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Original Research Article

AgSnBi powder consolidated by CEC reciprocal extrusion

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ABSTRACT

The reciprocating extrusion (cyclic extrusion compression – CEC) process was applied to the consolidation of silver powder AgSnBi leading to the bulk material formation. Continue process of deformation by hydrostatic extrusion and then by forging the electrical contacts were produced. Microstructure and microhardness were investigated in every stage of deformation showing development of process consolidation. The relative density of material was evaluated on the base of voids existing inside the microstructure.

1. Introduction

Silver-based refractory contact materials produced by powder metallurgy are used extensively as contact materials due to their high conductivity, good resistance to welding and corrosion properties, high melting temperature and hardness. There are several successful methods including powder metallurgy (PM) and internal oxidation (IO) to prepare contact materials. Compared to PM method, IO method offers high density, little arc erosion and long service life. Powder metallurgy, among several competing mass production methods, plays an important role, not only as a means of saving material and energy, but also as a technique, delivering materials with good sintering properties which are able to substitute for conventional materials. Sintering is the most common technique for consolidating powders. Essentially, it is the removal of the pores between the starting particles, combined with their growth and strong mutual bonding. The most important are: vapor-phase sintering; solid-state sintering; liquid phase sintering; reactive liquid sintering. Overpressure sintering uses also pressure to accelerate densification [1–4].

Recently, it has been shown that equal channel angular pressing (ECAP) is an effective method to consolidate the powders at relatively lower temperatures than that used in conventional powder processing. It has been shown that applying a back pressure during ECAP not only prevents delaminating but also improves the microstructure and properties of the consolidated material [5,6]. The reciprocating extrusion

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(cyclic extrusion compression – CEC) process was very successfully applied to the consolidation of silver powders [7,8]. The results have shown that the bulk material was produced at relatively low temperature preventing the grain growth and preserving the high hardness, high level of the hardening and reducing the abrasive wear. In the case of application the severe plastic methods (SPD), such as the reciprocating extrusion – CEC or ECAP, for powders consolidation, it is possible conducting consolidation process in the room temperature contrary to the conventional methods of mechanical consolidation.

The main aim of the present study was the presenting of the results of consolidation of the AgSnBi powder by reciprocating extrusion (cyclic extrusion compression – CEC) process. The results of investigations of microstructure and properties of bulk material after consolidation and electrical contacts produced from the consolidated material were shown.

Fig. 1 – Cyclic extrusion compression–reciprocal extrusion press.

2. Experimental setup

The AgSnBi (Sn 6.8–7.5%; Bi 0.35–0.40%; Ag reminder; fraction 40–150 μm), powder has been consolidated by cyclic extrusion compression (CEC) method – reciprocating extrusion (Fig. 1). The number of 2–60 ($\varphi = 0.84$–25.2) deformation cycles have been exerting, with the deformation of $\varphi = 0.42$ in a single cycle, leading to strong consolidation and formation of bulk material. After the CEC consolidation samples were sintered at 650 °C during 3 h in atmosphere 95%N_2 + 5%H_2. The sintering temperature was determined based on AgSnBi current data of the industrial technology applying to electrical contact production. Then the sintered samples were hydrostatically extruded (HE) by the method developed in Institute of High Pressure in Warsaw to deformation $\varphi = 1.85$. The wires of 3 mm in diameter were obtained as a final product of the combined deformation. After the hydrostatic extrusion the wires were annealed at 600 °C drowns to the wire of 2.0 mm in diameter, annealed again at 600 °C, drown to the wire of 1.9 mm in diameter and then the electrical contacts with the mushroom shape were produced by forging process.

The measurement of microhardness was carried out on polished samples at room temperature using a Vickers hardness tester PMT3 at load 100 G. The optical microscopy Olympus GX51 (MO) was used for the microstructure observation of samples after the consolidation, combined deformation and the final product – electrical contacts. The details of microstructure of consolidated samples were investigated by scanning electron microscopy SU-70 (SEM). The microstructure of consolidated samples was also investigated by transmission electron microscopy (JEOL 2010 ARB). Thin foils were prepared from cross sections by cutting, grinding and ion sputtering, using Struers and Gatan instruments.

