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Preparation of silver nanopowders by a controlled wet-chemical synthesis

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ABSTRACT

The synthesis of silver nanopowders prepared by the method of controlled wet-chemical reduction is reported. The effects of mixture ventilation of NO_2 and O_2 in process of silver nanopowders are investigated. It is found that the morphology and tap density of silver nanopowders can be influenced by the ventilation volume in the process. The ventilation effects are discussed on the basis of the aggregative growth theory. It is reasonable to speculate that ventilation has two effects on the silver nanopowders. On the one hand, ventilation chemically promotes the incorporation of silver nuclei, on another hand, the shearing action of ventilated gases physically improves the final tap density of nanopowders.

1. Introduction

Silver nanopowders with ultra-fine size and well-distribution are widely used in the electronics industry due to their abilities of moving freely [1]. As is reported in various studies, electronic properties of these silver nanopowders are strongly dependent on their shapes, size, crystallization, composition and surface modification [2]. In this regard, nano-sized silver powders have attracted a great deal of attention in recent years. Generally, silver nanopowders can be prepared by numerous methods such as sonochemical deposition [3], spray pyrolysis [4], electron beam evaporation [5], photochemical reduction [6,7], and thermal plasma [8]. Compared with other methods, chemical reduction method usually does not require special equipment or rigorous conditions [9,10]. From a practical point of view, chemical reduction method is most preferable for obtaining good quality silver nanopowders [11,12]. Besides these, shapes and crystalline of silver powders can be well-controlled by altering the concentration of the reactants and constituents of the solutions [13].

In this study, we develop a simple wet-chemical synthesis route for preparing spherical silver nanopowders with high tap density. With ventilation of NO_2 and O_2 gas, large scale silver nanopowders are obtained from a reduction of silver nitrate with ascorbic aqueous solution at room temperature. Moreover, it is interesting to find that morphology of spherical silver nanopowders can be easily controlled by altering the mixture ventilation of NO_2 and O_2 .

2. Experimental

To obtain non-agglomerated and high-tap-density silver nanopowders, the wet-chemical reduction in aqueous solutions of silver nitrate (SCRC 99.8% pure) is used. Ascorbic is widely used as a middle reducing agent, and gelatin aqueous is chosen as a dispersant in this work. Silver nanopowders are prepared by a controlled chemical reduction method, as presented in Eq. (1).

$$2AgNO_3 + C_6H_8O_6 = 2Ag + C_6H_6O_6 + 2HNO_3 \quad (1)$$

$$4NO_2 + O_2 + 2H_2O = 4HNO_3 \quad (2)$$

The solution is stirred at 200 rpm in 30 min. The mixture of NO_2 and O_2 (volume ratio, 6:4) is simultaneously ventilated into the solution, which leads to an oxidation reduction reaction in solution as Eq. (2). The products are centrifuged at speed of 4000 r min^{-1} and dried in vacuum oven. Direct observation of the silver termination is made by X-ray diffraction (XRD, Rigaku, D/MAX-RB) with a scanning rate of 0.02° s^{-1} in 2θ ranging from 30° to 80°. The crystal structure is investigated by scanning electron microscopy (JEOL, JSM-6060) and transmission electron

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microscopy (Hitachi, HT7700). Particle size is measured by laser diffraction particle size analyzer (Mastersizer, 3000). And the tap density is measured by a heap densitometer (Goodwill, JV2000).

3. Results and discussion

All dispersions are prepared by mixing aqueous solutions containing silver nitrate and gelatin aqueous with ascorbic acid solutions. By altering the mixture ventilation in the process, particles are formed in different shapes and sizes. The so-obtained silver nanoparticles are aggregated into the structures in Fig. 1.

In Fig. 1, SEM and TEM of the silver nanopowders synthesized in different ventilation volumes are illustrated respectively. Using actually the same conditions, but increasing the mixture ventilation, the silver particles tend to separate self-evidently and become spherical, and the tap density of silver powders monotonically increases from 4.2 g/cm^3 to 4.8 g/cm^3. Meanwhile, the particle size begins to increase then decreases and returns to increase again. It indicates the silver morphology and size are relevant to the ventilation volume, and they can be controlled by modifying the aggregation degree of silver nanoparticles. The particles described in this study shows X-ray patterns characteristic of silver, although the degree of crystallinity differs, as illustrated in Fig. 2.

