ARCHIVES OF METALLURGY AND MATERIALS
Volume 58 2013 Issue 1
DOI: 10.2478/v10172-012-0153-x

M.W. RICHERT*, J. RICHERT*, A. HOTLOŚ*, M. MROCZKOWSKI*, T. TOKARSKI*

THE EFFECT OF RECIPROCATING EXTRUSION (CEC) ON THE CONSOLIDATED SILVER POWDERS MICROSTRUCTURE

WPŁYW DWUSTRONNEGO WYCISKANIA (CWS) NA MIKROSTRUKTURĘ KONSOLIDOWANYCH PROSZKÓW SREBRA

The microstructure and properties investigations silver powders consolidated by cyclic extrusion compression (CEC) were performed. The AgSnBi and AgNi powders using for electrical contacts were investigated. The observation by using optical microscopy (MO) and transmission electron microscopy (TEM) were apply. The strong diminishing of microstructure inside the consolidated granules were found. This phenomenon cause microhardness increase, restricted by material porosity.

Keywords: Reciprocating Extrusion (CEC), SPD consolidation, Ag powders

W pracy przedstawiono badania mikrostruktury i własności proszków srebra konsolidowanych metodą cyklicznego wyciskania ściskającego – CWS. Badania objęły proszki AgSnBi oraz AgNi stosowane na styki elektryczne. Przeprowadzono obserwacje przy użyciu mikroskopu optycznego (MO) oraz transmisyjnego mikroskopu elektronowego (TEM). Stwierdzono silne rozdrobnienie mikrostruktury wewnątrz konsolidowanych granul proszków. Spowodowało to wzrost mikrotwardości, ograniczony porowatością materiału.

1. Introduction

The increase in electronic and computer controls in transport, machining and numerous other industrial and domestic applications has induced a considerably increase in electrical contacts applications during the last decades. A candidate for the electric contact alloy need to fulfill the following requirements: suitable melting temperature, good mechanical properties, good resistance to mechanical and thermal fatigue, corrosion resistance, good electrical properties, good for health and environment, availability and low material cost [1]. Silver is also used in composite materials [2].

The AgSnBi and AgNi silver powders, free from Pb, are used to production of the electrical contact, because of the high cost of the bulk materials. The Reciprocating Extrusion (Cyclic Extrusion Compression – CEC) process was applied to the consolidation of these silver powders [3]. The cyclic way of deformation and the preservation of the initial shape of samples are the advantages features of applicable method leading to the good consolidation of powders. The process of the consolidation in the conventional conditions requires high temperatures [4,5]. In the case of application severe plastic methods (SPD), such as the Reciprocating Extrusion – CEC, the consolidation is possible in the room temperature preserving the higher hardness, higher level of the hardening and reducing the abrasibility. The important is the kind of microstructure of the consolidation materials, which strongly influence on the properties of the electrical contacts and also on the possibility of contact production, in the successive plastic working processes after the consolidation [6]. Agglomerate consolidation through plastic deformation make the granules structure denser [7] and restricted the porosity of the material.

In the present work the AgSnBi and AgNi powders were mechanically consolidated by using cyclic extrusion compression method (CEC – Reciprocating Extrusion). The microstructure and properties of consolidated powders were investigated.

2. Experimental procedure

The AgSnBi and AgNi powders were mechanically consolidated by using cyclic extrusion compression method in the specially prepared copper containers. The mean size of AgSnBi initial powder achieved 40 μm in diameter, and AgNi 40 μm, measured by the mean chord method.

The powders were obtained by gas atomization. Powders were deformed in the range of strains ϕ = 0.42-25.3, which corresponds to 1-60 CEC cycles of reciprocating extrusion. In the case of the AgNi powder the samples were deformed in the range of strains ϕ = 0.42 to ϕ = 6.7 (1-16 CEC cycles). The ϕ = 0.42 deformation was exerted in a single CEC cycle. The microstructure of samples was studied by optical microscopy (Olympus GX50) and scanning electron microscopy (Hitachi SU-70). The microstructure of consolidated samples was also investigated by transmission electron microscopy (JEOL 2010 ARB). Thin foils, for TEM investigations, were prepared from cross sections by cutting grinding and ion thinning, using

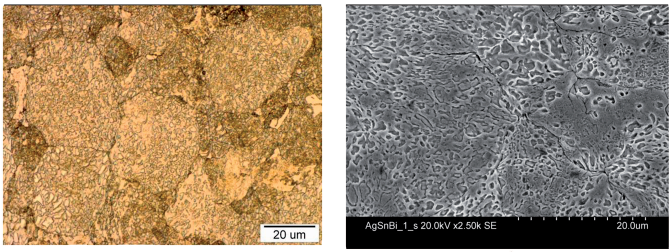
---

* AGH, UNIVERSITY OF SCIENCE AND TECHNOLOGY, FACULTY OF NON – FERROUS METALS, 30-059 KRAKÓW, AL. MICKIEWICZA 30, POLAND

Struers and Gatan instruments. The measurement of microhardness was carried out on polished samples using a Vickers hardness tester PMT3 at load 100 g.

