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Effect of Particle Size of Second Phase on Wettability and Electrical Contact Properties of AgSnO₂ Contact Materials

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Abstract: TiO_2 was selected as the additive of the contact material, and $AgSnO_2$ and $AgSnO_2/TiO_2$ contact materials with six different particle sizes of second phase (SnO_2) were prepared by powder metallurgy. The wettability and electrical contact properties of both contact materials were studied. The wetting angle between Ag and SnO_2 was measured by the sessile drop method, and the wettability was characterized. The JF04C electrical contact material testing system was used to test the electrical contact properties, and the change laws of wettability and electrical contact performance of contact materials with different particle sizes of second phase were analyzed. The results show that the properties of the two contact materials with the particle size of second phase ranging in 100~300 nm are better than those with other particle sizes.

Key words: particle size of second phase; wettability; contact resistance; arcing energy; material loss

The electrical contact is the key component of switching apparatus and is responsible for connection and disconnection of circuit. The performance of electrical contact material is directly related to the electrical life and reliability of electrical equipment^[1]. Faults usually occur in the contact area due to the high operating frequency of contacts, so it is necessary to study the properties of contact material. The eco-friendly AgSnO₂ material has excellent resistance to arc erosion and fusion welding is very suitable for contact material^[2,3]. However, AgSnO₂ contact material has two main disadvantages. (1) SnO₂ particles are easily diffused to the contact surface for forming an enriched area under high temperature arc. The contact resistance is increased due to the formation of enriched area, and the temperature is increased accordingly, which seriously affects the performance of the contact material^[4]. (2) The processing and forming are difficult to proceed due to the poor ductility and plasticity of AgSnO₂ contact material^[5], which restricts its application. The poor wettability between molten Ag and SnO₂ is the main reason for these defects^[6,7]. Hence, the wettability has a significant effect on the performance of AgSnO₂ contact material.

The preparation process was modified^[8-10] and different additives were selected^[11-13] in order to overcome these defects. In addition, some researchers optimized the properties of contact materials by changing the particle size of powders^[14,15]. The AgSnO₂ contact material consists of metal matrix and the second phase of dispersed SnO₂ particles. The particle size of the powder affects the interaction and distribution of components. The second phase SnO₂ accounts for a large proportion in the original powders, so its particle size inevitably affects the performance of the contact material. Zhang et al^[16] studied the influence of SnO₂ particle size on the arc erosion behavior of AgSnO2 contact material and found that the arc duration and mass loss of AgSnO₂ contact material are decreased with decreasing the particle size. Lin et al^[17] revealed that the SnO₂ particle size has a significant influence on the morphology of the AgSnO₂ composite powders, which also affects the microstructure and physical properties of the electrical contact materials. Chen et al^[18] studied the effects of SnO₂ particle size on microstructure,

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density, mechanical properties, and electrical properties of $AgSnO_2$ material. However, the effect of the particle size of second phase SnO_2 on the wettability of $AgSnO_2$ contact materials is rarely studied.

In this research, the influence of the second phase with different particle sizes on wettability and electrical contact performance of AgSnO₂ contact material was discussed, which is of great significance to the research and production of AgSnO₂ contact material.

1 Experiment

 TiO_2 was selected as the additive of the AgSnO₂ contact material. The SnO₂ particles with size of 50, 100, 300, 500, 800, and 1000 nm were selected to prepare specimens. The SnO₂ and SnO₂/TiO₂ substrates were prepared, and the mass ratio of SnO₂: TiO₂=11: 1 was selected for SnO₂/TiO₂ substrates. The AgSnO₂ and AgSnO₂/TiO₂ contact materials were prepared according to the mass ratio of Ag: SnO₂=88: 12 and Ag:SnO₂:TiO₂=88:11:1, respectively.

The specimens were prepared by powder metallurgy. To avoid the moisture influence on the powders, the composite powders were heated at 120 °C under 0.08 MPa in the vacuum drying oven for 30 min. The dried powders were mixed in the Simoloyer mill for 2 h with a mixing speed of 500 r/min. Finally, the mixed powders were processed and formed by pressing, sintering, and polishing.

