

Fabricating the spherical and flake silver powder used for the optoelectronic devices

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Abstract. The spherical and flake silver powder with different particle size for the optoelectronic devices was partly prepared by using chemical reduction and ball milling method, and characterized by scanning electron microscope (SEM), X-ray diffraction (XRD), laser particle size analyzer and thermo-gravimetric(TG) analyzer. The particle size of three series of spherical silver powder fabricated by chemical reduction is about 1.5 μm , 1 μm and 0.6 μm , respectively; after being mechanical milling, the particle size of flake silver powder with high flakey rate is about 10 μm , 6 μm and 2 μm respectively. Thermo gravimetric (TG) and XRD analyses showed that the silver powders have high purity and crystalline, and then the laser particle size and SEM analyses showed that the silver powders has good uniformity.

1. Introduction

As a key type of material of the optoelectronic devices, the silver powder (SP) is widely used in electronic industry, such as photovoltaic battery, membrane switch, wave filters and other electronic equipments [1-6] et al. The physical performance of SP has an important influence on the photoelectric devices. In recent years, researchers had done a lot of work in preparing the SP by various methods. Wang H. first prepared the spherical and mono-disperse silver powder with average particle size of about 1~2 μm by chemical reduction [7]. Liang H. Z. fabricated the hexagonal sheet silver with the thickness of less than 0.1 μm , and the maximum projection length of 0.2~0.5 μm in the medium of glycol [8]. Chen S H and his workers fabricated nanodisc with the thickness of 20~30nm and the diameter of 40~300nm in the CTAB solution containing silver crystal nucleus [9].

In the paper, the spherical and flake silver powder were prepared by chemical reduction and mechanical milling. And the influencing factors of fabricating the silver powder were investigated, such as surfactant concentration, reaction time etc.



2. Experiment

2.1 Reagent

Distilled water; Silver nitrate; Ammonia; Ascorbic acid; Ethanol; Polyvinylpyrrolidone (PVP, K30); Ethylene glycol; Oleic acid.

2.2 Fabricating the spherical silver powder

The PVP was dissolved in distilled water to form the reaction medium. Then the silver nitrate was dissolved in the PVP solution to form 0.1mol.L⁻¹ silver nitrate solution. Finally the solution was confected into the silver ammonia solution. At the temperature of 80°C, the reducer solution was added into the silver ammonia solution at even rate and stirred at the same time. After the reaction finished, the silver powder was obtained by centrifugation, then washed by distilled water and ethanol successive for several times, finally dried in a vacuum oven.

2.3 The fabrication of the flake silver powder

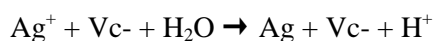
During the ball milling, ethanol, oleic acid and the agate ball of a diameter of 1mm acted as the milling medium, the assistant, and the milling ball. The mass ratio of the milling ball to the spherical silver powder, and to the spherical silver powder was 12:1 and 1:1, respectively. The milling speed achieved 500rad/min.

When the ball milling finished, ethanol was used to clean the silver powder adhering to the surface of milling ball. Separated silver powder was dried in vacuum drying oven.

3. Result and discussion

3.1 The influence of the PVP concentration

PVP could chelate with silver ions, change the reduction potential of oxidation-reduction reaction, thus nucleation and growth rate of silver particles was reduced. In addition PVPs which adsorbed on the surface of silver particles reduce the surface energy of crystal nucleation and make it consistent in all directions. In this case the silver atoms could grow evenly on the surface of crystal nucleus, which results in that the SPs with uniform size and spherical morphology were easily prepared. Furthermore, there was steric hindrance effect which made the potential barrier among the particles increase rapidly due to the PVP adsorbed on the surface of the particles. Thus PVP could make the SPs be monodisperse during reaction, solid-liquid separation and vacuum drying. Reaction was shown as follows:



As shown in Figure 1 (a), (b) and (c), the SP was prepared when 2%, 5% and 10% of PVP was added to the solution, respectively. The SP's size decreases with the increase of PVP concentration. The reason was that with the increasing of PVP concentration, the more PVP covered on the surface of SPs to prevent the crystal particles from growth. It was estimated that the concentration of PVP was inversely proportional to the square of the SP's size.