3. Experimental results

3.1. *Microstructure*

The almost equiaxial grains have been found in the microstructure of samples, observed by optical microscopy (Fig. 2). The three distinctly different phases are recognized in AgSnBi samples: Ag, Ag_3Bi and $Ag_{6.7}Sn$. It is also probable that the oxides could appear between the grains. The existence of phases is expressed by the differentiated microstructure contrast. Using the TEM investigation methods the characteristic microstructure was found with the numerous twins inside the grains, which express the nanometric thickness (Fig. 3).

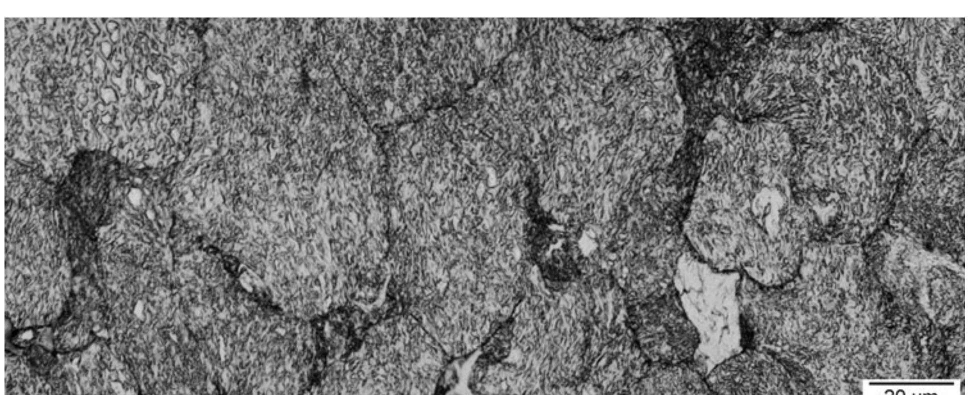

Fig. 2 – Microstructure of AgSnBi powder consolidated by CEC ($\varphi = 4.2$, MO).

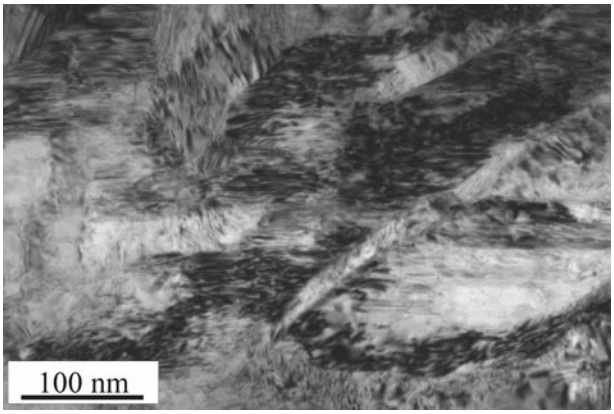

Fig. 3 – Microstructure of AgSnBi sample after $\varphi = 4.2$ by CEC (TEM).

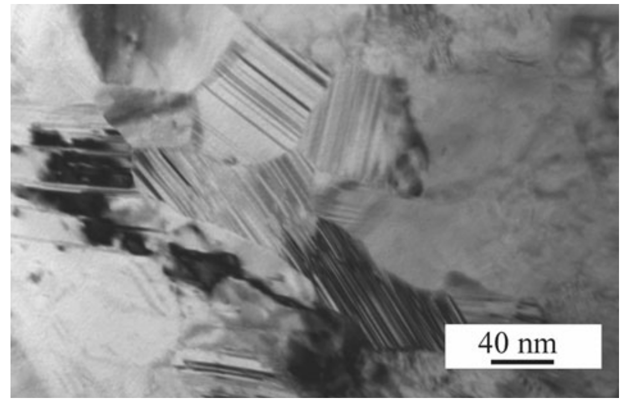

Fig. 6 – Microstructure of AgSnBi sample after $\varphi = 16.8$ by CEC and hydrostatic extrusion (TEM).

After the hydrostatic extrusion the grains become elongated to the extrusion direction (Fig. 4). Inside the elongated grains the mutually crossing microbands have been observed. Bands and microbands are also typical in microstructure after the combined deformation CEC and hydrostatic extrusion, especially after exertion of deformation, $\varphi = 16.8$. The areas with elongated microbands (Fig. 5) and also areas with nano-grains with numerous nano-twins (Fig. 6) have been observed inside the samples.