The SnO_2 substrates used for wettability test only underwent one time of pressing and sintering processes. These mixed powders were compacted under the pressure of 10 MPa for 5 min in the mould, and then sintered in the vacuum sintering furnace at 960 °C for 90 min. The AgSnO₂ contact materials used for electrical contact performance test underwent two times of pressing and sintering processes. These mixed powders were compacted under the pressure of 38 MPa for 5 min, then sintered at 500 °C for 90 min, repressed under the pressure of 58 MPa for 5 min, and resintered at 800 °C for 90 min. Finally, the AgSnO₂ contact materials were cut into contact terminals with 4.5 mm in diameter and 3.5 mm in thickness.

The wetting angle between Ag and SnO₂ or SnO₂/TiO₂ was measured by the sessile drop method. The Ag powder was placed on the center of substrates in a heating device. The mass of Ag powder was less than 0.3 g in order to reduce the gravity influence. Because the melting point of Ag is 961.93 °C, the Ag powder and substrates were heated at 1050 °C for 30 min to make the molten Ag fully spread on the surface of SnO₂ substrate. During the heating process, the protective gas N₂ was introduced to prevent the occurrence of oxides.

The JF04C electrical contact material testing system was used to test electrical contact performance of $AgSnO_2$ and $AgSnO_2/TiO_2$ contact materials, and real-time data including contact resistance and arcing energy were collected. For each cycle of test, the instrument must be readjusted: the direct current (DC) was 13 A, the voltage of DC was 24 V, and the system protection voltage was ± 40 V. After the test started, the contact pressure was adjusted to 86 cN. The whole test was

conducted at room temperature, and the related parameters are shown in Table 1.

2 Results and Discussion

2.1 Wetting angle

Wettability is one of the important characteristics of solid material surface, which is usually characterized by wetting angle^[19,20]. Young's equation indicates that the smaller the wetting angle, the better the wetting ability of the material. The wettability between the liquid and solid phases directly affects the crystal growth, phase distribution, and bonding strength of the composite powders in powder metallurgy method. The wettability of specimens was measured by the wetting angle measuring device, as shown in Fig.1 and Fig.2.

The wetting angle was automatically acquired by the measuring device. However, some burrs are formed on the surface of Ag particles after melting, as shown in Fig. 1d. In this case, the wetting angle image was processed by CAD software and then the wetting angle could be obtained, as shown in Fig. 3. The obtained wetting angle is the average of contact angles on both sides.

 Table 1
 Related parameters of electrical contact material testing system

Parameter	Value
Number of trials	25 000
Voltage/V	24
Current/A	13
Power type	DC
Electrical contact test method	Mechanical breaking
Contact resistance range/ Ω	10-5~20
Contact pressure/cN	86
Contact gap/mm	2
Contact form	Face contact



Fig.1 Wetting angle images between Ag and SnO₂ substrates with different SnO₂ particle sizes: (a) 50 nm, (b) 100 nm, (c) 300 nm, (d) 500 nm, (e) 800 nm, and (f) 1000 nm



Fig.2 Wetting angle images between Ag and SnO₂/TiO₂ substrates with different SnO₂ particle sizes: (a) 50 nm, (b) 100 nm, (c) 300 nm, (d) 500 nm, (e) 800 nm, and (f) 1000 nm



Fig.3 Schematic diagram of wetting angle measurement of specimens with burrs

The wetting angles of specimens are shown in Fig.4. It can be seen from that the wetting angles between Ag and SnO_2 or SnO_2/TiO_2 substrates with different SnO_2 particle sizes are different.

Fig. 4 shows that the wetting angle between Ag and SnO₂ substrates is decreased firstly, then increased, and finally decreased with increasing the SnO₂ particle size. The minimum wetting angle is 74.1° with the particle size of 1000 nm. The maximum wetting angle is 87.2° with the particle size of 800 nm. After TiO₂ addition, the wetting angle is increased with increasing the particle size to 800 nm, and decreased with further increasing the particle size to 1000 nm. The optimal wettability with the minimum wetting angle of 70.3° is achieved when the SnO₂ particle size is 50 nm. When the SnO₂ particle size is 800 nm, the maximum wetting angle of 83.9° can be observed. In general, the wetting angle of



Fig.4 Wetting angles between Ag and SnO₂ or SnO₂/TiO₂ substrates with different SnO₂ particle sizes

specimens with TiO_2 additive is smaller than that of specimens without TiO_2 additive, which indicates that the additive can improve the wettability of contact materials.

