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Influence of Type of Process Control Agent on the Synthesis of Ag8ZnO Composite Powder

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In this study, the effect of different types of process control agents on the particle size and morphology of Ag8ZnO composite powder was investigated. For this purpose, commercial elemental silver and zinc oxide powders were initially mixed with different types of process control agents and then milled in a two stationary planetary-type ball mill with a ball-to-powder weight ratio of 10:1 and a milling speed of 300 rpm. The usage of process control agents is advantageous to eliminate or minimize agglomeration and to decrease the tendency of excessive cold welding among powder particles. However, contamination from the process control agent essentially leads to interstitial contamination, since the process control agents used are mostly organic compounds containing carbon, oxygen, and nitrogen. Hence, it is of importance to investigate the nature of PCA and milling duration that both influence the performance of electrical contacts. Characterization of starting and ball-milled powders was investigated using scanning electron microscopy and laser diffraction analysis. It was found that final powder particle sizes significantly affected by the nature of process control agent. Besides, morphology of powders and microstructural analyses prove that the type of process control agent is also crucial in obtaining homogeneous mixture of the constituent powder particles.

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PACS/topics: ball milling, composite powders, process control agent, silver-based electrical contact materials, zinc oxide

1. Introduction

Silver (Ag) is the most widely used material for electrical contacts of electromagnetic switches such as relays and contactors due to its highest electrical and thermal conductivities among all metals. However, Ag has also some drawbacks such as low melting and boiling points, low mechanical strength, possible contact welding and a tendency to form sulfide films. These undesired properties may adversely affect the arc-erosion performance of electrical contacts, and therefore some compositional arrangements and/or production methods must be performed to enhance both physical and mechanical properties. Due to the reasons mentioned above, it is necessary to form a composite material containing metal oxide in order to increase strength against arc-erosion and to minimize welding. Hence, different types of silver-based composite electrical contact materials have been developed in order to meet the requirements for various applications [1–3]. The most common metal oxides used in electrical contacts are cadmium oxide (CdO), tin dioxide (SnO_2) and zinc oxide (ZnO).

Powder processing technique is the first and one of the most important step of achieving a composite having superior properties. Following powder processing, some powder metallurgical parameters such as compaction pressure, sintering time and temperature are also crucial and they significantly affect the microstructure. In this content, ball-milling technique is used to produce composite powders having improved homogeneity with finer particle sizes. However, ball-milling technique has several parameters that needs to be optimized (such as ball-to-powder weight ratio (BPR), milling speed, type of process control agent (PCA), milling duration, etc.) to enhance ball milling process efficiency [4, 5]. PCAs are basically used to establish a balance between cold welding and fracturing events and the non-use of the PCAs leads excessive cold welding which inhibits further size reduction, especially in compositions containing a substantial fraction of ductile component. Much work has been done towards producing and evaluating the electrical performances of contact materials [6–15]. However, studies regarding the type of PCA on the characterization of composite powders containing noble metals, especially silver are very limited. For this aim, the present study investigated the effect of different types of PCAs, namely polyethylene glycol (PEG), stearic acid and zinc stearate, on the synthesis of Ag8ZnO composite powder.

2. Experimental procedures

In the present study, various types of PCAs, namely stearic acid, PEG and zinc stearate were added separately to powder mixtures containing commercial elemental Ag (maximum particle size of 20 μm, 99.99% purity) and ZnO (average particle size (APS) of 2 μm, 99.999% purity) powders. ZnO powders were added to Ag powders in the amount of 8 wt% whereas the percentage of PCA additives were 2 wt% for each type. Morphology of the starting or as-received powders is shown in Fig. 1.

