PREPARATION OF SILVER POWDER FOR CONDUCTIVE PASTE①

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ABSTRACT

A novel techonology for preparing silver flake powder has been studied. The solution containing $KAg(CN)_2$ was reduced by suspension mixture of formaldehyde and benzaldehyde with surfactant TN, and luminous silver flake powder with an average particle size of 9.6μm was obtained. Furthermore, a silver paste was prepared using aforesaid novel flakes, whose bulk resistivity reaches 7.5×10^{-5} Ω•cm and shear strength 24.74 kg/cm².

Key words: suspension mixture $KAg(CN)_2$ luminous silver flake powder silver paste

1 INTRODUCTION

The silver paste made up of silver powder, resin, and organic solvent in portion is an important novel material in the electronic and electric industries. Many factors affect its properties, but silver powder is the most critical material in silver paste[1]. Three typical sorts of silver powder texture, the portion of the silver powder and resin etc. were studied. The properties of the silver paste comprised by silver flake powder are excellent and the paste is silver-white[2]. The process for producing silver flake powder directly has been reported abroad[3,4]. But in China, silver powders of dendritic or spheric shapes were produced first[5-8], then ground to flakes after a long time. The shape and texture of the silver powder produced by this way are inhomogeneous, and it is easy to be polluted. Therefore, the technology for preparing silver flake powder directly for conductive paste is described in the paper.

2 EXPERIMENTAL

The solution containing $KAg(CN)_2$ was heated to a certain temperature, then the ready reduction agent with surfactant TN was added. Hold-up time was 30 min, and pH value was kept about 6.5. After the reaction finished, phases, morphologies and average particle size of the product were analyzed. The three factors-three levels orthogonal experimental method ($L_9(3^4)$) was adopted. Primary factors affecting the reduction were investigated, e.g. total concentration of [Ag], volume ratio of formaldehyde and benzaldehyde, reaction temperature etc.

3 RESULTS AND DISCUSSION

The experimental conditions and results were listed in Table 1, and Figs. 1, 2.

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Table 1 The experimental conditions and results

Sample No	Total Conc of [Ag] (factor A)	Volume Ratio of Formal/ Benzal. (factor B)	Hold-up Temp(C) (factor C)	Product Phase	Product Topography	Average diameter /μm
1	0.1	1:1	30	Ag	granular	6.2
2	0.1	1:3	65	Ag	granular	2.9
3	0.1	1:9	100	Ag	granular	4.4
4	0.01	1:1	65	Ag	granular	4.0
5	0.01	1:3	100	Ag	flake	9.6
6	0.01	1:9	30	Ag+AgCN	fibre	24.4
7	0.001	1:1	100	Ag	granular	5.1
8	0.001	1:3	30	Ag	flake	9.7
9	0.001	1:9	65	Ag	flake	9.6

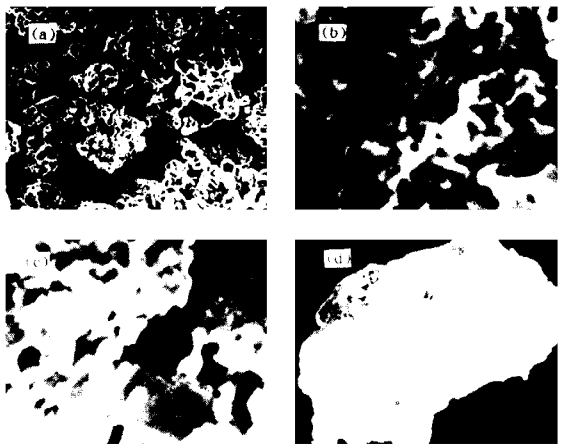

Fig. 1 SEM micrographs of silver powder
(a) Sample No. 4, ×3 000; (b Sample No. 5, ×1 000;
(c) Sample No. 8, ×2 000; (d) Sample No. 9, ×10 000

