Investigation of the process of obtaining nanopowders for promising powder compositions, applicable in additive technologies, by electrospark dispersion of conductive materials

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Abstract. An improved process for obtaining nanopowders by electrospark dispersion of conductive materials was investigated. To solve the problem of a significant variation in the size of the particles obtained, it was proposed to modify the standard scheme of an electrospark installation with the possibility of smoothly adjusting the energy of the discharge pulse in the range of less than 100 mJ. The obtained experimental results showed the possibility of obtaining nanopowders with a small scatter of particle sizes.

1. Introduction
The beginning of the 21st century was marked by the active introduction of additive technologies in the high-tech industry, which in turn led to the emergence of demand for a large range of powder materials from a variety of metals and alloys. Moreover, many types of consumable powders are required in small quantities, due to the small number of high-tech products produced from them. For the production of such powders of greatest interest are universal technologies that allow you to work with a variety of materials and obtain powders of different fractions in small quantities.

At the same time, in the production of metal powders for additive technologies, work is underway to improve their compositions and compositions in order to obtain their products of higher quality. In particular, it is proposed to mix micron-sized powders with nanopowders, which will improve accuracy, reduce roughness, reduce porosity and improve the parameters of products obtained by layer-by-layer laser or electron beam sintering [1].

It should be noted that the current level of cost of metal powder materials for additive technologies with only micron-sized particles is quite high [2], which is one of the obstacles to the introduction of additive technologies. Adding nanopowders to the composition of powder compositions will lead to an even greater increase in their cost. Thus, at present, it is important to create new technologies for the production of powder materials and compositions for additive technologies characterized by versatility and economic availability.

At the end of the last century, a group of American scientists from the University of California proposed to use the process of electrospark erosion of conductive materials in a liquid dielectric to produce submicron powders of magnetic materials for magnetic recording devices [3]. This process has long been used for electrospark processing of materials profiled or not profiled electrode,
including for molds and other critical parts of solid and refractory materials. That is, american researchers have proposed to use the already well-known phenomenon, optimizing this process so that the electrodes are mutually subjected to more intense destruction, accompanied by the formation of a powder from the electrode material. Conducted by them and other researchers, experimental studies have shown that the use of electrical erosion for dispersion of conductive materials is a convenient, versatile and low-cost method for producing both micron-size powders and nanopowders [4, 5]. However, when obtaining nanopowders in this way, it turned out that this method has a drawback, consisting in a large spread of nanoparticles in size. The difference in the size of nanoparticles obtained simultaneously in one process can reach two orders of magnitude, i.e. at the same time, nanoparticles ranging in size from several nanometers to hundreds of nanometers are obtained. With such a large variation in the particle size of the nanopowder, it is not an easy task to calibrate the particle size for the preparation of powder compositions with the desired properties for additive technologies.

Studies aimed at studying the effect of liquid dielectric media on the process of obtaining nanopowders, including organic and inorganic liquids, as well as cryogenic liquids, helped to better understand the process of obtaining nanoparticles, but did not allow to find a solution to the problem of significant dispersion of their sizes [6, 7]. Based on theoretical studies of this problem, we concluded that it can be caused by the fact that the process is based on a known method of electrospark processing of materials, so the researchers used standard equipment for electrospark installations. At the same time, in [6] it is suggested that for the synthesis of nanopowders, discharge pulses should have an energy of less than 100 mJ, which is impossible for many standard pulse generators of electric spark installations, which are usually used in experiments.

2. Experimental

To solve the problem of significant dispersion of the size of the obtained particles, we decided to modify the standard scheme of the electrospark installation, so as to be able to create a significant overvoltage on the dispersed electrodes, to stabilize the conditions of the breakdown between them and at the same time to smoothly adjust the energy of the discharge pulse, including in the range of less than 100 mJ. For this purpose, the circuit of the experimental spark installation, in series with the discharge interval, included a tunable switching element, simultaneously performing the function of a power divider of the discharge pulse. As such a tunable switching element, an uncontrolled air discharger with an adjustable interelectrode gap was used. Schematic standard electric-spark system and experimental setup is shown in figures 1(a) and (b), respectively.

