Using Nanosecond Electron Beam to Produce a Silver Nanopowder

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Abstract – Experiments with a URT-0.5 accelerator (0.5 MeV, 50 ns, 1 kW) generating a nanosecond electron beam for irradiation of silver nitrate in various liquid solutions (water and toluene) were performed with the aim of producing a silver nanopowder.

A radiochemical reaction allows making weakly agglomerated pure Ag powders with particles 3–5 nm and 20–50 nm size by irradiation in toluene and water respectively.

The injection of the nanosecond electron beam energy to the solution is optimal. If the absorbed dose increases, the output of the radiochemical reaction does not grow, but more agglomerated powders are synthesized.

1. Introduction

Synthesis of weakly agglomerated silver nanopowders is important for making of nanostructured materials used in microelectronics, electrochemistry and synthesis of optoelectronic sensors, pigments, etc. [1].

Bactericidal properties of silver ions are of special importance since thanks to these properties silver nanopowders may serve as the basis for development of new classes of bactericidal preparations and various pharmaceutical substances in medicine and agriculture [2, 3].

Methods used for synthesis of silver nanopowders can be classified by their production techniques. Today the basic methods for production of silver nanopowders and their aqueous suspensions are chemical methods. In this case, it is important to provide conditions facilitating the formation of fine silver particles.

The most widespread among chemical methods is reduction of silver particles from aqueous solutions of silver salts in the presence of stabilizers [4–6]. The reducing agents are hydrogen and hydrogen-containing compounds (tetrahydroborates [4, 7] and citrates of alkali metals [8], hypophosphites, alcohols [9], organometallic compounds [10]). Silver nanoparticles can be reduced either on the surface of pre-synthesized latex microspheres in the presence of a reducing agent or at the stage of monomer polymerization [11].

Physical methods include sputtering or mechanical grinding of bulky materials [12, 13], photoreduction of silver salts [14], laser ablation of solids in liquids [15], and the use of electron beams [16].

The chemical methods have some drawbacks such as the influence of the concentration of the starting components on the progress of the reaction and the post-effect showing up as the coagulation of particles in the solution. Coagulation can be suppressed in the physical methods by addition of surfactants.

The greatest interest among the physical methods is attached to the use of electron beams since it was found that nanopowders with particles 10–60 nm in size can be produced at a relatively small fluence of electrons ($2 \cdot 10^{13}$–$3 \cdot 10^{15}$ particles/cm^2) [16]. As the electron fluence increases, the particle size diminishes unlike in the chemical methods when the increase in the reaction time leads to the growth of the particle size.

Nanosecond repetitive accelerators [17] allow a rather simple control of the exposure of reaction products and, with this in mind, we decided to study the possibility of producing nanopowders with the use of these accelerators.

2. Description and experimental results

Experiments on irradiation of solutions of silver nitrate in different liquids by a nanosecond electron beam from an URT-0.5 accelerator (0.5 MeV, 50 ns, 1 kW [18]) were performed with the aim of producing silver nanopowders.

The following silver nitrate solvents (0.3 g per 10 ml of the solvent) were studied:

1) distilled water;
2) toluene with addition of ethylene glycol (0.3 ml) and ethyl alcohol (0.3 ml);
3) ethyl alcohol;
4) isopropyl alcohol;
5) 10% ammonia solution;
6) isopropyl alcohol with addition of 2 ml of a 10% ammonia solution.

The solutions were poured into a Petri dish so that the liquid layer was not more than 1 mm thick. The accelerator operated at a frequency of 10 pps for irradiation of the samples and the absorbed dose on the liquid surface in one minute was 0.36 MGy. The level of the doses was chosen considering the data of the study [16] since the design value of the absorbed dose at the lower limit of the fluence was about 0.6 MGy. The microscopic analysis was made in LEO-982 scanning electron microscope with EDX firm RONTEX and transmission electron microscope JEM-200.

It should be noted that the silver reduction reaction was vigorous in all the solvents, especially in the aqueous solutions.

