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Microstructure of AgSnBi powder consolidated in reciprocating extrusion process

M.W. Richert [a,*], J. Richert [a], A. Hotloś [a], M. Perek-Nowak [a], T. Tokarski [a], J. Skiba [b]

[a] AGH University of Science and Technology, Al. Mickiewicza 30, 30-059 Kraków, Poland

[b] Institute of High Pressure Physics of the Polish Academy of Sciences, ul. Sokołowska 29/37, 01-142 Warszawa, Poland

* Corresponding e-mail address: mrichert@agh.edu.pl

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ABSTRACT

Purpose: The AgSnBi powder used for electrical contacts has been consolidated in the process of reciprocating extrusion (cyclic extrusion compression - CEC) in 2, 4, 8 and 16 CEC cycles at room temperature. It corresponds to the deformations: 2 CEC cycles - $\varphi = 0.84$, 4 CEC cycles - $\varphi = 1.68$, 8 CEC cycles - $\varphi = 3.36$ and 16 CEC cycles - $\varphi = 6.72$. The microstructure of consolidated powder has been characterized by optical microscopy (MO), scanning electron microscopy (SEM) and transmission electron microscopy (TEM). It was found characteristic granular microstructure with the oxides film at granular boundaries. Some voids and cracks were observed in consolidated samples, especially at higher magnifications. Inside the consolidated granules the nano-microstructure with nano - twins was found. Microhardness of AgSnBi after the consolidation by the CEC process achieved level of about 100-110 μHV. The microhardness of samples consolidated by CEC and then hydrostatically extruded increase of about 20 μHV units.

Design/methodology/approach: The investigations of microstructure were performed by optical microscopy (MO) Olympus GX51, and scanning electron microscopy SU-70 with field emission gun thermally aided. The microhardness of consolidated samples was measured by Vickers method.

Findings: The microstructures of consolidated AgSnBi powders were observed and analyzed. On the base of the microstructure and microhardness investigations the mechanisms of formation of bulk material from powder by severe plastic deformation (SPD) methods was discussed.

Practical implications: The performed investigations contribute to the understanding of processes of deformation in the range of unconventional strains exerted by the SPD methods. The new way of consolidation of powders using to the production of electrical contacts was presented.

Originality/value: It was assumed that AgSnBi powder consolidated by CEC and hydrostatic extrusion exhibited the nano-microstructure.

Keywords: Consolidation; Cyclic extrusion compression; Hydrostatic extrusion; Microstructure; Powders; Electrical contacts

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MATERIALS

1. Introduction

Nanocrystalline (NC) and ultrafine-grained (UFG) materials have received considerable attention within the last decade owing to their improved properties as compared with the conventional coarse-grained materials. Among possible techniques in powder consolidation the severe plastic deformation (SPD) processes offers several advantages over the competing processes. It was found that the powder consolidation by the equal channel angular pressing (ECAP) technique is more effective in obtaining bulk samples with less porosity at lower temperatures compared to bulk specimens obtained by the commonly utilized hot isotactic pressing technique [1]. The nanocrystalline copper was obtained at room temperature by powder consolidation using equal channel angular pressing (ECAP) [2]. On the cyclic stability of nanocrystalline copper obtained by powder consolidation at room temperature [3]. Also using ECAP technique Al89 Gd7Ni3Fe1 alloy powder produced by gas atomization was consolidated to bulk material [4]. After compaction at 210°C, the material was about 70% amorphous and it had 98-99% of the theoretical density. High pressure torsion (HPT) was used to consolidation of Cu-NbC at room temperature. Starting from elemental powders Cu-NbC composite was synthesized through mechanical alloying and cold consolidation of electrical conductivity and tensile strength were obtained. The silver powders AgSnBi and AgNi using to production of electrical contacts were cold consolidated by cyclic extrusion compression (reciprocating extrusion - CEC), obtaining good hardness and nanometric microstructure [5,6].

The quality of the product obtained through SPD consolidation is very much influenced by the various processes parameters such as temperature, initial preform density, alloying elements, flow stress and initial size of powders and its step of agglomeration. The important is also strain rates of deformation, lubrication and deformation level.

The cohesion and adhesion processes are responsible for bulk material formation from consolidated powders. Adhesion concerns numerous technologies related to surface engineering, such as paint and enamel coating, or application of coats with special properties. Moreover, adhesion as a phenomenon plays an important role in such technologies as bimetal production, powder metallurgy, flotation processes, production of laminated composites, and many others. Adhesion is also highly important in the interpretation of numerous processes in engineering. This concerns in particular the techniques and processes of diagnostics as well as the processes of friction and wear.

