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Investigation of the synthesis of nanoparticles by the method of spark erosion with overvoltage of the discharge gap

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Abstract. The silver nanoparticles were studied, which were obtained by spark erosion with an overvoltage of the discharge gap. Experimental results show the possibility of producing nanoparticles of conductive materials with a narrow size distribution.

1. Introduction

The unique properties of nanoparticles have been the subject of intense research for several decades. Modern science has shown the possibility of using them in many applications, including for storage devices, creating new energy storage devices, MEMS, etc. [1-3]. A number of methods for obtaining nanoparticles have been developed, which can be conditionally divided into two groups: chemical methods and physical methods (dispersion) [4-6].

To obtain submicron and nanoscale particles of conductive materials (metals, alloys and semiconductors), a method of synthesis based on the electrospark erosion of raw materials in a liquid dielectric using the Electrical Discharge Machine (EDM), which is very attractive in its simplicity and accessibility, was proposed [7]. However, this method and a number of other known methods for synthesizing nanoparticles have the disadvantage of a large dispersion in the sizes (from 1 to 100 nm) of produced nanoparticles, which makes it extremely difficult to obtain or isolate nanoparticles with the required characteristics.

Further studies aimed at studying the method of electrospark erosion confirmed the possibility of obtaining nanoparticles of various conductive materials, including complex composition [8, 9]. Investigations of the influence of dielectric liquid media used in the synthesis of nanoparticles [10], including cryogenic liquids [11], have been carried out. A decrease in the size of the resulting metallic nanoparticles upon exposure to a liquid dielectric in the discharge zone by ultrasound due to cavitation and mixing has been established [12].

The results obtained by different groups of researchers made it possible to better understand the process of electrospark synthesis of nanoparticles, but could not eliminate the drawback associated with a large scatter of nanoparticle sizes. This can be due to several factors. In [10], it is proposed to use pulses with minimal energy for the synthesis of nanoparticles, which is difficult with the use of standard pulse generators for EDM. In addition, as shown by the results of mathematical modeling of the behavior of vapor-gas bubbles formed in a liquid dielectric in breakdown [13], their collapse is in most cases accompanied by division into several parts whose dimensions depend on the condition of their formations, which subsequently leads to the formation of several nanoparticles.



On the basis of these results and arguments, we made the assumption that a decrease in the spread of the sizes of the produced nanoparticles is possible due to a change in the conditions for the occurrence of a spark breakdown, so that the process proceeds much faster. This can be achieved if a significant overvoltage is created on the EDM electrodes, which is not possible in any previously described scheme. To create an overvoltage, another switching element must be added to the circuit, which would commute the charged capacitor and the EDM electrodes to a high voltage.

2. Experimental

An experimental EDM was developed with the possibility of creating an overvoltage at the electrodes. The functional diagram of the experimental EDM is shown in Fig. 1.

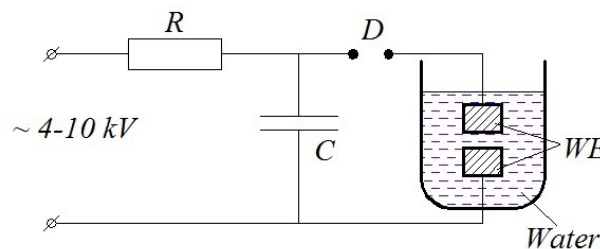


Figure 1. Functional diagram of the experimental EDM. C - high-voltage capacitor, R - limiting resistor, D - spark gap, WE - working electrodes

A high-voltage capacitor (C) with a capacity of 1000 pF was used in the experimental EDM. The capacitor was charged from a high-voltage AC transformer (50 Hz) to a voltage of 1-10 kV. For capacitor switching (C), an air electric spark gap (D) is used, which is an air gap of 1 mm width between two 50 mm² electrodes. The photo of the air electric spark gap is shown in Fig. 2a. The charge rate of the capacitor is determined by the resistor (R). The capacitor charged to a voltage sufficient to breakdown the air gap (D) and the electrode system (WE) is discharged through the air gap D to the working electrodes (WE) immersed in the liquid dielectric.