Fig. 2 shows the XRD patterns of the silver nanopowders prepared only in same conditions but varying the ventilation volume. Characters of a, b, c, d, e, f and h indicates the ventilation volume of 5, 8, 10, 12, 16, 18 and 20 mL respectively. From the pattern, the diffractogram exhibits the crystalline characteristic peaks of (111), (200), (220) and (311) crystal planes at 37.81°, 43.93°, 64.21° and 77.14° respectively. According to Debye–Scherre formula [14], the

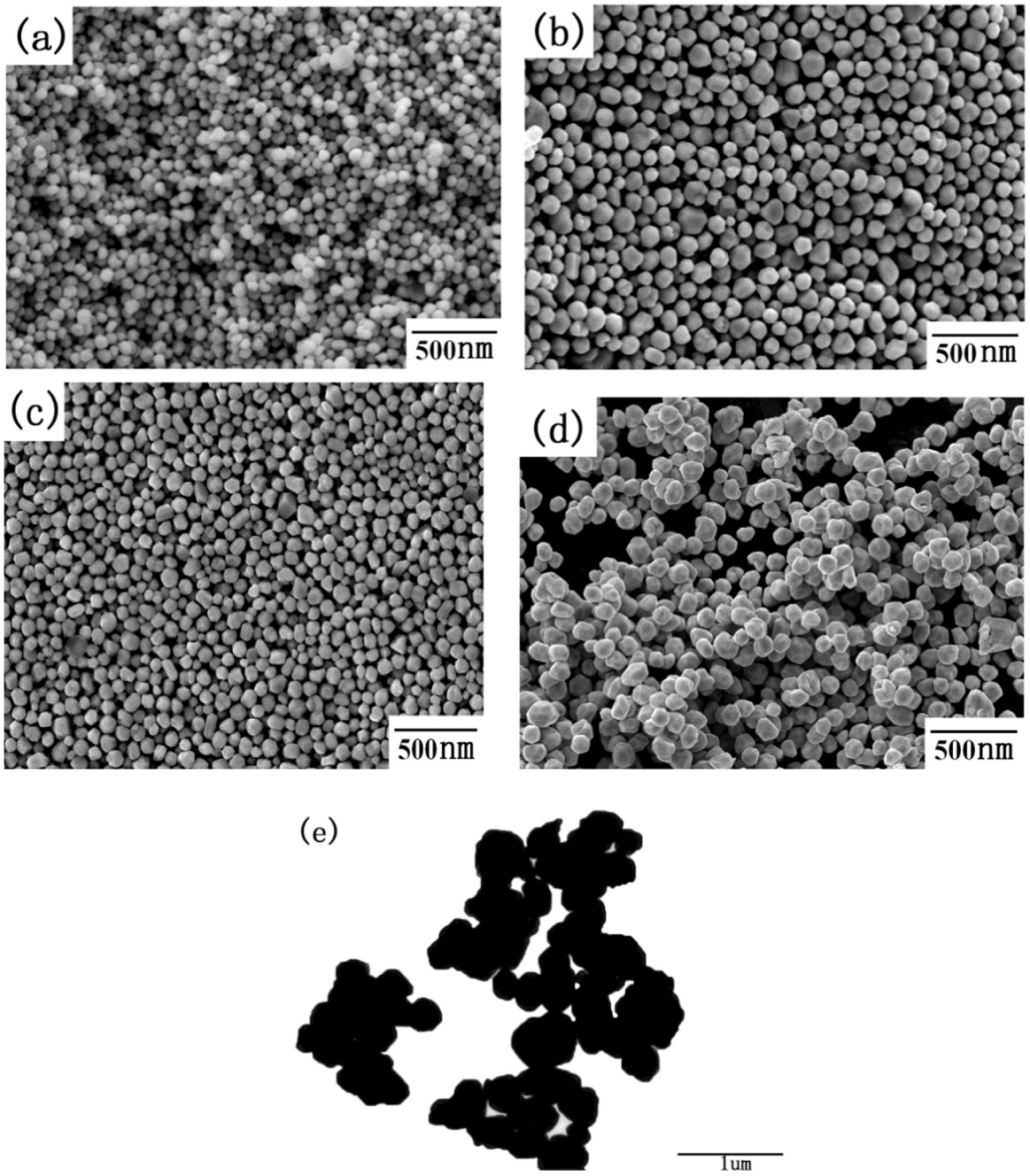

Fig. 1. SEM of different silver nanopowders synthesized with the mixture ventilation volumes of (a) 8 mL, (b) 10 mL, (c) 16 mL, (d) 20 mL and (e) TEM of silver nanopowders.