Processing of materials through conventional powder metallurgy starts with the consolidation of metal or alloy powders by applying uniaxial or biaxial pressure followed by sintering [7]. The density levels obtained in sintering are always much less than the theoretical values because of the difficulties involved in elimination of small rounded pores. Presence of such micro pores always renders the material weak because these pores act as sites of origination of cracks during service. Elimination of porosity in the sintered components calls for subsequent deformation processing of the preforms such as forging, extrusion, etc. Powder metallurgy (PM) followed by compaction or sintering is a common solid state manufacturing technique. In addition, vacuum hot pressing (VHP) can enhance the sintering rate by providing additional stress at elevated temperatures. In accordance with the ability to obtain a uniform distribution of reinforcement and near net-shape formability, it is possible to use the processes efficiently [8].

This aim for the present paper is mechanical consolidation of the AgSnBi powder by using cyclic extrusion compression (CEC, reciprocating extrusion). The effects of the processing conditions on consolidation and the microstructure evolutions of the powder materials were investigated. The relationships of the different processing conditions and the evolutions of microstructure and its subsequent properties were analyzed.

2. Experimental basis

The AgSnBi powder (Fig. 1) was mechanically consolidated by using cyclic extrusion compression method (CEC) in the special preparing copper containers. The mean size of AgSnBi initial powder had 40 μm in diameter, measuring by mean chord method. Powder was deformed in the range of deformation $\varphi = 0.42\text{-}25.3$, which corresponds to 1-60 CEC cycles of reciprocating extrusion. The $\varphi = 0.42$ deformation was exerted in a single CEC cycle. After the CEC consolidation AgSnBi samples were sintered in 923K (650°C) during 3 hours in atmosphere 95%N_2+ 5%H_2. Than the sintered samples were hydrostatically extruded by the method developed in the 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. The microstructure of samples was studied by Olympus GX51 optical microscopy (MO) and scanning electron microscopy SU70 with gun emission. Thin foils to microstructure observation were prepared from cross sections by cutting grinding and ion sputtering, using Struers and Gatan instruments. The measurement of microhardness was carried out on polished samples at room temperature using a Vickers hardness tester PMT3 at load 100 G.

3. Investigation results

Fig. 1 to Fig. 4 exhibit representing microstructure of AgSnBi powder consolidated by cyclic extrusion compression (CEC), observed by the optical microscope. The powder granules with the inert microstructure have almost equiaxial shape. The distinct boundaries of granules correspond to oxide film. A few interconnected porosity are also seen in the structure, as a black places. Total elimination of the porosity and hence the attainment of cent percent density during upsetting is not possible under pure upsetting mode of deformation.

The CEC and CEC + HE deformed samples are compared. The combined deformation CEC and HE shows higher hardening level in comparison to the level of CEC samples microhardness. The hardening increase result from shear bands development in the hydrostatically extruded bulk samples consolidated by CEC and lamella twins rotation inside the granules. Shear bands crossing lamella twins considerably increase bulk sample hardening.

Fig. 1. Initial AgSnBi powder (SEM)

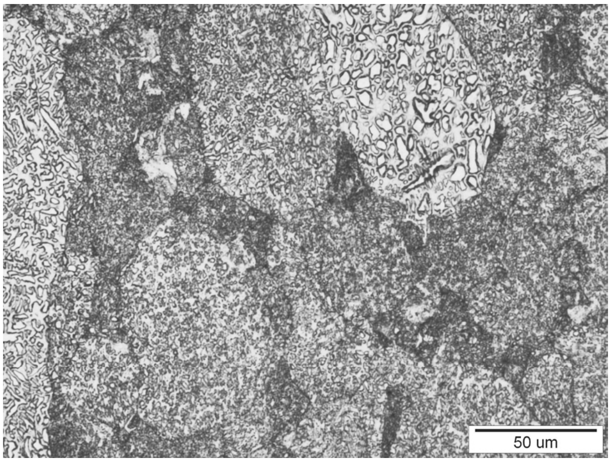

Fig. 2. Consolidated AgSnBi powder after 2 CEC cycles (φ = 4.84) (MO)

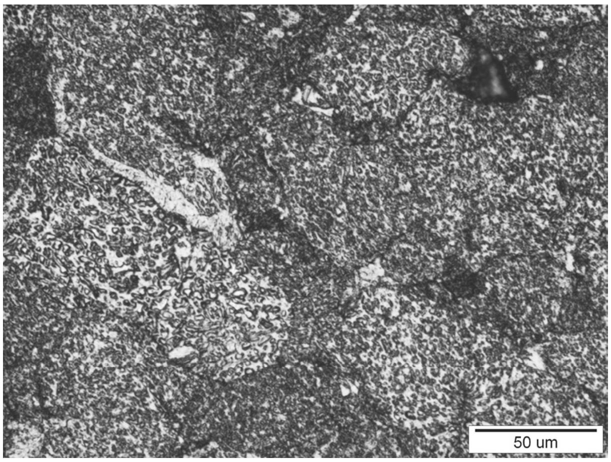

Fig. 3. Consolidated AgSnBi powder after 4 CEC cycles (φ = 1.68) (MO)

