$AgSnO_2$ Electrical Contact Material Prepared by Spark Plasma Sintering

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Abstract: Ag-10%SnO_2 electrical contact material was prepared by spark plasma sintering. The microstructures, fracture appearances, arc erosion surfaces and sintering process of the material were investigated. The experimental results showed that there are three basic stages in the spark plasma sintering process. During the sintering process, the shape and size of each particle changed with the formation of grain boundaries. The SnO_2 particles distribute diffusely in Ag matrix, and there is no particles gathering together in the matrix. The Ag-10%SnO_2 material thus prepared could keep the grains fine and has high strength and good ductility due to fine grain strengthening. The tensile fracture morphology of the material heated by SPS at 800℃for 5 min shows a great number of large and deep dimples, in which the oxide particles exist. The fracture mode of the Ag-10%SnO_2 material is the microporous polycondensation plastic fracture. The arc erosion surfaces of the material show a large number of paste-like coagulum and bubbles.

Key words: metal material; Ag-10%SnO_2 material; spark plasma sintering; microstructure; sintering process
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用放电等离子烧结技术制备 $AgSnO_2$ 电接触材料

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摘 要：采用放电等离子烧结(SPS)技术制备了 Ag-10%SnO_2 电接触材料。对材料的微观结构，断口形貌，电弧侵蚀表面形貌和烧结过程进行了研究。实验结果表明，Ag-10%SnO_2 电接触材料的 SPS 过程由 3 个基本阶段构成。在 SPS 过程中，颗粒的形状和尺寸随晶界的形成而发生变化。所得材料显微组织显示，SnO_2 颗粒弥散分布于 Ag 基体中，无颗粒聚集现象。SPS 技术烧结的 Ag-10%SnO_2 材料由于晶粒细小而具有较高的强度与较好的塑性。在 800 ℃/5 min 的烧结条件下，材料的拉伸断口形貌出现较多大而深的韧窝，韧窝中包含氧化物颗粒。Ag-10%SnO_2 电接触材料的断裂模式为微孔缩聚塑性断裂。材料的电弧侵蚀表面显示出大量的浆糊状凝固物和气泡。

关键词：金属材料；Ag-10%SnO_2 电接触材料；放电等离子烧结；微观结构；烧结过程

Silver-tin oxide materials have acceptable arc resistance, sufficient safety with respect to contact welding, comparable low material migration with low contact resistance and good over temperature behavior, and practical processing and jointing properties. The material has been considered to be best substitute for traditional AgCdO material[1-4]. Ag-10%SnO_2 material is the focus of researches in contact materials in recent

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years. It has got a rapid development and has gradually been applied to current contactors, power relays and low-voltage circuit breakers[5-10]. Many researchers have been interested in how to fabricate the Ag-10%SnO_2 electrical contact material more efficiently, including powder metallurgy, internal oxidation and hot pressing[11-15]. Recently, it was reported that the spark plasma sintering (SPS) technology can prepare the electrical contact material successfully and efficiently[16-18]. In present study, the authors attempted to obtain Ag-10%SnO_2 electrical contact material by SPS. The microstructures, sintering process, fracture appearances and arc erosion surfaces of the material were comparatively investigated.

1 Experiment

Ag and SnO_2 powders with purity of more than 99.9% were used as the raw material in the experiment. Fig.1 shows the SEM images of Ag and SnO_2 powders. Ag powders are sphere-like and SnO_2 powders are porous sponge-like. Ag and SnO_2 powders were mixed with a certain mass ratio (Ag/SnO_2=90/10), and then sintered into blocks of ∅15 mm by SPS-515S system at 800℃ for 5 min under 40 MPa. Microstructure observations were carried out by Leica DM4000M optical light microscope and Hitachi S-3400N scanning electron microscope. The arc erosion experiment was held by JF04C.

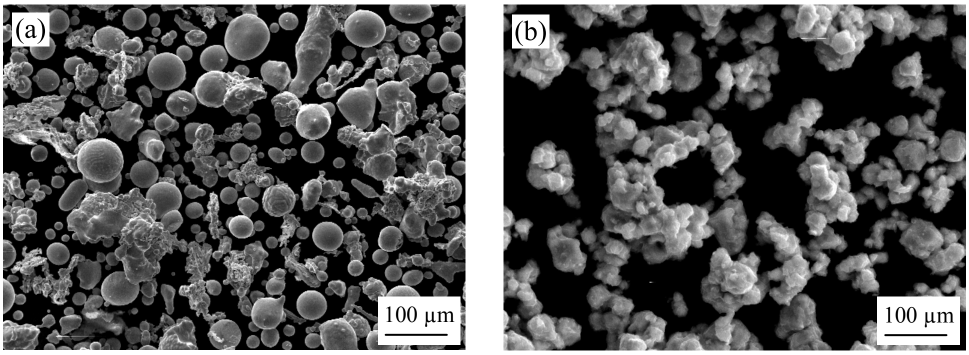

Fig.1 SEM images of the raw powders for Ag (a) and SnO_2 (b)

图 1 银粉(a)和 SnO_2 (b)原始粉末的扫描电镜照片

The arc erosion experimental parameters are showed in Tab.1. The tensile test was performed by AG-IC10KV tester at room temperature with a crosshead speed of 2 mm/min.