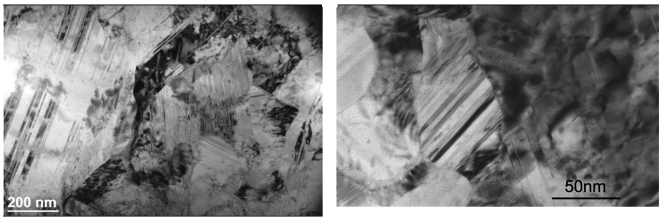
3. Results

The AgSnBi and AgNi silver powders were consolidated applying the CEC method in the range of the true strains $\phi = 0.48$ (1 CEC cycle) to $\phi = 25.3$ (60 CEC cycles). The bulk samples were obtained just after the first CEC cycle (Fig. 1). Optical microscopy observation of AgSnBi shows good consolidate samples (Fig. 1a). Microstructural details observed by SEM revealed some flattened pores visible as dark lines along the granules boundaries (Fig. 1b). Transmission electron microscopy observations show that consolidated powder is mainly composed of equiaxial grains of 100-500 nm containing nano-twins and lamellas with a few nanometers in thickness (Fig. 2). The mean size of grains inside powder granules versus the deformation exerted in CEC process is presented in Fig. 3. The result indicated that after the strong diminishing from 120 nm to about 50 nm, the further deformation generally doesn't change the grain size.



a b

Fig. 1. Microstructure of AgSnBi consolidated by CEC, a) optical microscopy (MO) observation after the1 CEC cycle $\phi = 0.42$, b) scanning microscopy (SEM) observation after the 1 CEC cycle



a b

Fig. 2. Microstructure of AgSnBi observed by transmission electron microscopy (TEM), a) after 10 CEC cycles ($\phi = 4.2$), b) after 32 CEC cycles ($\phi = 13.5$)

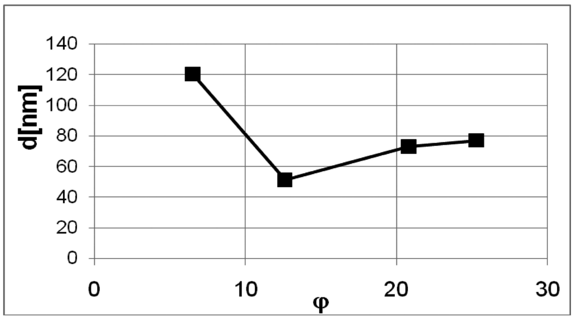


Fig. 3. The mean size of AgSnBi grains inside the consolidated granules versus the deformation exerted in the CEC process, at the base of TEM results

The microhardness of consolidated AgSnBi samples is presented in Fig. 4. The strong increase of hardening from initial level was found just after the first CEC cycle, from 20 to 110 μHV. In the range of 0.42 (1 CEC cycle) to 25.2 (60 CEC cycles) the only very slight increase of microhardness was found. It suggests that just after the first CEC cycle the dense structure was obtained and the further deformation causes only slight increase in sample density. The analysis showed no grain growth and also no grain diminishing during the CEC deformation, which explain the observed behavior. Some slight results deviations are probably connected with the inhomogeneity of samples.

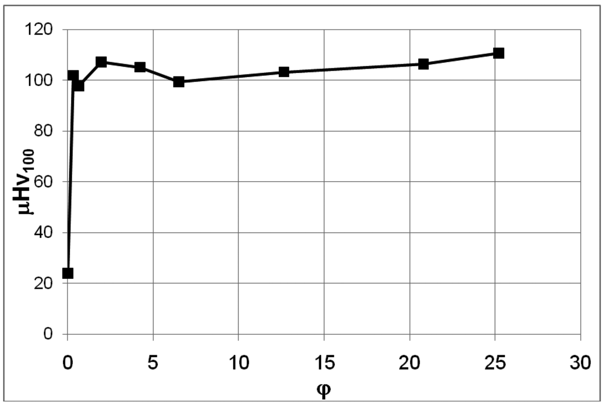
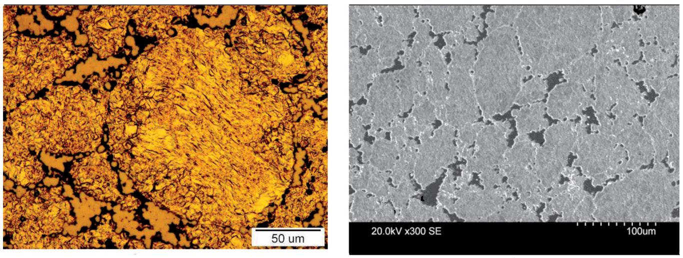


Fig. 4. The AgSnBi microhardness after the consolidation by CEC method

Consolidated AgNi powder exhibited more pores even after the 2 CEC cycles (Fig. 4). The pores are visible just after the optical microscopy observations (Fig. 4a) and they are very clearly visible in the scanning microscope observations as black places (Fig. 4b). Characteristic is their location in Ag-Ni phases boundaries.



a b

Fig. 5. Microstructure of AgNi consolidated by CEC, a) optical microscopy observation after 2 CEC cycles ($\phi = 0.84$), b) SEM observation after the 4 CEC cycles ($\phi = 1.7$)

The TEM observations indicated that area of Ag and Ni well adjoins to each other. Fig. 5 shows Ni phase without deformation twins close to Ag (Fig. 5a) and in Fig. 5b only Ag phase with the nano-twins. The subgrain dimensions are much larger than in AgSnBi samples. The microhardness course of consolidated AgNi powder is similar to AgSnBi. It keeps at the almost the same level of about 70-80 μHV in the whole deformation range (Fig. 6). However the level of AgNi microhardness is about 30 units lower than level of AgSnBi microhardness.