![Figure 1. Schematic standard electric installation (a) and experimental electric installation (b).](image)

For generation of discharge pulses in the experimental electrospark installation, a high-voltage capacitor of the KV1-3-16KV-1000 (C) brand with an electric capacity of 1000 pF was used. The discharge capacitor was charged from an adjustable high-voltage AC transformer to voltages from 1 to 10 kV. The limitation of the charging current and, accordingly, the duty cycle of the discharge pulses were carried out by the resistor R.
Unmanaged air discharger with adjustable gap (FV1) was two metal electrodes with an area of mutual overlap of about 50 mm², while the distance between the electrodes can be adjusted within a sufficiently wide range by rotating the thread of one of them. The photo of the air discharger installed in the experimental installation is shown in figure 2.

![Figure 2. The air discharger.](image1)

![Figure 3. Dispersible electrodes.](image2)

The electrodes subjected to electrospark dispersion in the experimental setup were made of silver, for experiments between them an interelectrode gap of 70 µm (FV2) was exposed. Taking into account the results presented in [6], the rotation of the dispersed electrodes relative to each other was provided, which ensures a more uniform consumption of the material of the dispersed electrodes. A photo of the dispersible electrodes prepared for the experiments is shown in figure 3. During the experiment, the dispersed electrodes were immersed in a bath with a liquid dielectric, which was used as distilled water.

During the experiments, the discharge capacitor was charged to a voltage that was sufficient for the simultaneous breakdown of both the uncontrolled air discharger and the interelectrode gap between the dispersed electrodes. It was found that the value of the gap of the air discharger in 1 mm, the voltage at which there was an electric breakdown of both (air and water) gaps was 4 kV, and the pulse discharge current reached a value of 2 kA.

![Figure 4. Diagram distribution of the obtained particles by size.](image3)

![Figure 5. TEM image of the obtained particles](image4)

To study the particles obtained in the experimental electrospark installation, the process of dispersion of silver electrodes was carried out for 10 minutes. The nanopowders obtained in the form of a colloidal solution were send to study the particle size distribution. Measurement of the size of the obtained particles was carried out in two ways to improve the reliability of the data. In the first case,
nanopowders were investigated by dynamic light scattering (DLS) using the device Photocor Compact-Z. The results obtained in the measurement are shown in figure 4. As can be seen in the figure, the size spread of the obtained particles is quite small, the diameter of more than 80% of the particles is in the range from 20 to 45 nm. To confirm the results of the DLS measurement, a study of the obtained particles was carried out using a transmission electron microscope (TEM) of LEO-912 AB OMEGA. A typical TEM image of the nanopowder particles is shown in figure 5. As can be seen in the figure, TEM images confirm the measurement results obtained by the DLS method. For studies of the composition of the obtained nanopowder particles were compared for their electronograms with reference electronograms silver. The comparison confirmed that the obtained particles consist of crystalline silver and do not contain significant impurities of oxides or salts.

3. Conclusions
The obtained experimental results showed the possibility of obtaining nanopowders with a small particle size spread by the method of electrospark dispersion of conductive materials, which makes it possible to create a new technology for the production of powder compositions for additive technologies, characterized by versatility and economic availability in small-scale production.

The proposed scheme of the experimental electrospark installation provides additional opportunities for controlling the process of production of nanopowders by more accurate control of the parameters of the electric discharge at low values of the energy of discharge pulses.

To create a new technology for the production of powder compositions, it is necessary to conduct further studies aimed at establishing the relationship between the parameters of the experimental electrospark installation and the characteristics of the nanopowders obtained for various metals, alloys and working dielectric fluids.

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