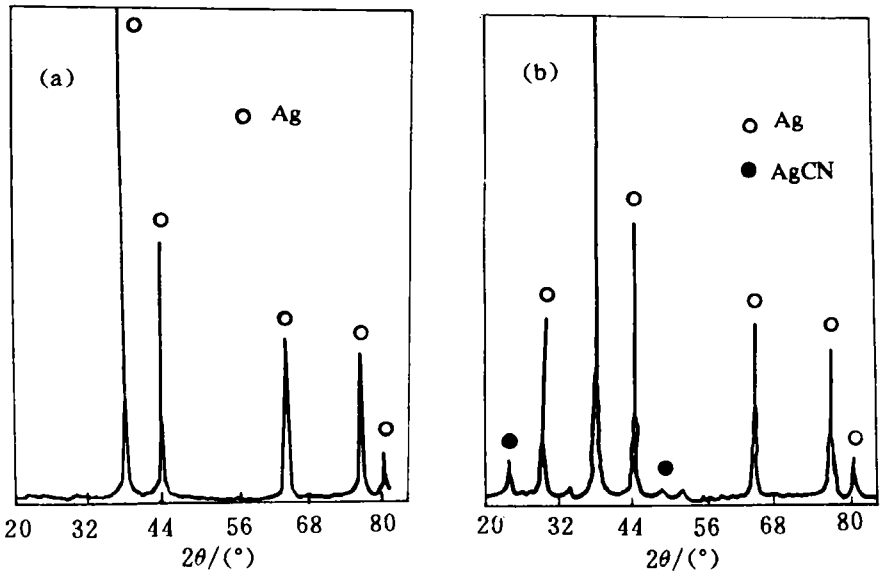

Fig. 2 X-Ray diffraction feature of reaction products
(a)—complete reaction; (b)—incomplete reaction

Table 1 shows that when the reaction conditions are controlled properly, $[Ag]_T$ is 10^{-3} mol/L, volume ratio of formaldehyde and benzaldehyde is 1 : 3 or 1 : 9, reaction temperature is 30 ℃ or 65 ℃; or $[Ag]_T$ is 10^{-2} mol/L, volume ratio of formaldehyde and benzaldehyde is 1 : 3, temperature is 100 ℃, silver flake powder with an average particle size of 9.6 μm can be prepared.

The average particle sizes of the reaction products have direct relation to the reaction rate. The faster the reaction rate is, the more crystal nuclei form. On the contrary, interproduct AgCN of coarse fibre texture forms. So the average effect of each factor each level on the reduction was investigated according to the average particle size.

It can be drawn from the above analyses that the average particle size of reduction products is affected first by the total concentration of [Ag]; second by the reaction temperature; and then by the volume ratio of the formaldehyde and benzaldehyde.

		Factor A	Factor B	Factor C
Average effect	Σ1/3	4.5	5.1	13.5
of each fact	Σ2/3	12.7	7.3	5.5
each level	Σ3/3	8.1	12.8	6.4
Maximum difference between levels		8.2	7.7	7.9
Influence order			A>C>B	

4 PREPARATON OF SILVER PASTE

The silver powders and resin solution were mixed completely in ratio of 95/5 to prepare silver pastes. The main parameters of the silver pastes coating, such as bulk resistivity and shear strength were measured according to the methods in Ref. [2]. The results were shown in Table 2.

As seen in Table 2, the silver paste prepared by the silver flake powder has the advantages of that prepared by spheric powder, and the bulk resistivity of the previous reaches 7.5×10^{-5} Ω · cm, the shear strength 24.74 kg/cm^2.

Table 2 The properties of the silver pastes

Sample No	Volume of Pastes /cm³	Total Resistance /$\times 10^{-2}\Omega$	Bulk Resistivity /$\times 10^{-4}\Omega$·cm	Cohesive Area of Pastes/cm²	Shear Force /kg	Shear Strength /kg·cm^{-2}	Luster of Pastes
4	2.0×0.5×0.1	2.720	6.80	1.00×0.55	9.54	17.34	Grey
5	2.0×0.5×0.1	0.332	0.83	1.00×0.55	13.61	24.74	Silver-white
8	2.5×0.5×0.1	1.050	2.10	1.10×0.60	14.10	21.36	Silver-grey
9	2.5×0.5×0.1	0.375	0.75	1.10×0.60	13.96	21.15	Silver-grey

5 CONCLUSIONS

(1) The $KAg(CN)_2$-bearing solution is reduced by suspension mixture of formaldehyde and benzaldehyde with surfactant TN. When the reaction conditions are controlled properly, the silver flake powder with an average particle size of 9.6 μm can be obtained directly.

(2) Orthogonal experimental results show that the reaction rate and reduction products are affected first by the total concentration of [Ag]; second by the reaction temperature and then by the volume ratio of the formaldehyde and benzaldehyde.

(3) The silver paste is prepared using aforesaid silver flake powder and its resistivity reaches $7.5\times10^{-5}\ \Omega$·cm, shear strength 24.74 kg/cm².

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