As shown in Fig.4, the wetting angle between Ag and SnO₂ substrate is decreased with increasing the particle size from 50 nm to 100 nm, and that between Ag and either SnO_2 or $SnO_2/$ TiO, substrate is also decreased with increasing the particle size from 800 nm to 1000 nm. The wetting angle between Ag and SnO₂ substrate with SnO₂ particle size of 50 nm is larger, because these SnO₂ particles are relatively small, which are prone to agglomeration. With increasing the particle size, the specific surface is decreased, which significantly reduces the surface energy of solid phase SnO₂. Thus, the wettability between the molten Ag and the solid SnO₂ substrate becomes worse. The TiO₂ additive in the SnO₂/TiO₂ substrate changes the performance of second phase SnO₂ and improves the wettability between Ag and SnO₂, so the wetting angle between Ag and SnO₂/TiO₂ substrate with the SnO₂ particle size of 50 nm is smaller. When the particle size reaches 1000 nm, the agglomeration of particles is less, which is beneficial to the wettability between Ag and substrates, so the wetting angle is greatly reduced.

2.2 Contact resistance

The range, average value, and variance of contact resistance of $AgSnO_2$ and $AgSnO_2/TiO_2$ contact materials are shown in Table 2.

It can be seen from Table 2 that as the particle size of second phase increases, the average contact resistance of $AgSnO_2$ contact material is increased accordingly. When the

 Table 2
 Contact resistance of AgSnO2 and AgSnO2/TiO2 contact materials

Particle size/nm		$AgSnO_2$			$AgSnO_2/TiO_2$	
	Range/mΩ	Average/mΩ	Variance/m Ω^2	Range/mΩ	Average/mΩ	Variance/m Ω^2
50	0.62~3.28	1.11	0.224	1.85~10.97	3.72	1.305
100	0.54~4.73	1.18	0.291	0.70~4.15	2.09	0.815
300	0.56~6.66	1.50	0.670	1.48~2.79	2.05	0.085
500	1.00~3.38	1.57	0.175	1.14~6.34	2.38	0.395
800	1.36~3.89	2.11	0.126	1.57~5.16	2.52	0.255
1000	0.88~5.74	2.10	0.660	0.92~9.76	1.90	0.783

particle size is 50 nm, the average contact resistance reaches the minimum value of $1.11 \text{ m}\Omega$. The change trends of average contact resistance and wetting angle are opposite when the SnO₂ particle size of AgSnO₂ specimen increases from 50 nm to 100 nm. As for the AgSnO₂ specimens with SnO₂ particle size of 100~1000 nm, the change laws of average contact resistance and wetting angle are similar, showing the firstly increasing and then decreasing trend. Under the high temperature arc, the molten pool is formed on the contact surface, and SnO₂ with poor conductivity is dispersed to the surface, resulting in the increase in contact resistance of contact material^[21].

Compared with that of $AgSnO_2$ contact material, the average contact resistance of $AgSnO_2/TiO_2$ contact material is relatively large. When the SnO_2 particle size is 50 nm, the average contact resistance reaches the maximum value of 3.72m Ω . As for the $AgSnO_2/TiO_2$ specimens with SnO_2 particle size of 300~800 nm, the average contact resistance is basically increased with increasing the particle size. When the particle size is 1000 nm, the average contact resistance is decreased significantly, reaching the minimum value of 1.90 m Ω , which indicates that the additive has a positive effect on the contact resistance of $AgSnO_2$ contact material.

In general, when the SnO_2 particle size is greater than 100 nm, the change trends of average contact resistance and wetting angle of $\text{AgSnO}_2/\text{TiO}_2$ contact materials are similar. When the SnO_2 particle size is 50 nm, the wettability is good but the average contact resistance is large. It is reported that the composition of the contact surface may change during the arc erosion, which affects the contact resistance^[6]. When the particle size of second phase SnO_2 is 50 nm, TiO_2 particles with relatively high resistivity (compared with that of Ag and SnO_2) are precipitated and accumulated on the contact surface during the test, which leads to the increase in contact resistance.