Tab.1 Parameters of the arc erosion experiment

表 1 电弧侵蚀实验参数

Variables	Magnitude
Voltage /V	17
Current/A	15
Distance between the contacts/mm	1
Closure pressure /cN	70
Contact frequency/(times/min)	60
Number of operation	10000

2 Results and discussion

2.1 Microstructure and physical properties of $AgSnO_2$ material

SEM images of Ag-10%SnO_2 material prepared by spark plasma sintering at 800℃ for 8 min are shown in Fig.2. The dark and gray parts are SnO_2 particles and Ag matrix, respectively. The SnO_2 particles exist diffusely in Ag matrix and there is no particles gather together in the matrix. It can be concluded that due to the rapid sintering of SPS process, the original status of the material is maintained and the particles do not grow up excessively. The binding between Ag phase and SnO_2 phase is very strong.

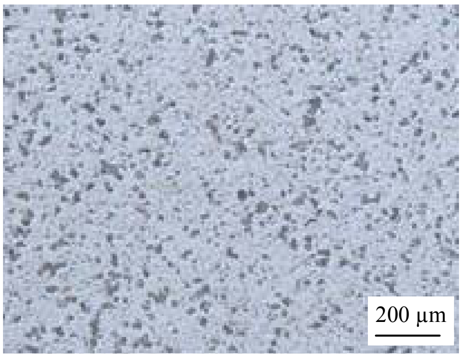

Fig.2 Microstructure of Ag-10%SnO_2 material

图 2 Ag-10%SnO_2 材料的微观组织

Tab.2 shows the physical properties of the Ag-10%SnO_2 material by SPS at 800℃ for 5 min under 40 MPa. As we can see form Tab.2, the tensile strength, elongation, density, resistivity, hardness of the Ag- 10%SnO_2 are 287 MPa, 21%, 9.85 g·cm^{-3}, 2.21 μΩ·cm, HV 90, respectively. Due to the advantages of rapid sintering, short sintering time and fast cooling of SPS technology, the Ag-10%SnO_2 material could keep the fine grains and obtain high strength and good ductility by fine grain strengthening.

Tab.2 Physical properties of Ag-10%SnO_2 material by SPS

表 2 放电等离子烧结的 Ag-10%SnO_2 材料的物理性能

Physical properties	Magnitude
Tensile strength/MPa	287
Elongation/%	21
Density/(g·cm^{-3})	9.85
Resistivity/(μΩ·cm)	2.21
Hardness/HV	90

2.2 Spark plasma sintering process

Sintering is a fundamental process to densify the Ag and SnO_2 powders. During the sintering process, the shape and size of each particle change with the formation of grain boundaries. This change is accompanied by the change of shape, size and fraction of pores in the Ag matrix. Images of the SPS process of AgSnO_2 material were shown in Fig.3.

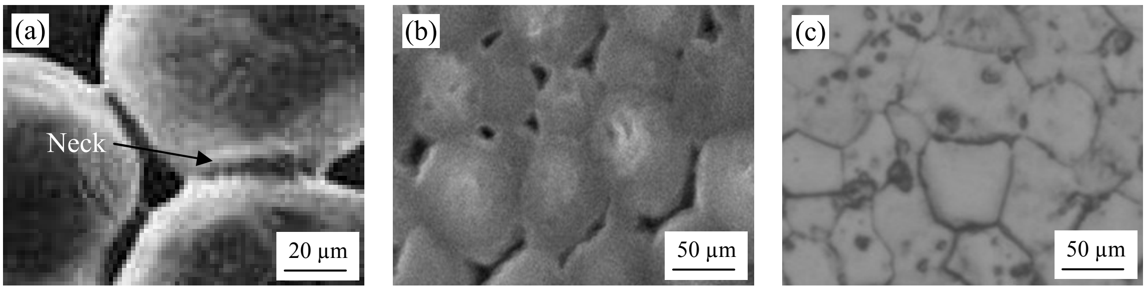

Fig.3 Images of the SPS process of AgSnO_2 material

[(a). initial stage; (b). intermediate stage; (c). final stage]

图 3 AgSnO_2 材料的放电等离子烧结过程照片

[(a). 初始阶段；(b). 中间阶段；(c). 终了阶段]