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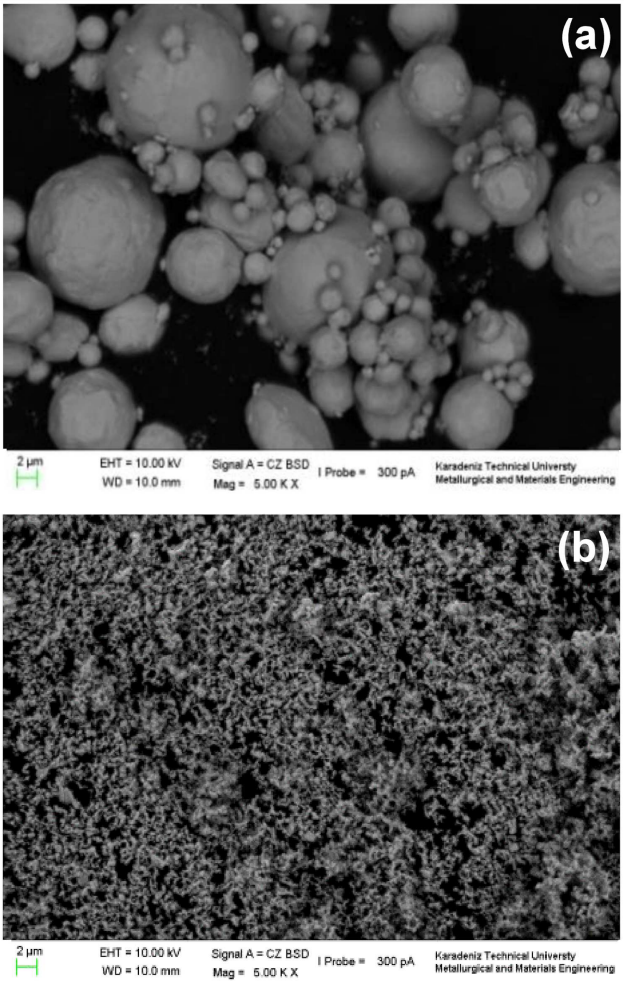

Fig. 1. Morphology of as-received a) Ag and b) ZnO powders.

Three different powder mixtures obtained by this way were then milled to synthesize composite powders using a two stationary planetary-type ball mill (Retsch PM 200) with a milling speed of 300 rpm and a BPR of 10:1. Both the milling container and the grinding balls are made of tungsten carbide. The diameter of the grinding balls Φ is 10 mm. Morphological evolution of powder mixtures as a function of PCA type and milling duration was investigated using scanning electron microscopy (SEM; Zeiss Evo LS 10). The powder samples were withdrawn from the milling bowls at certain milling runs to conduct particle size measurements using laser diffractometry (Mastersizer 2000, Malvern Instruments).

3. Results and discussion

Microscopic examination provides information on the morphology and particle size of both matrix (Ag) and reinforcement (ZnO) materials. It can be seen from Fig. 1a that Ag powder presents a spherical morphology whereas ZnO powder exhibits an irregular shape (Fig. 1b). Besides, ZnO powder is much smaller in particle size than that of Ag powder. Table I shows the APS values of three different powder compositions as a function of PCA type and milling duration. After each certain milling runs, namely 0.5, 2, 4, 10, 16 and 25 h, particle size variations of all compositions are recorded and the curves obtained by this way are presented in Fig. 2. The general trend in particle size of all compositions tends to slow down up to 4 h of milling duration. After this point, the efficiency of particle size reduction is very low for the specimen having PEG as an additive. On contrary, particle size is continuously decreased with increasing milling duration for the specimens having stearic acid and zinc stearate as additives. It can be seen from Fig. 2 that the rate of particle size reduction slows down after the milling duration of 16 h for the specimens having stearic acid and zinc stearate. After completion of the ball-milling experiments, namely the milling duration of 25 h, the Ag8ZnO composite containing zinc stearate as an additive possesses minimum particle size (6.217 μm).