As the final product of combined deformation, the wires with the 3 mm in diameter were obtained. Fig. 7 shows the subsequent stages of electrical contacts: (A) CEC sample, (B) wire and (C) electrical contacts in the shape of mushrooms. The electrical contacts consist with the ''leg'' and the ''hat'',

Fig. 4 – Microstructure of AgSnBi sample after $\varphi = 8.56$ by CEC and hydrostatic extrusion (MO).

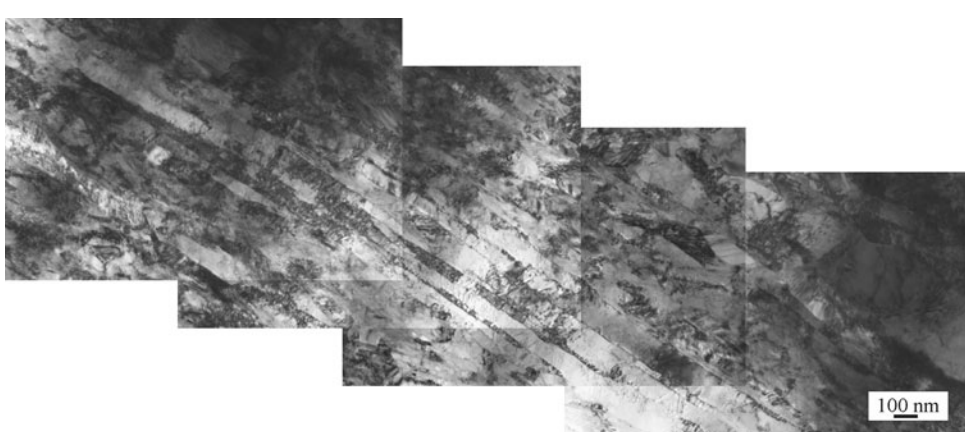

Fig. 5 – Microstructure of AgSnBi sample after $\varphi = 16.8$ by CEC and hydrostatic extrusion (TEM).

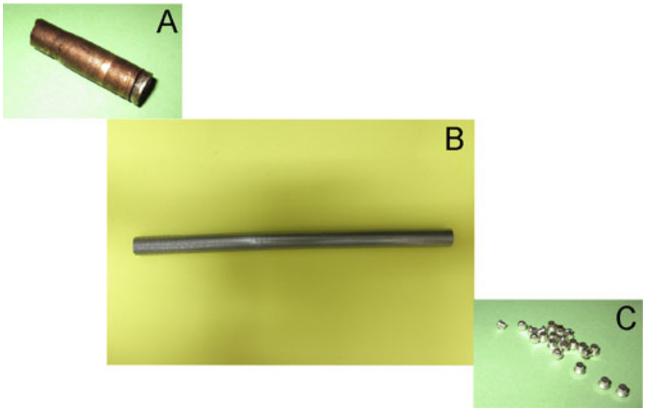

Fig. 7 – The subsequent stages of electrical contacts production.

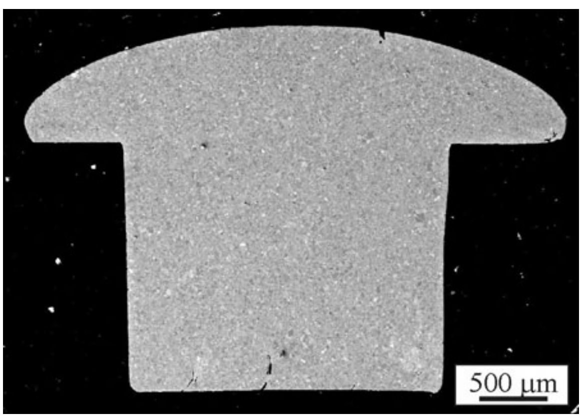

Fig. 8 – Shape of the electrical contact.

which is obtained in the forging process after the consolidation by the CEC and hydrostatic extrusion (Fig. 8).

Microstructure of electrical contacts is presented respectively: "hat" in Fig. 9 and "leg" in Fig. 10. The equiaxial grains with numerous twins inside are typical for both parts of electrical contact. They are much differentiated areas visible with very small and large grains. The second phases are placed along the grain boundaries.