It can be found from Fig.3 that there are three basic stages in the process of the spark plasma sintering of AgSnO_2 material. As showed in Fig.3 (a), in the initial stage**,** material is transported from higher-energy convex particle surfaces to the lower-energy, concave intersections between adjacent particles to form necks. The powder particles of AgSnO_2 material fuse together and the area of contact gradually increases. Mass is only transported from the convex to the concave areas and the relative density is about 40%~50%. As showed in Fig.3 (b), in the intermediate stage, the inter-particle necks grow. The area of grain boundaries increase, the pore diameters decrease, and the relative density increases to about 70%~85%. As showed in Fig.3 (c), in the final stage pores are gradually eliminated and the relative density further increases. The relative density is about 99%. In present study, we obtained AgSnO_2 material with the relative density of 99.7% by SPS at 800℃ for 8 min under 40 MPa.

2.3 Fracture appearances

Fig.4 shows the tensile fracture appearances of the

$AgSnO_2$ material sintered by SPS at 800℃ for 5 min under 40 MPa. The tensile fracture morphology shows a great number of large and deep dimples. It is illustrated that the severe plastic deformation occurs before breaking failure of the material. Plastic deformation takes place first at the crack tip and spreads across the solid as the applied load is increased beyond general yielding. It can be also seen from Fig.4 (b) that the particles are occasionally separated and pulled out, and the tear ridges leave behind in the large and deep dimples. This deformation feature demonstrates that the SnO_2 particles combine firmly with the Ag matrix. The fracture mode of the material is the microporous polycondensation plastic fracture.

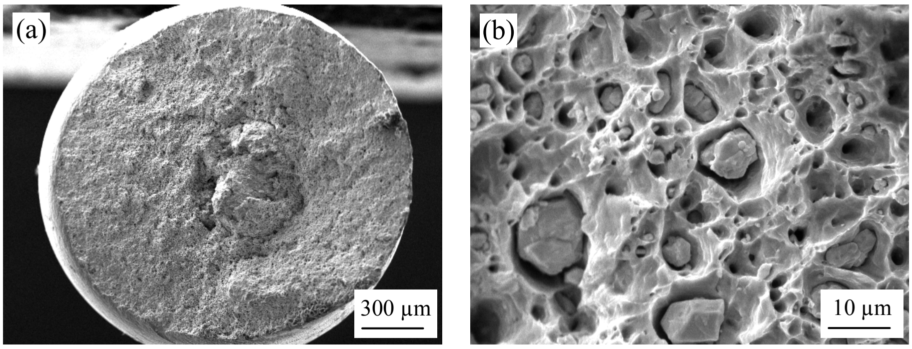

Fig.4 Fracture appearances of Ag-10%SnO_2 material

[(a). low magnification; (b). high magnification]

图 4 Ag-10%SnO_2 材料的断口形貌

[(a). 低倍照片；(b). 高倍照片]

2.4 Arc erosion surface analysis

Fig.5 shows the arc erosion surface of the $AgSnO_2$ material sintered by SPS at 800℃ for 5 min under 40 MPa. The arc erosion surface shows a large number of paste-like coagulum and bubbles. Because of the sticky liquid metal and the fast solidification, there is no time let liquid to spread out. The paste-like coagulum is formed. As shown in Fig.5, we can clearly see the sags and crests on the surface of the coagulum. This is because the liquid metal splash and the liquid bridge breaking formed by the action of the arc. With the melt of the $AgSnO_2$ material, the oxygen in the air quickly dissolves in the liquid Ag matrix. Due to the fast solidification, the dissolved oxygen can not be immediately discharged and then the bubbles are formed.

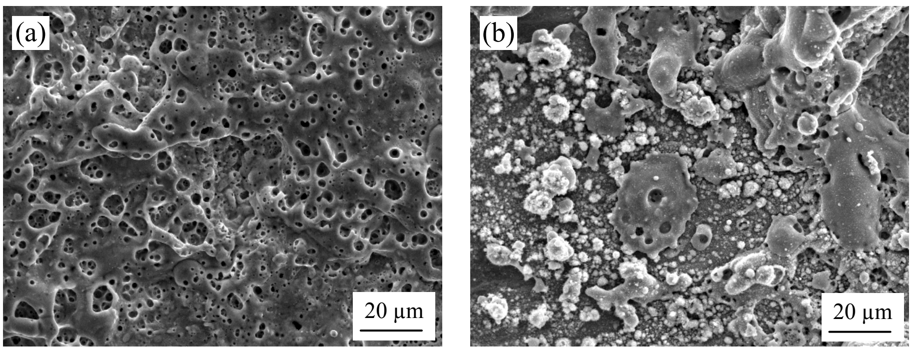

Fig.5 Arc erosion surface of $AgSnO_2$ material for anode (a) and cathode(b)

图 5 阳极(a)和阴极(b)$AgSnO_2$ 材料的电弧侵蚀表面形貌

3 Conclusions

(1) Spark plasma sintering technology has been used to prepare the Ag-10%SnO_2 material successfully. The SnO_2 particles distribute diffusely in Ag matrix, and there is no particles gathering in the Ag matrix.