The SP with a diameter of about $1.5\mu\text{m}$ was tested by the laser particle size analyzer, as was shown in Figure 2. Compared with SEM characterization, it also testified that the SP had the property of monodispersity.

3.2 The Influence of the milling time

There were SEM photographs of the three kinds of SP milled for 8h and 15h, respectively in Figure 3. The flake rate was higher and higher with the increase of the milling time. In addition, the forming process of the flake silver powder was analyzed from the photographs. Firstly, large bulk particles formed gradually under the external force. With the increase of the milling time, the number of spherical particle was getting smaller and smaller, and vanished eventually. Then, the bulk particles were squeezed by the milling ball, and began to be thin. Finally, the flake silver powder formed after enough milling time.

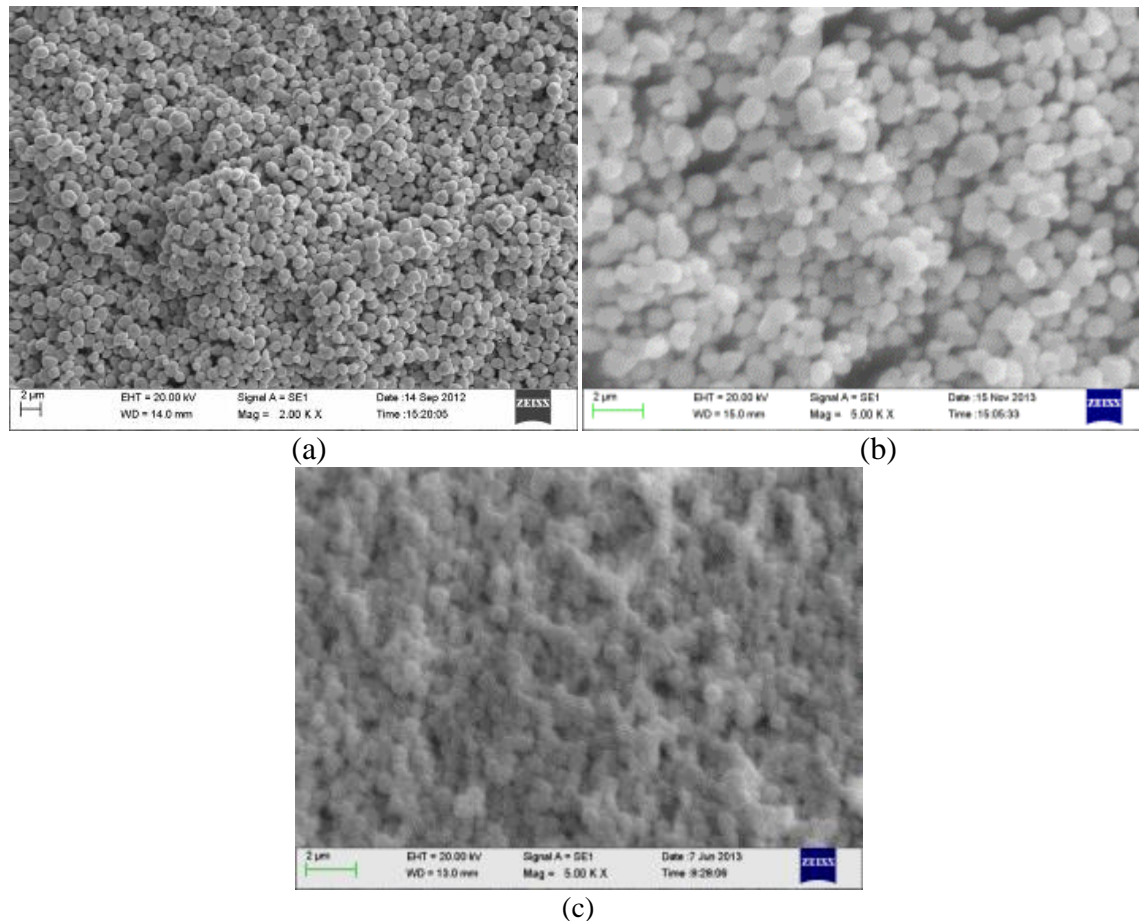


Figure 1. The SEM photographs of silver powder reduced in the case that the mass percentage concentration of PVP was 2% (a), 5% (b) and 10% (c)