Upon irradiation of the aqueous solutions, some reaction products emerged to the surface as foam, while the others precipitated. The foam had a layered structure (Fig. 1) and the precipitate contained a sufficiently agglomerated nanopowder (Fig. 2) with particles 30 to 200 nm in size. Foam was not formed and the powders were less agglomerated (Fig. 3) after irradiation in the ammonia solution. In any case, the EDX analysis was achieved the silver.

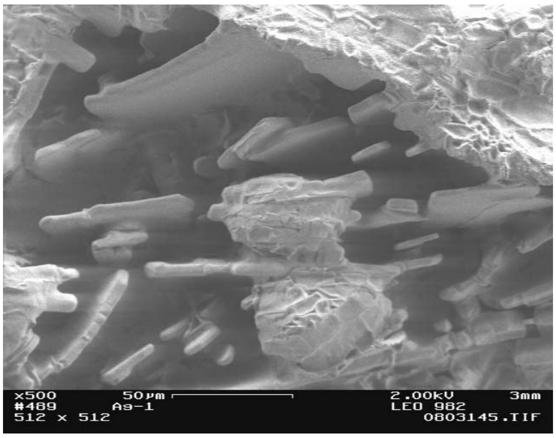

Fig. 1. SEM photograph of foam on the aqueous solution

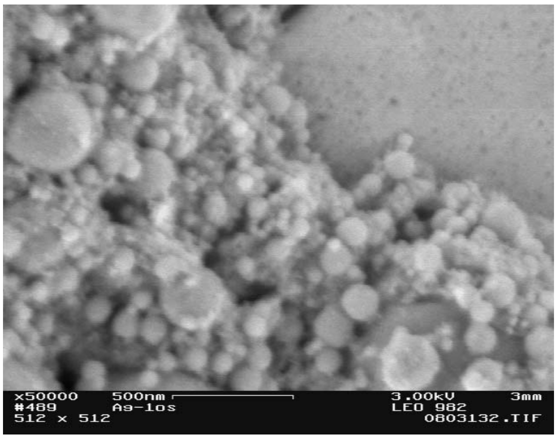

Fig. 2. SEM photograph of the precipitate in the aqueous solution

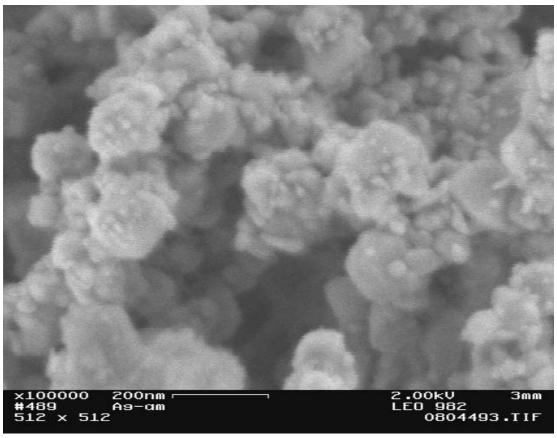

Fig. 3. SEM photograph of the nanopowder in the ammonia solution

Weakly agglomerated silver powders with particles 3–5 nm in size (Fig. 4) were synthesized by irradiation of the toluene solution. It was found that the increase in the irradiation time from 1 to 10 minutes did not lead to the decrease in the particle size as was the case in [16] or the increase in the reaction yield; at the optimal dose and the irradiation time of 5 minutes (Fig. 5) the degree of agglomeration of the synthesized powders increased and junctions were formed between the particles (Fig. 6).

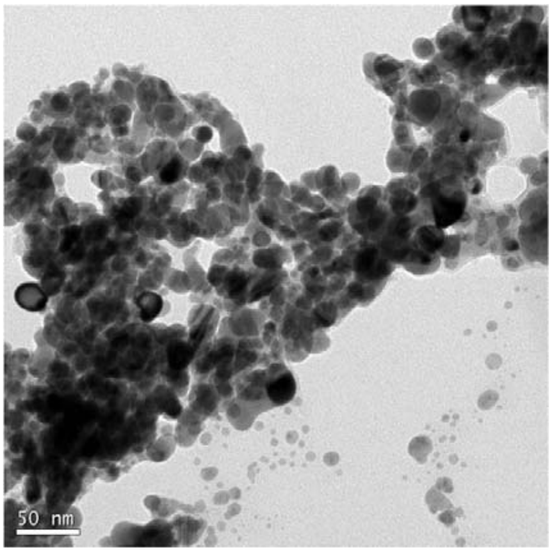

Fig. 4. TEM photograph (irradiation in toluene for 1 minute)