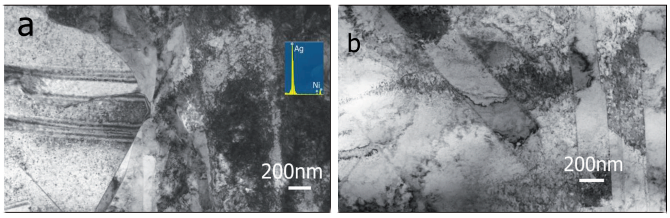


Fig. 6. AgNi powder consolidated by CEC, a) microstructure after the 4 CEC cycles Ag and Ni areas, b) Ag area with nano – twins (4 CEC cycles), ϕ = 1.7

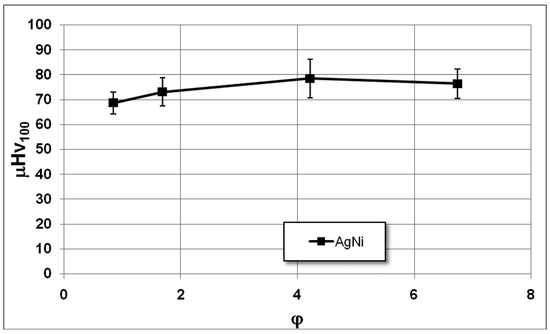


Fig. 7. Microhardness of AgNi consolidated by CEC method

4. Summary

The reciprocating extrusion (cyclic extrusion compression – CEC) [3] was used to fabricate the bulk AgSnBi and AgNi samples from powders by mechanical consolidation. Achievement of a fully dense material is an important task in the powder processing. The key to the powder consolidation process is to obtain as high as possible densification with minimal microstructural coarsening and/or undesirable microstructural transformations. In addition, the fully dense specimen must be of sufficient size for reliable testing of final properties or a useful final product. The application of high plastic strains induces dynamic grain growth, which can lead to the weakening processes. The CEC method allows to consolidate powders at room temperature, which can suppress the dynamic coarsening processes. Large strain accumulation cause diminishing powder microstructure and as in the case of investigated AgSnBi can produce nanostructures. The densification of powders has been developed just from the first CEC cycle, especially successfully in AgSnBi samples. No significant hardness change with the increase of the CEC deformation during the AgSnBi and AgNi powder consolidation has been found. It suggests the good consolidation from the beginning (just after the first CEC cycle) and only slight increase of density with the increase of deformation accumulation.The existence of pores in AgNi is generally connected with the lack of solubility of Ag and Ni atoms and separation of phases. The high level of AgSnBi and AgNi microhardness, about 110 and 80 μHV respectively, assure good wear and tear resistance during the electrical contact exploitation, which are produced from the consolidated powders.

5. Conclusions

1. The CEC (cyclic extrusion compression) process is the effective method to consolidation of the silver powders for electrical contacts.
2. The bulk AgSnBi samples, fabricated by CEC consolidation, from silver powders showed the microstructure with nanometric features
3. No significant hardness change with the increase of the CEC deformation during the AgSnBi and AgNi powder consolidation has been found, the level of microhardness of AgSnBi achieved 110 μHV and 80 μHV for AgNi respectively

Acknowledgements

This work is supported by European Program No POIG.01.01.02-00-015/09-00.

REFERENCES

[1] Ł.Ł. Wierzbicki, W. Malec, J. Stobrawa, B. Cwolek, B. Juszczyk, Studies into new, environmentally friendly Ag-Cu-Zn-Sn brazing alloys of low silver content, Archives of Metallurgy and Materials **52**, 1, 147-158 (2011).

[2] M. Madej, Silver-based infiltrated composites, Archives of Metallurgy and Materials **57**, 2, 605-612 (2012).

[3] J. Richert, M. Richert, Aluminium **62**, 8, 604 (1986).

[4] M.T. Marques, J.B. Correia, R. Vilar, Rev.Adv.Mater.Sci. **18**, 403 (2008).

[5] E. Ayman, V. Junko, K. Katsuyoshi, Magnesium **38**, 31 (2009).

[6] M. Richert, J. Richert, A. Hotloś, P. Pałka, W. Pachla, M. Perek, Materials Science Forum **667-669**, 145 (2011).

[7] H.J. Cheng, S.S. Hsian, The study of granular agglomeration mechanism powder technology **199** , 272-283 (2010).

Received: 15 February 2012.