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Investigation of the synthesis of nanoparticles by pulsed electrical erosion in the conditions of overvoltage of the discharge gap

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Abstract. The process of obtaining silver nanoparticles by the method of electro erosion of metal electrodes when over-voltage discharge gap therebetween. For this purpose, a new scheme of an electric pulse erosion unit is proposed and investigated. Experimental results show the possibility of obtaining nanoparticles of conductive materials with a narrow size distribution by this method.

1. Introduction

The unique properties of nanoparticles have been the subject of intensive research for several decades. Modern science has shown the possibility of their use in many applications, including storage devices, creating new energy storage devices, MEMS, etc. [1–3]. To date, a number of methods for producing nanoparticles have been developed, which can be divided into two groups: chemical methods and physical methods [4–6]. The most common physical methods for producing nanoparticles are dispersion and laser ablation, which are carried out in vacuum or liquid media.

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. However, it should be noted that such a disadvantage is typical for most physical high-performance methods for producing nanoparticles.

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

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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 figure 1.

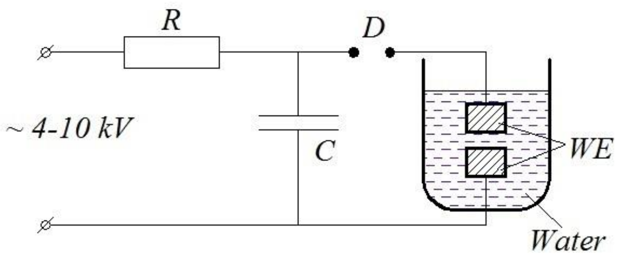
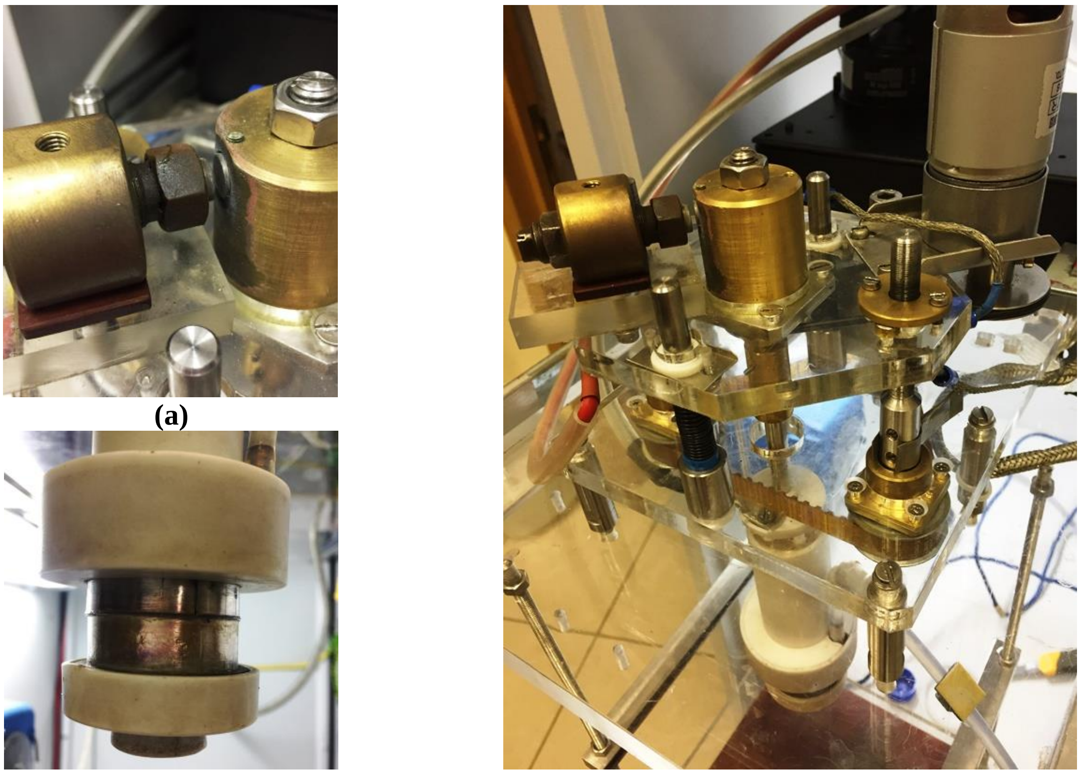


Figure 1. Functional diagram of the experimental EDM: C - high-voltage capacitor, R - limiting resistor, D - spark gap, WE - working electrodes.