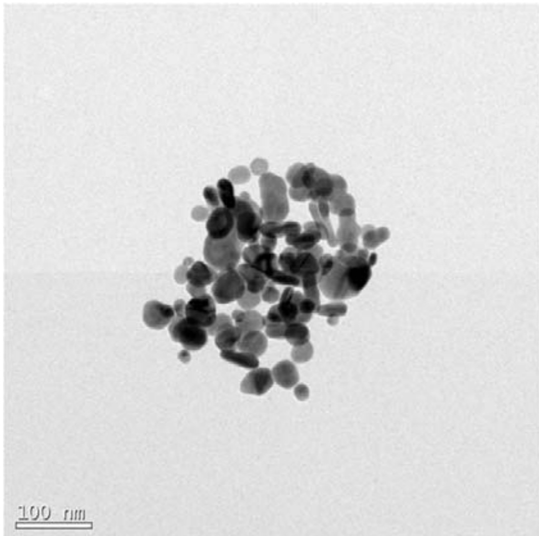

Fig. 5. TEM photograph (irradiation in toluene for 5 minutes)

However, the best results were obtained after irradiation of the hydrocarbon solvents.

Notice that the presence of ethylene glycol in the solution considerably complicated the preparation of the samples for the TEM examination. For this reason, the solution was irradiated in isopropyl alcohol, producing weakly agglomerated powders (Fig. 7). An ammonia solution had to be added for good solubility of silver nitrate.

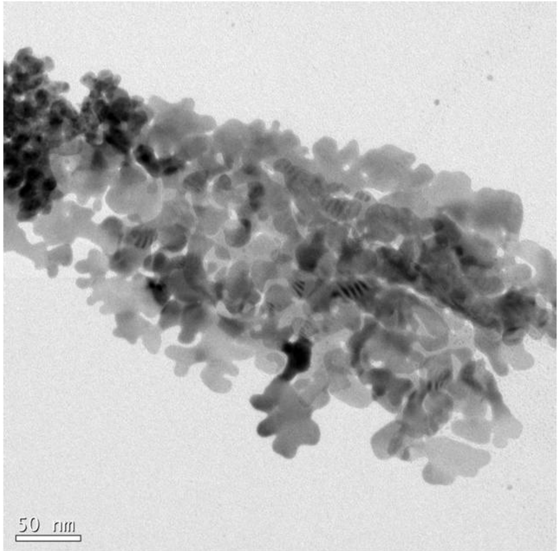

Fig. 6. TEM photograph (irradiation in toluene for 10 minutes)

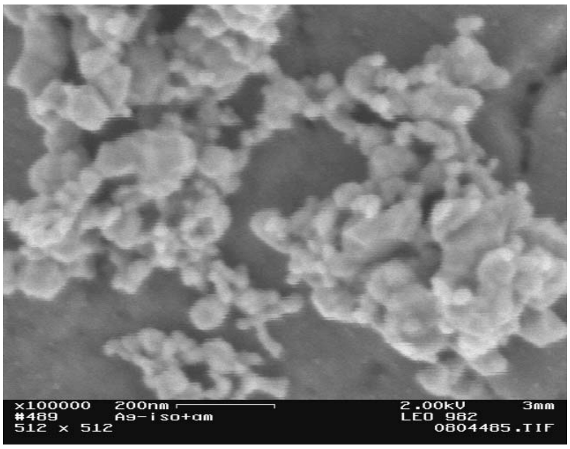

Fig. 7. SEM photograph of the nanopowder in the isopropyl alcohol solution with ammonia

3. Conclusion

It is found that weakly agglomerated silver powders with particles 3–5 nm and 30–50 nm in size can be produced by exposure of a silver nitrate solution in toluene and water to an electron beam respectively.

The optimal input of the electron beam energy to the solution and a considerable increase in the absorbed dose do not lead to the increase in the reaction yield, but cause a larger agglomeration of the synthesized powders.

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