2.3 Arcing energy

In practical application, arc erosion is the main factor causing the erosion of electrical contact materials. Due to the multiple effects of high temperature arc, the surface of electrical contact materials is damaged, the contact resistance is increased, and thereby the temperature is further increased, resulting in a vicious circle which eventually leads to the failure of electrical contact materials. The arcing energy is an important parameter in the process of electrical contacting. The smaller the arcing energy, the less the burning degree of surface and the better the electrical life as well as reliability of electrical contact materials.

The range of arcing energy, average arcing energy, and variance of arcing energy of $AgSnO_2$ and $AgSnO_2/TiO_2$ contact materials are shown in Table 3.

With increasing the particle size of second phase, the average arcing energy of $AgSnO_2$ contact material is decreased firstly, then increased, and finally decreased. When the particle size is 300 nm, the average arcing energy reaches the minimum. The change trend of average arcing energy is similar to that of wetting angle with only one exception that the minimum value is at the particle size of 100 nm for wetting angle, while that is at the particle size of 300 nm for arcing energy.

The average arcing energy of AgSnO₂/TiO₂ contact material is decreased firstly and then increased with increasing the particle size. When the particle size is 100 nm, the average arcing energy achieves the minimum value of 172.81 mJ. The variance at this moment is relatively small, suggesting that the stability of AgSnO₂/TiO₂ contact material with particle size of 100 nm is relatively good, and its arcing characteristics are better than those of AgSnO₂/TiO₂ contact materials with other particle sizes. It is clear that when the particle size is 50~100 nm and 800~1000 nm, the change trends of wetting angle and average arcing energy are different. When the particle size is 100~800 nm, the change trends of wetting angle and average arcing energy are similar, i.e., with increasing the particle size from 100 nm to 800 nm, the wetting angle is increased, thereby the wettability of AgSnO₂/TiO₂ contact material becomes worse, and the arc erosion on the contact surface becomes more serious, resulting in a larger arcing energy^[22].

2.4 Material loss

The mass of electrical contact materials was measured before and after the electrical contact performance tests, and the mass change and material loss of the two contact materials are shown in Table 4.

It is clear that with increasing the particle size, the material loss of the two contact materials is decreased firstly, then increased, and finally decreased. The minimum material loss of AgSnO₂ contact material is 0.14 mg when the particle size is 100 nm; while the maximum material loss is 0.59 mg when the particle size is 800 nm. The minimum material loss of AgSnO₂/TiO₂ contact material is 0.07 mg when the particle size is 100 nm; while the maximum material loss of AgSnO₂/TiO₂ contact material is 0.07 mg when the particle size is 100 nm; while the maximum material loss is 1.56 mg

Particle size/nm	AgSnO ₂			AgSnO ₂ /TiO ₂		
	Range/mJ	Average/mJ	Variance/mJ ²	Range/mJ	Average/mJ	Variance/mJ ²
50	165.73~177.42	171.30	3.93	124.81~230.79	193.04	226.78
100	160.60~181.29	170.45	14.71	132.20~182.82	172.81	103.31
300	149.64~178.68	157.75	19.37	143.01~187.71	174.14	90.64
500	156.46~180.12	166.65	17.43	144.67~190.75	175.10	116.73
800	165.18~181.37	172.56	8.19	150.67~180.03	176.85	88.13
1000	156.41~182.58	165.72	13.65	144.27~199.68	182.65	120.01