3.2. Microhardness

The microhardness measurements are presented in Fig. 11. In the case of CEC deformation after the strong initial increase the stabilization of material microhardness has been observed in the range of 98–111 HV0.1. The microhardness of the samples after the combined deformation: CEC + sintering + hydrostatic extrusion has been shown that the level of microhardness after the combined deformation increases to about 10–20 units in comparison to the level after the CEC. However the course of microhardness is similar and also stays stable after the initial increase. In the range of "plateau" the level of microhardness achieved about 120 HV0.1.

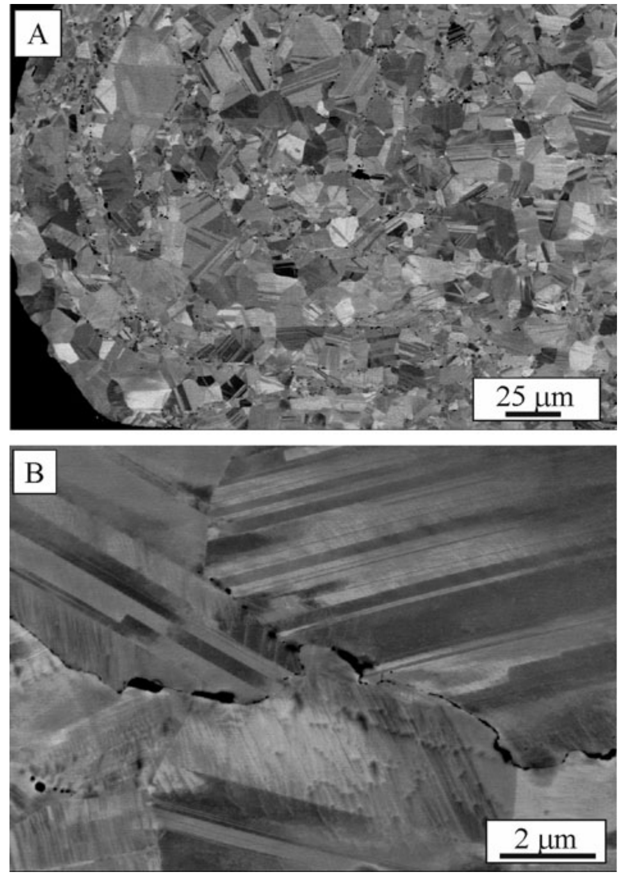

Fig. 9 – Microstructure of "hat" of electrical contact (SEM), (A) equiaxial grains, B) microtwins inside grains.

The measurement of microhardness of "hat" of the produced electrical contacts shows that it reached the value of about HV0.1 = 81. The microhardness of "leg" achieved level of about HV0.1 = 68. The higher value of "hat" microhardness is connected with the additional plastic working exerted during the compression process connected with the formation of "hat" shape.

3.3. Porosity

The porosity of consolidated samples has been estimated from the void distribution existing inside the microstructure. The microstructure was observed by using scanning electron microscope SU-70. The investigated area for single sample to determine density was about 2 cm^2, which represents about 15% of cross section of sample area. The surface of microvoids was determined by the own software "Structure". The obtained results show average porosity for investigated area which was statistically selected from observed sample area. After the combined deformation CEC and hydrostatic extrusion the density of samples is higher than that after only the CEC process; however, in the range of higher deformation the density achieved the same level (Fig. 12). The porosity investigations revealed that with the deformation increase the density of the material increased. It could be found that after the some value of deformation the border level of density