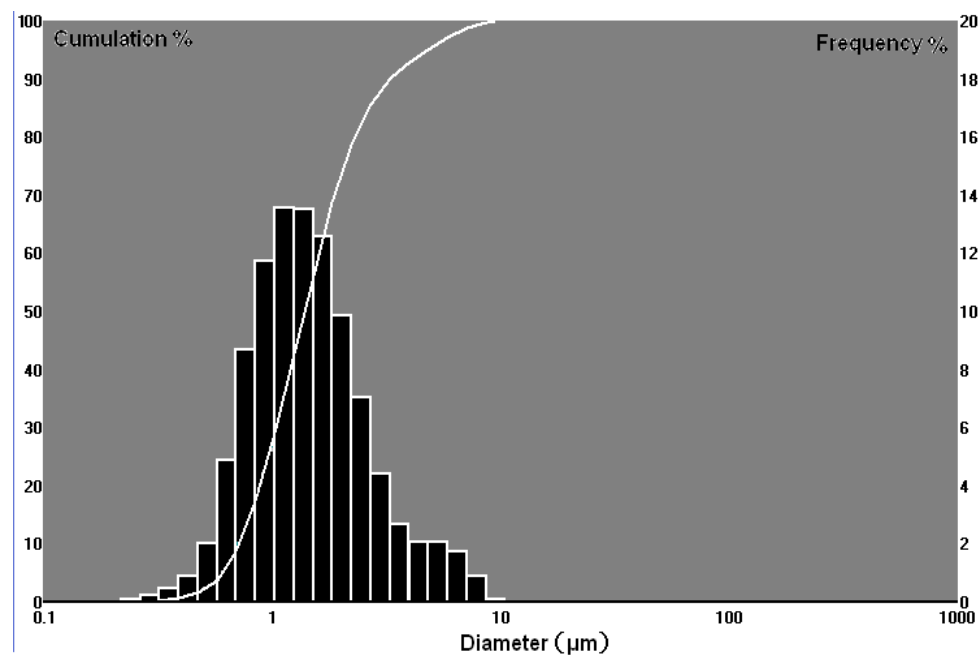
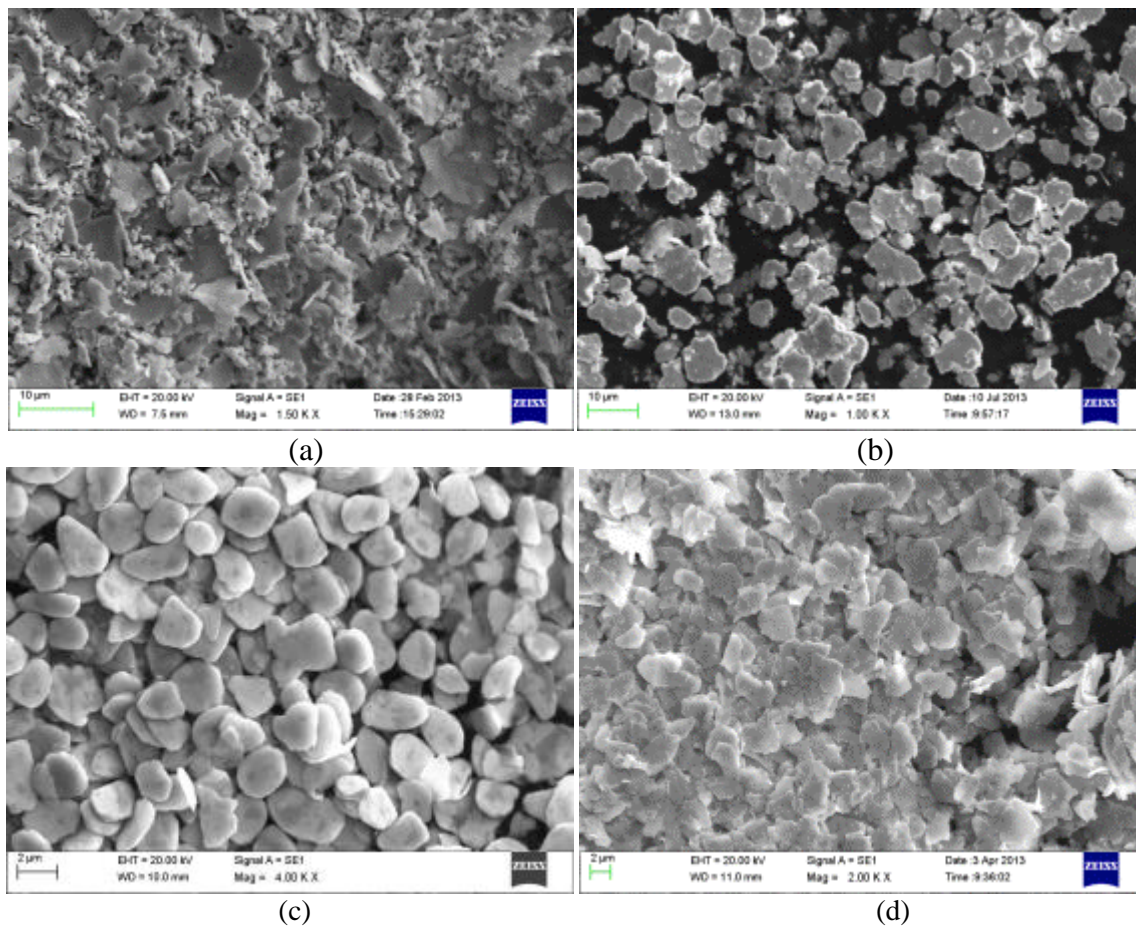


