make determination of size distribution of particles more difficult. But the analysis of the images revealed that this distribution will not be significantly wide. Almost all particles have the sizes of the same order – hundreds of nanometers. In the process of electrode erosion not only the molybdenum plate but also the copper electrode was destructed to the same extent.

X-ray diagrams were registered from two areas of the sample: the area having a porous black layer on its surface and the light area. The corresponding x-ray diagrams are presented on figure 3.
According to figure 3 one can see that both intensive and weak reflections are observed in x-ray diagrams. The major difference of x-ray diagrams is noticed in the area of weak reflections. Quality phase analysis was done by the way of comparison of experimental x-ray diagrams with the x-ray diagrams theoretically modeled on the basis of data on the atomic structure of molybdenum, molybdenum carbides of different compositions and carbon allotropic phases presented in the base of crystallographic characteristics of inorganic compounds ICSD.

It was found out that strong lines are the reflection of (002) graphite and lines (200), (211), (220) of molybdenum. The further analysis of diffraction lines was done by Ritweld method. According to figure 4 the result of two stages of calculation of x-ray diagram is presented.

At the first stage only the molybdenum phase with volume-centered cubic lattice (space group of symmetry Im$ar{3}$m) was taken into account. Profile factor weight of unreliability R$_{wp}$ is 43% (according to 4 (a)). Having taken into consideration the contribution made by dissipation of hexagonal graphite
(space group of symmetry P6\textsubscript{3}mc), profile factor weight of unreliability R\textsubscript{wp} decreases to 17.5% (according to figure 4(b)).

According to figure 5, all lines of the experimental x-ray diagram are described by the x-ray diagram of molybdenum substrate and the layer of hexagonal graphite on it 83: 17. In the process of calculation all profile characteristics of x-ray diagrams of both phases were defined more precisely. Values of periods of elementary cell correspond to literature data: for Mo a=3.147(1); for graphite a=2.462(1), b=6.727(1) Å.

![Figure 5. Structure of powder](image)

The studies of formation of metal-carbon structures in electric impulse discharge by X-ray analysis have been conducted. It was stated that strong lines are the reflection of graphite and lines are the reflection of molybdenum.

The analysis of diffraction patterns showed that carbide film is formed through the reaction with carbon on the molybdenum surface.

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