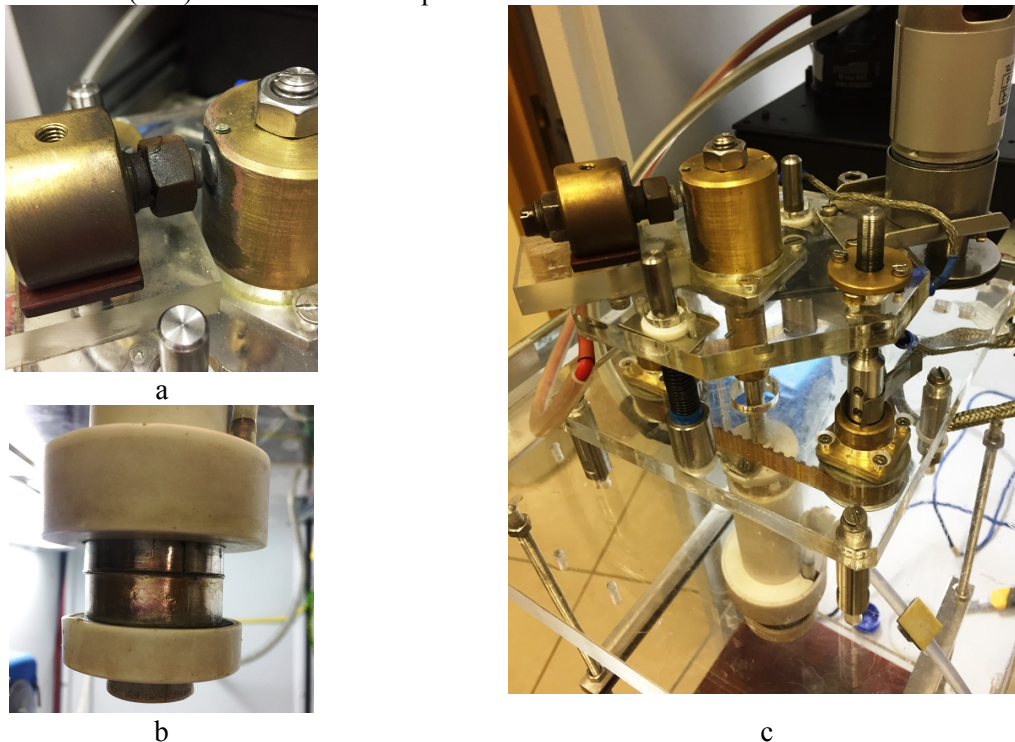


Figure 2. Air electric spark gap (a), immersed silver electrodes (b), experimental EDM (c)

As a liquid dielectric, distilled water was used in the experiment. The working electrodes were made of silver, their photograph is shown in Fig. 2b. For the experiment, a gap of 70 μm was established between them. For uniform consumption of electrode material during erosion, rotation of working electrodes relative to each other was provided. The general view of the collected experimental EDM is presented in Fig. 2c.

The experiment showed that at the indicated values of the parameters of the experimental setup, the breakdown voltage was of the order of 4 kV, and the discharge current in the pulses reached values of 1 \div 2 kA. The obtained samples of silver nanoparticles were subjected to the investigation of the size distribution by the method of dynamic light scattering (DLS), the measurement was carried out with the Photocor Compact-Z. The resulting particle size distribution is shown in Fig. 3. As can be seen from the figure, the distribution of the nanoparticles obtained in size is rather narrow. The diameter of more than 80% of nanoparticles lies in the range of 20-45 nm.

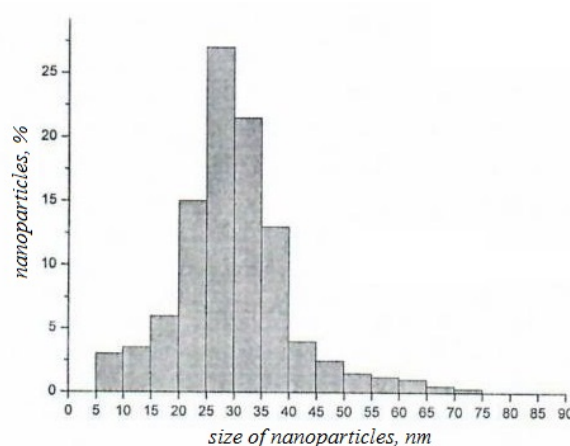


Figure 3. Distribution of silver nanoparticles by size

In addition to the DLS method, images obtained with the transmission electron microscope LEO-912 AB OMEGA were used to confirm the nanoparticle size distribution. In the pictures, it is seen (Fig. 4) that most particles are spherical and have a size of 20 to 40 nm.

The electron diffraction pattern of the obtained nanoparticle samples and its comparison with a sample of crystalline silver showed that the nanoparticles obtained consist of crystalline silver, without a noticeable admixture of oxides or salts (Fig. 5).



Figure 4. Photographs of the silver nanoparticles on an electron microscope

3. Conclusions

The possibility of producing nanoparticles by electrospark erosion under conditions of considerable overvoltage of the discharge gap was demonstrated. The resulting silver nanoparticles had a spherical shape and a rather narrow size distribution.

For a better understanding of the effect of overvoltage of the discharge gap on the production process of nanoparticles, it is planned to investigate this process for different ratios of discharge gaps and various conductive materials.

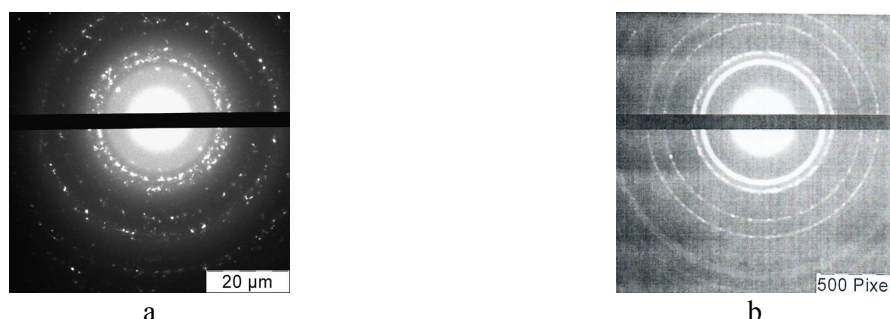


Figure 5. Electronogram of silver nanoparticles (a) and solid silver (b)

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