Figure 2. The result of silver powder by the laser particle size analyzer



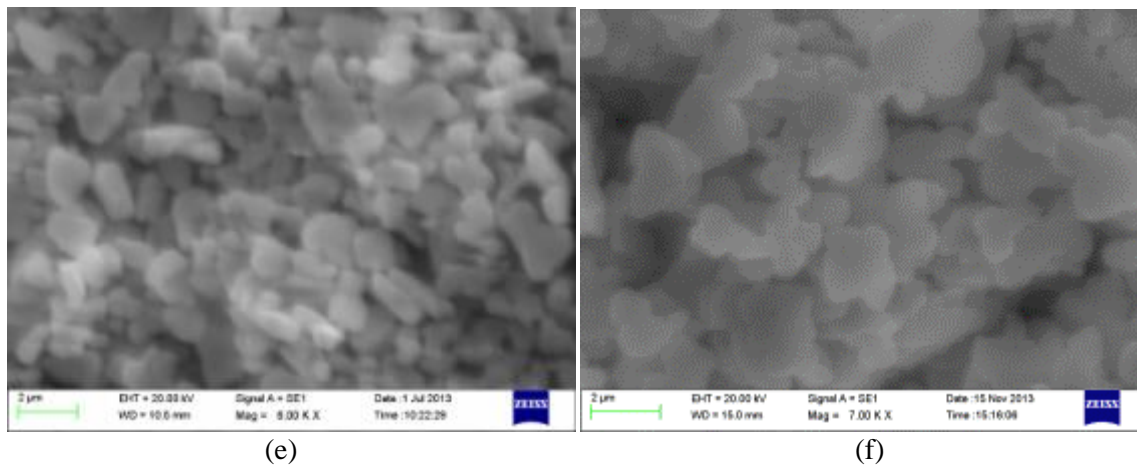


Figure 3. The SEM photographs of the silver powder with 1.5 μ m, 1 μ m and 0.6 μ m particle size milled for 8h (a), (b) and (c); 15h(d), (e) and (f).