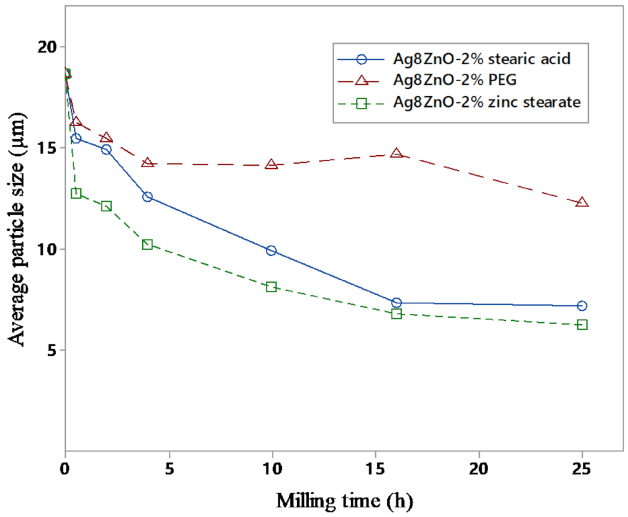

Fig. 2. Particle size variation of Ag8ZnO powder mixtures as a function of PCA type and milling duration.

Average particle size values with respect to PCA type and milling duration. TABLE I

Composition	Milling time [h] and average particle size d_{50} [μm]						
	0	0.5	2	4	10	16	25
Ag8ZnO-2% stearic acid	18.646	15.479	14.871	12.594	9.938	7.327	7.215
Ag8ZnO-2% PEG	18.646	16.245	15.435	14.234	14.128	14.700	12.263
Ag8ZnO-2% zinc stearate	18.646	12.679	12.055	10.241	8.149	6.786	6.217

