Influence of parameters of high-voltage electrospark dispersion of materials on granulometric composition of nanopowders

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Abstract. The influence of parameters of high-voltage electric spark dispersion of materials on the granulometric composition of nanopowders of metals and alloys is considered. The most interesting feature of the developed experimental setup is the presence of an air discharge gap (FV1), which creates an overvoltage on the working discharge gap (FV2). The discharge gaps form a voltage divider when the capacitor (C) is discharged. It was assumed that the voltage divider should have a significant impact on the operation of the experimental setup. Studies have shown that when the air gap (FV1) is much larger than the working gap (FV2), their exact ratios do not affect the size of the resulting nanoparticles. At such ratios of discharge gaps, the voltage divider allows to regulate only the performance of the process of obtaining nanopowders. The resulting silver nanopowders had a spherical shape and a rather narrow size distribution. It is necessary to further study the process of high-voltage spark dispersion of materials with a significant overvoltage of the discharge gap.

1. Introduction
Powder materials of nanometer dimensions use in the manufacture of electronic technics, instrument making and additive technologies. One of the available methods of their production in practice is the electric spark dispersion of materials [1]. However, the production of powders of submicron and nanometer sizes by the method of electrospark erosion has one noticeable drawback. The resulting particles have a significant range of sizes, which can differ by an order or even two orders of magnitude. In the previous work, we reported on the experimental confirmation of our earlier assumption, which allowed us to solve this problem [2]. Such a solution is the process of electrospark erosion with significant overvoltage in the discharge gap. To implement such a solution, it was necessary to abandon the use of the usual scheme for constructing an electrospark installation (EDM) and we developed an experimental electrospark laboratory installation with overvoltage on dispersible electrodes (OV-EDM).

The developed experimental setup demonstrated the stable generation of a spark discharge in the discharge gap at a pulse energy of less than 100 mJ. Using an OV-EDM experimental setup, silver nanopowder was obtained with a narrow range of nanoparticle sizes. The functional diagram of the experimental OV-EDM installation in comparison with the diagram of the standard EDM installation is shown in figure 1.
The obtained positive results required further study of the features of the process of electric spark dispersion of materials with significant overvoltage of the discharge gap. As can be seen from the scheme of the OV-EDM experimental setup, its most interesting feature is the presence of an air discharge gap FV1, which ensures the creation of overvoltage on the working discharge gap FV2. Both discharge gaps together form a voltage divider during the discharge of capacitor C. In a first approximation, this voltage divider should have a significant impact on the operation of the OV-EDM experimental setup. Perhaps this divider will allow us to control the process of electrospark dispersion of materials. At the first stage of studies of the OV-EDM experimental setup, it was most interesting to study the effect of the ratio of the discharge gaps FV1 and FV2 on the process of producing nanopowders by electrospark erosion.

2. Experimental

In a new series of experiments, we decided to use silver working electrodes and produce silver nanopowders. The choice of silver as a dispersible material in the experiments was due to several aspects. First, as shown by experiments conducted earlier, silver nanopowders can be obtained by using distilled water as a working dielectric medium and they do not oxidize [2]. The choice of another material for dispersion could require the use of liquid nitrogen or other inert, primarily cryogenic liquids as a working fluid [1], which would significantly increase the cost of research. Secondly, we could use a well-studied plasmon resonance effect to quickly estimate the size of the resulting silver nanoparticles, which allows us to determine their characteristic size from the absorption spectrum of the colloidal solution of silver nanoparticles [3]. This approach to the study of the size of silver nanoparticles has already been used by other researchers and experimentally proved its adequacy [4, 5].

To study the effect of the ratio of the discharge gaps FV1 and FV2, a high-voltage capacitor (C) with a capacity of 1500 pF was installed in the OV-EDM experimental setup. The capacitor was charged from a high-voltage transformer with a frequency of 50 Hz to a voltage of 10 kV. When the capacitor was charged to the maximum voltage, the energy stored in it was 75 mJ, if the electric breakdown of the discharge gaps FV1 and FV2 did not occur at a lower voltage. The capacitor charge rate was set by the resistance R of the charging circuit, the resistance value (R) was 50 kOhm. Both dispersible silver electrodes, forming the gap FV2, were completely immersed in distilled water with a volume of 1 liter. For uniform dispersion of the electrode material during spark erosion, rotation of the working electrodes with respect to each other was provided.