(a) (b) (c)

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

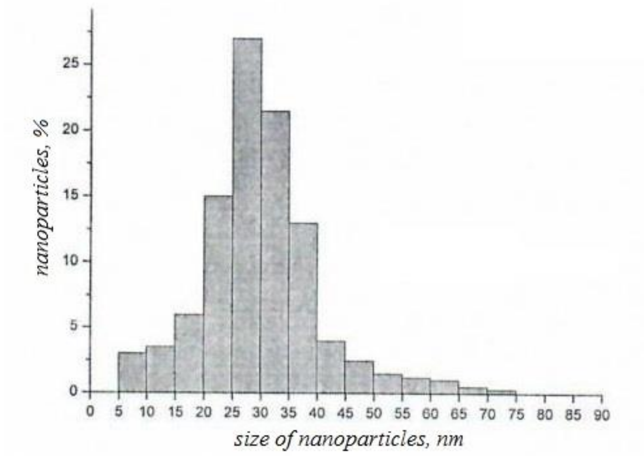


Figure 3. Distribution of silver nanoparticles by size.

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 mm^2 electrodes. The photo of the air electric spark gap is shown in figure 2(a). 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.

Distilled water was used as a liquid dielectric in the experiment. The working electrodes were made of silver, their photo is shown in figure 2(b). For the experiment, a gap of 70 μm was established between them. For uniform consumption of the electrode material during erosion, the rotation of the working electrode surfaces relative to each other was provided. A general view of the assembled experimental setup is shown in figure 2(b).

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 ÷ 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 figure 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.

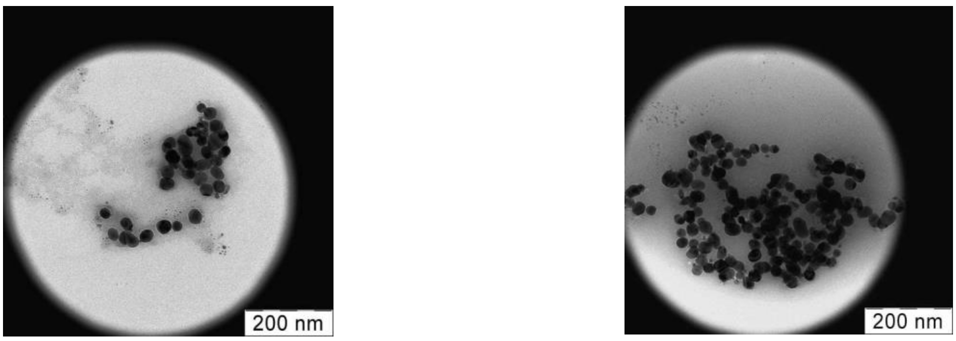
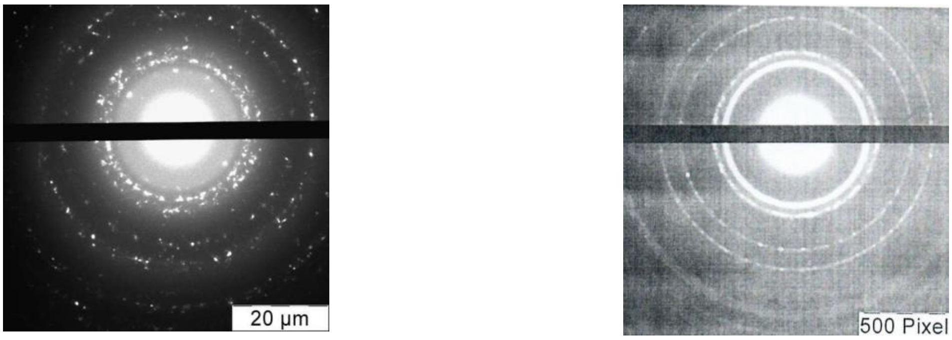


Figure 4. Typical photographs of the silver nanoparticles on an electron microscope.

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 (figure 4) that most particles are spherical and have a size of 20 to 40 nm.

Electronograms samples obtained nanoparticles is shown in figure 5(a), electronograms sample of crystalline silver is shown in figure 5(b). The comparison showed that the obtained nanoparticles consist of crystalline silver, without a noticeable impurity of oxides or salts.



(a) **(b)**

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

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.

Acknowledgement

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