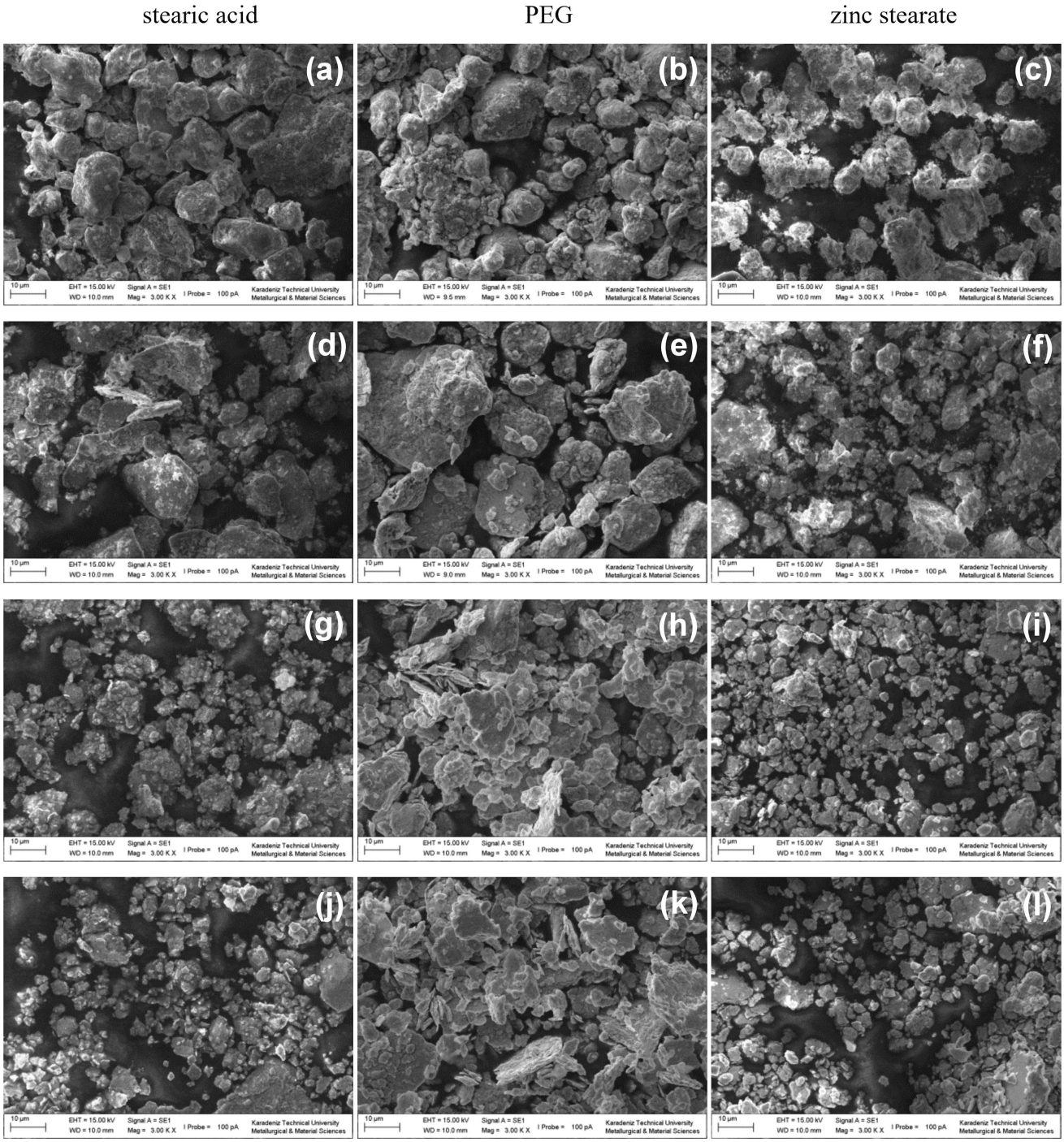

Fig. 3. Morphological evolution of Ag8ZnO powder mixtures containing different types of PCAs after milled for various milling durations; (a,b,c) 0.5 h, (d,e,f) 4 h, (g,h,i) 16 h and (j,k,l) 25 h.

Figure 3 shows the morphological evolution of Ag8ZnO powder mixtures containing different types of PCAs after milled for various milling durations, namely 0.5, 4, 16 and 25 h. It may be emphasized that the powder particles have a range of sizes and that most of the particles have flaky and irregular morphology in the early stages of ball milling process, especially for the specimens having stearic acid (Fig. 3a) and PEG (Fig. 3b) as additives. However, the Ag8ZnO-zinc stearate powder mixture exhibited a rapid decrease in particle size (12.679 μm) after milled for 0.5 h (Fig. 3c).

Powder mixtures having stearic acid (Fig. 3d and g) and zinc stearate (Fig. 3f and i) allow fracturing takes place more easily and effectively to produce finer particles, especially at certain values corresponding to 4–16 h of milling duration. Therefore, the most efficient particle

size reduction is observed at milling durations ranging between 4 to 16 h for the specimens having stearic acid and zinc stearate as additives. On contrary, efficiency of particle size reduction is very low for the specimen having PEG as an additive in the same milling interval (Fig. 3e and h).

The later stages of ball milling process are characterized by a narrow particle size distribution and the compositions, especially having stearic acid and zinc stearate as additives, becoming almost uniform after the milling duration of 25 h. Fracturing of powder particles of the specimen having PEG as an additive starts after the milling duration of 16 h, and powder particle size is reduced to 12.263 μm after completion of milling runs (Fig. 3k). It is clear from Figs. 2 and 3 that the powder mixtures having stearic acid (Fig. 3j) and zinc stearate (Fig. 3l) exhibit almost same regime in milling durations between 16 to 25 h which remains almost stable in powder particle sizes; and therefore steady-state condition is reached for both compositions. As a result of narrow size distribution, the composition having zinc stearate was exhibited equiaxed powder morphology with an APS of 6.217 μm.

4. Conclusions

Powder particle size generally decreases with increasing milling duration for all types of compositions. The efficiency of particle size reduction is very low for the specimen having PEG as an additive. However, powder mixtures having stearic acid and zinc stearate allow fracturing takes place more easily and effectively to produce finer particles. By comparing all compositions, the usage of zinc stearate as a PCA was found to be much more effective to obtain a composite powder having smaller particle sizes. Steady-state condition is reached for the powder mixtures having stearic acid and zinc stearate as both compositions exhibit almost same regime in milling durations between 16 to 25 h which remains almost stable in powder particle sizes. As a result of narrow size distribution, the composition having zinc stearate was exhibited equiaxed powder morphology with an APS of 6.217 μm. Therefore, the optimal condition for synthesizing Ag8ZnO composite powder was determined to be 25 h of milling duration with the usage of zinc stearate as a PCA.

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