3.3 XRD characterization of silver powder

It indicates that characteristic spectrum of SP was completely accordance with the standard crystal card of silver in the graph (Ag65-2871), and there are no other obvious impurity diffraction peaks (Figure 4). It is illustrated that SP had the property of high purity and low impurity content.

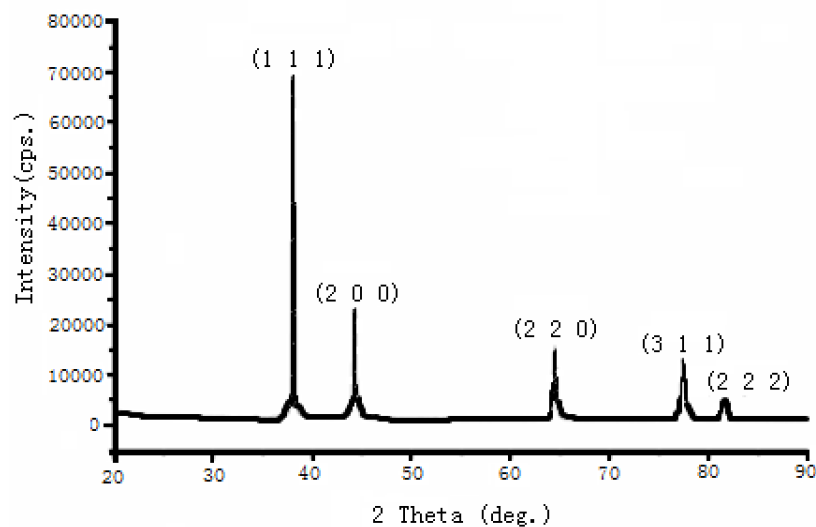


Figure 4. XRD pattern of silver particle

3.4 Thermo gravimetric analysis

The test result of thermogravimetric analyzer was shown in Figure 5. As seen, the quality loss of the flake silver powder was tiny during the process of heating-up, and the rate of slope of the differential of the heating-up curve was close to zero. It indicated that the amount of organic components adsorbed on the surface of SPs was very small. So, the flake silver powder was favorable to the preparation of conductive adhesive and the electrical performance of circuit.

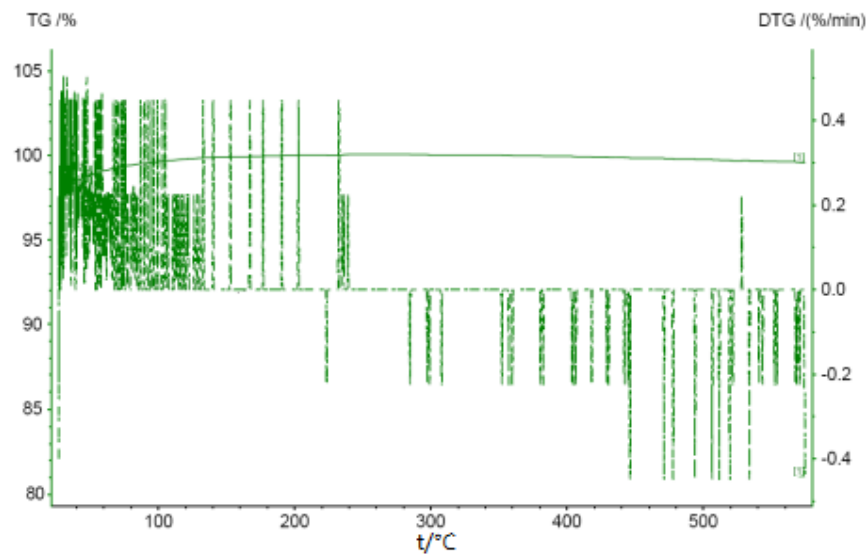


Figure 5. Thermogravimetric result of the flake silver powder

4. References

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Acknowledgments

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