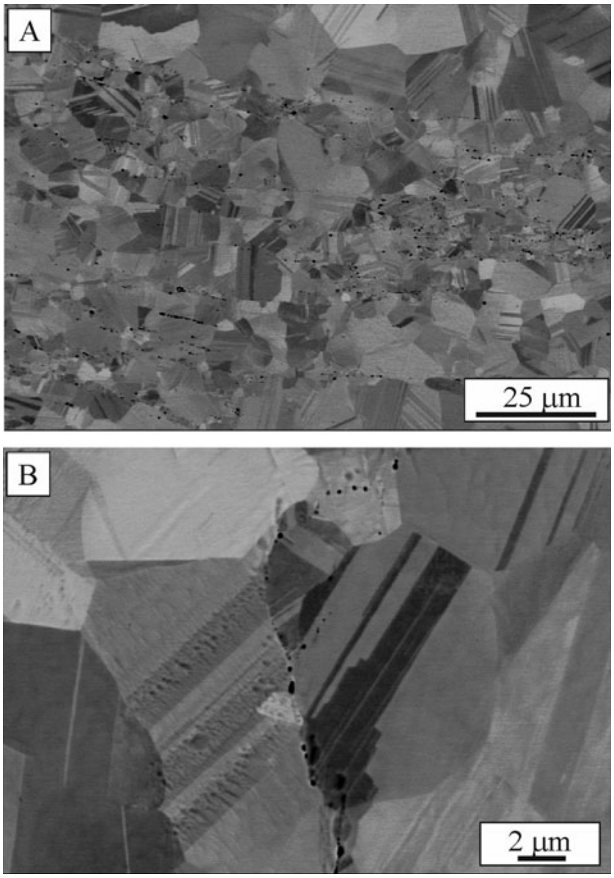

Fig. 10 – Microstructure of "leg" of electrical contact (SEM), (A) equiaxial grains, B) microtwins inside grains.

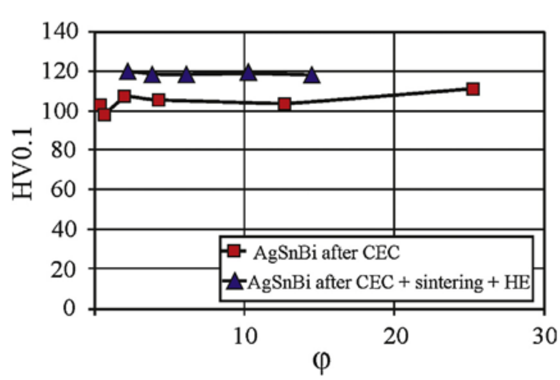

Fig. 11 – Microhardness of AgSnBi after the CEC deformation and CEC + sintering + HE.

is achieved and the subsequent deformation do not improve material consistency.

4. Summary

The performed investigations cover little known area of AgSnBi alloy applying to electrical contact production. The work shown the new way of formation of bulk AgSnBi alloy

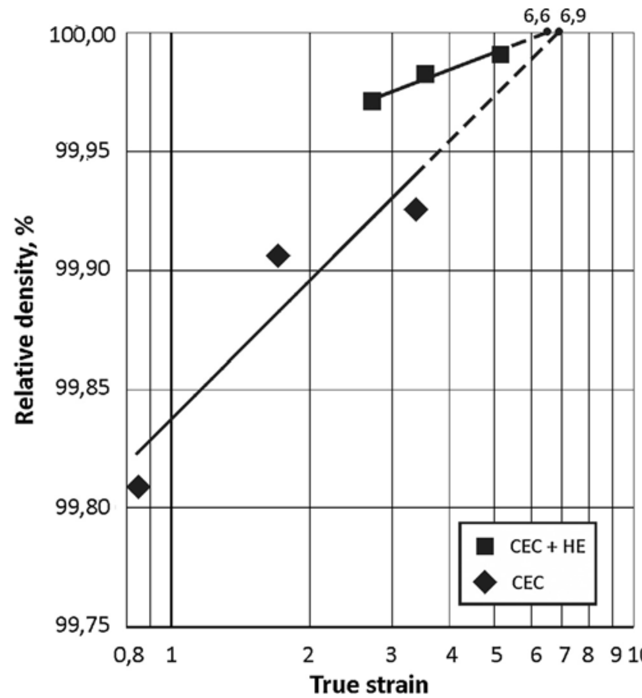

Fig. 12 – Density of the consolidated samples.

from powders precursors and gave new results till now not existing in the words literature. The studies performed have shown that by using severe plastic deformation methods it is possible to produce the bulk material from powders. The reciprocating extrusion (cyclic extrusion compression – CEC) is very useful for the consolidation of silver powder AgSnBi, applying for electrical contacts. The microhardness of consolidated powder achieved level from 100 to 120 HV0.1 respectively for CEC deformation and combined CEC + sintering + hydrostatic extrusion. The microstructure of samples is very diverse and consists from areas of larger and smaller grains with the numerous microtwins inside. It was found that after the some values of deformation the subsequent deformation increase does not increase the material density.

Acknowledgment

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