During the experiments, the value of the interelectrode gap FV1 varied in the range from 800 to 1300 microns. The gap between the dispersed electrodes FV2 varied in the range from 50 to 100 microns. The dispersion time for each mode without changing the working medium was 120 seconds. During this time there is no accumulation of nanopowder in quantities that can affect the process of electric spark discharge in the liquid at the discharge gap FV2.

The results of the experiments showed that an increase in the discharge gaps FV1 and FV2 leads to an increase in breakdown voltage, as was to be expected. Figure 2 shows the waveforms of the
discharge voltage pulses measured on capacitor C, obtained at a value of 50 μm on the discharge gap FV2 and at values 1000, 1100, 1200, and 1300 μm of the air discharge gap FV1.

Figure 2. Oscillograms of discharge pulses with an FV2 gap of 50 μm and FV1 gaps of 1000 (a), 1100 (b), 1200 (c) and 1300 (d) μm.

Figure 3. Absorption spectra of a working medium with silver nanoparticles with an FV2 gap of 50 μm and FV1 gaps of 1000 (a), 1100 (b), 1200 (c), and 1300 (d) μm.
It is seen that the value of the breakdown voltage increases from a level of about 6.5 kV to a level of 8.0 kV. The absorption spectra of the working medium with silver nanoparticles obtained in these modes are shown in Figure 3. According to the spectra obtained, the peak of plasmon resonance for all experimental modes is 393 nm, which corresponds to an average size of silver nanoparticles of 10 nm [3]. A small half-width of the resonance indicates a small spread in the particle sizes of the nanopowder. At the same time, the visible difference between experimental modes is the resonance value, which is determined by the concentration of nanoparticles in the measured medium. With an increase in the FV2 gap from 1000 to 1300 μm, it changes almost twice.

In Figure 4 shows the oscillograms of the discharge voltage pulses measured on capacitor C, obtained at values of 1000, 1100, 1200, and 1300 μm of the gap FV1 with a value of 100 μm for the gap FV2. In this case, there is also an increase in breakdown voltage from 6.5 kV to 8.0 kV with an increase in the size of the discharge gaps FV1 and FV2. An increase in breakdown voltage is accompanied by an increase in the level of electromagnetic interference, leading to a malfunction of the measuring equipment, which is also seen in the oscillograms. The absorption spectra of the working medium with silver nanoparticles obtained in these modes are shown in Figure 5. It can be seen from the spectra that for all experimental modes, the resonance peak is 393 nm, as in the previous case. There is also a difference in the concentration of nanoparticles depending on the ratio of the sizes of the discharge gaps FV1 and FV2. It should be noted that in this case a more intense absorption in the ultraviolet region of the spectrum is also observed, which is especially seen in Figure 5 a.

3. Conclusions

Experimental studies of the effect of the ratio of the discharge gaps FV1 and FV2 on the process of obtaining nanopowders by electrospark erosion in OV-EDM have shown quite interesting and unpredictable results.

As a result of the studies, it was found that when the air discharge gap FV1 is much larger than the discharge gap FV2, their exact ratios do not affect the size of the resulting nanoparticles. The nanopowder obtained in the experiments are characterized by a small dispersion of particle sizes, but the particle size itself is determined by other factors of the process. With such ratios of discharge gaps, the voltage divider formed by the gaps FV1 and FV2 allows us to regulate only the performance of the process of obtaining nanopowders of materials.
Figure 5. Absorption spectra of a working medium with silver nanoparticles with an FV2 gap of 100 μm and FV1 gaps of 1000 (a), 1100 (b), 1200 (c), and 1300 (d) μm.

The obtained results show the necessity of further investigation of the process of electric spark dispersion of materials for obtaining nanopowders of conductive materials with a significant overvoltage of the discharge gap. Further research requires improvement of the experimental installation OV-EDM, including shielding discharge gaps FV1 and FV2 to reduce the level of electromagnetic interference affecting the measuring equipment.

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