(2) There are three basic stages in the process of the spark plasma sintering of Ag-10%SnO_2 material. During the sintering process, the shape and size of each particle change with the formation of grain boundaries.

(3) The tensile fracture morphology of Ag-10% SnO_2 material shows a great number of large and deep dimples, which means than the material has good plastic deformation capacity. The arc erosion surface of the material shows a large number of paste-like coagulum and bubbles.

References:

[1] Swingler J, Sumption A. Arc erosion of $AgSnO_2$ electrical contacts at different stages of a break operation [J]. Rare Metals, 2010, 29(3): 248-254.

[2] Lungu M, Gavriliu S, Canta T, et al. $AgSnO_2$ sintered electrical contacts with ultrafine and uniformly dispersed microstructure[J]. Journal of Optoelectronics and Advanced Materials, 2006, 8(2): 576-581.

[3] Liu X M, Wu S L, Paul K, et al. Effects of coating process on the characteristics of Ag-SnO_2 contact materials[J]. Materials Chemistry and Physics, 2006, 98(2/3): 477-480.

[4] Lee J W, Lee H C. Effects of tellurium addition on the internal oxidation of Ag-Sn alloys[J]. Scripta Mater, 1999, 42(2): 169-173.

[5] Wang J Z, Wang Y P, Yang Z M, et al. Effect of CuO additive on the wettability and interface behavior of Silver/tin oxide[J]. Rare Metal Materials and Engineering, 2005, 34(3): 405-408.

[6] Ken S, Makoto K, Shigeo S, et al. Sintering-resistant and self-regenerative properties of Ag/SnO_2 catalyst for soot oxidation[J]. Applied Catalysis B: Environmental, 2011, 108: 39-46.

[7] Tripathy S K, Kwon H W, Leern Y M, et al. Ag@SnO_2 core-shell structure nanocomposites[J]. Chemical Physics Letters, 2007, 442(1/3): 101-104.

[8] Lungu M, Gavriliu S, Nitu S, et al. New Ag-SnO_2-MeO ecological advanced materials for electrical contacts used in electromagnetic contactors[J]. Journal of Optoelectronics and Advanced Materials, 2008, 10(5): 1064-1068.

[9] Jo H H, Cho H, Hwang D Y. A study on the development of environment-friendly Ag-SnO_2 electric contact materials through a powder metallurgy[J]. Materials Science Forum, 2007, 539: 2761-2766.

[10] Lorrain N, Chaffron L. Sintering and evolution of nanocomposite powders Ag-SnO_2 prepared by reactive milling[J]. Materials Science Forum, 1999, 312: 153-158.

[11] Feng J, Chen J C, Xiao B, et al. Stability, thermodynamic and mechanical properties of the compounds in the Ag-Sn-O system[J]. Physica B, 2009, 404(16): 2461-2467.

[12] Kang S, Brecher C. Cracking mechanisms in Ag-SnO_2 contacts materials and their role in the erosion process[J]. IEEE Transactions on Components Hybrids and Manufacturing Technology, 1989, 12(1): 32-38.

[13] Ye Q B, Wang Y P. Redistribution of SnO_2 particles in Ag/SnO_2 materials during rapid solidification[J]. Materials Science & Engineering A, 2007, 449/451: 1045-1048.

[14] Mater J. Interface structure of Ag/SnO_2 nanocomposite fabricated by reactive synthesis[J]. Journal of Materials Science and Technology, 2010, 26(1): 49-52.

[15] Jiang F Y, Wang J B, Fu C, et al. Electrical property of nanocomposite Ag/SnO_2 electrical contact material prepared by plasma spraying[J]. Rare Metal Materials and Engineering, 2012, 41(8):1143-1146.

[16] Wang S, Xie M, Zhang J M, et al. Development of spark plasma sintering technology[J]. Precious Metals, 2012, 33(3): 73-76.

[17] Chen X H, Jia C G, Liu X B. Influence of sintering temperature on the Ag/La_2O_3 contact material prepared by spark plasma sintering[J]. Powder Metallurgy Technology, 2007, 25(6): 420-423.

[18] Liu X B, Jia C G, Wang F X, et al. Al_2O_3/Cu composite fabricated by hot pressing and spark plasma sintering[J]. Acta Materiae Compositae Sinica, 2007, 24(4): 76-81.