Table 3 Arcing energy of AgSnO₂ and AgSnO₂/TiO₂ contact materials

Particle size/nm $AgSnO_2$ $AgSnO_2/TiO_2$ Anode mass changeCathode mass changeMaterial lossAnode mass changeCathode mass changeMaterial loss50 $+0.10$ -0.59 -0.49 $+0.07$ -1.63 -1.56			0	0 1		(B)	
Faiture size/initAnode mass changeCathode mass changeMaterial lossAnode mass changeCathode mass changeMaterial loss50 $+0.10$ -0.59 -0.49 $+0.07$ -1.63 -1.56	Particle size/nm	AgSnO ₂			AgSnO ₂ /TiO ₂		
50 + 0.10 - 0.59 - 0.49 + 0.07 - 1.63 - 1.56		Anode mass change	Cathode mass change	Material loss	Anode mass change	Cathode mass change	Material loss
50 (0.10 0.5) 0.7) (0.07 1.05 1.50	50	+0.10	-0.59	-0.49	+0.07	-1.63	-1.56
100 +0.14 -0.28 -0.14 +0.13 -0.20 -0.07	100	+0.14	-0.28	-0.14	+0.13	-0.20	-0.07
300 +0.12 -0.40 -0.28 +0.89 -0.98 -0.09	300	+0.12	-0.40	-0.28	+0.89	-0.98	-0.09
500 +0.09 -0.53 -0.44 +0.10 -0.20 -0.10	500	+0.09	-0.53	-0.44	+0.10	-0.20	-0.10
800 +0.03 -0.62 -0.59 +0.17 -0.46 -0.29	800	+0.03	-0.62	-0.59	+0.17	-0.46	-0.29
1000 +0.18 -0.37 -0.19 +0.15 -0.34 -0.19	1000	+0.18	-0.37	-0.19	+0.15	-0.34	-0.19

Table 4 Mass change and material loss of AgSnO, and AgSnO₂/TiO, contact materials (mg)

when the particle size is 50 nm. In general, the material loss of $AgSnO_2/TiO_2$ contact material is less than that of $AgSnO_2$ contact material, indicating that the TiO_2 additive improves the erosion resistance of $AgSnO_2$ contact material.

Based on these analyses, it can be found that the change trend of material loss of $AgSnO_2$ contact material is generally consistent with that of wetting angle of $AgSnO_2$ contact material. When the particle size of second phase of $AgSnO_2/TiO_2$ contact material is greater than 100 nm, the change trend of material loss is similar to that of wetting angle of $AgSnO_2/TiO_2$ contact material.

3 Conclusions

1) With increasing the SnO_2 particle size, the wetting angle of $AgSnO_2$ and $AgSnO_2/TiO_2$ contact materials is increased firstly and then decreased. The TiO_2 additive can optimize the wettability. When the particle size is 50 nm, the minimum wetting angle between Ag and SnO_2/TiO_2 substrate of 70.3° can be obtained, indicating that the optimal wettability is achieved.

2) The electrical contact properties and material loss of $AgSnO_2$ and $AgSnO_2/TiO_2$ contact materials are fluctuating. In general, the contact resistance, arcing energy, and material loss of the two contact materials are better with the SnO_2 particle size of 100~300 nm, and are relatively stable. Therefore, the overall performance of the two contact materials is better when the SnO_2 particle size is 100~300 nm.

3) When the particle size of second phase is $100 \sim 800$ nm, the change trend of contact resistance, arcing energy, and material loss is similar to that of wetting angle. All these characteristics are increased with increasing the SnO₂ particle size. However, when the particle size is $50 \sim 100$ nm and $800 \sim 1000$ nm, the change trend of these characteristics tends to be different.

4) The contact materials with particle size below 100 nm and over 1000 nm have quite different properties which should be further investigated.

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第二相粒度对AgSnO₂触头材料润湿性和电接触性能的影响

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摘 要:选用TiO₂作为触头材料的添加剂,采用粉末冶金法制备了6种不同第二相(SnO₂)粒度的AgSnO₂和AgSnO₂/TiO₂触头材料。 研究了2种触头材料的润湿性和电接触性能。用座滴法测量了Ag与SnO₂之间的润湿角,并表征了材料的湿润性,使用JF04C电接触触 头材料测试系统对材料电接触性能进行了测试,分析了不同第二相粒度的触头材料的润湿性和电接触性能的变化规律。结果表明,当第 二相粒度为100~300 nm时,2种触头材料的性能均优于其它粒度的触头材料。 关键词:第二相粒度;润湿性;接触电阻;燃弧能量;材料损失

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