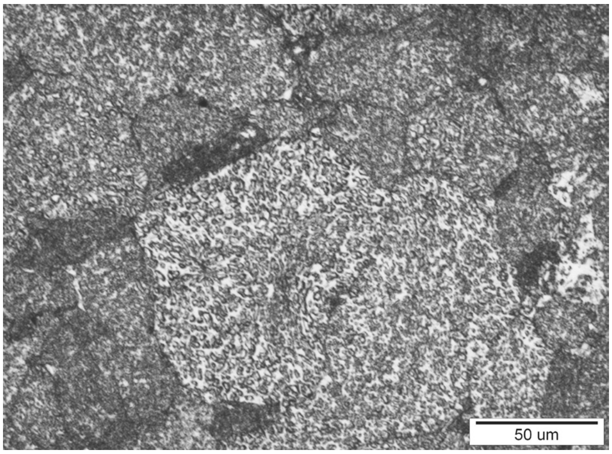

Fig. 4. Consolidated AgSnBi powder after 8 CEC cycles (φ=3.4) (MO)

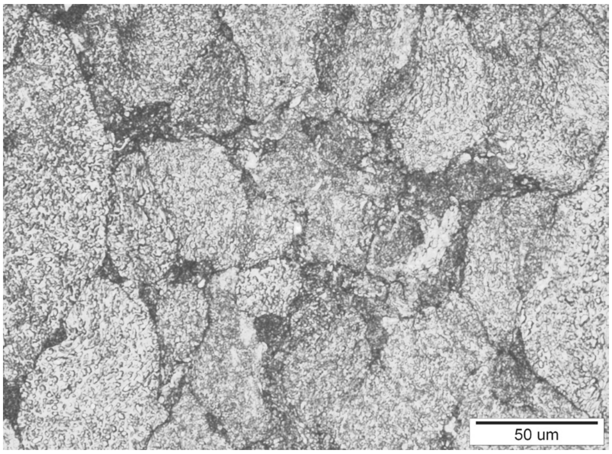

Fig. 5. Consolidated AgSnBi powder after 16 CEC cycles (φ = 6.7) (MO)

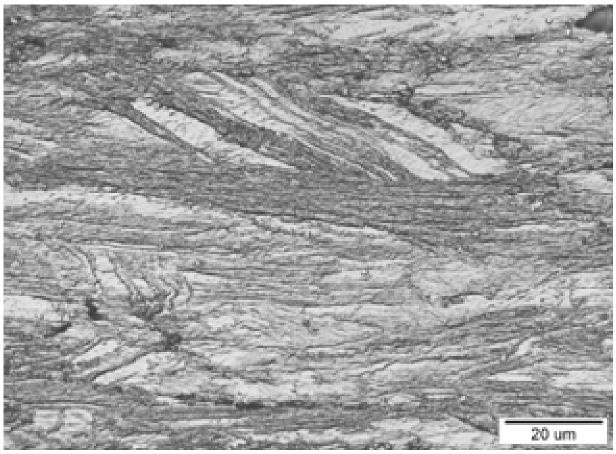

Fig. 6. AgSnBi powder after 2 CEC cycles (φ = 4.84) and hydrostatic extrusion (φ = 1.82) (MO)

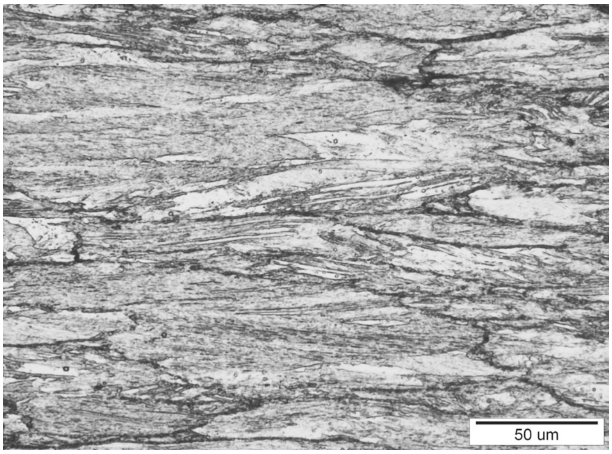

Fig. 7. AgSnBi powder after 4 CEC cycles (φ = 1.68) and hydrostatic extrusion (φ = 1.82) (MO)

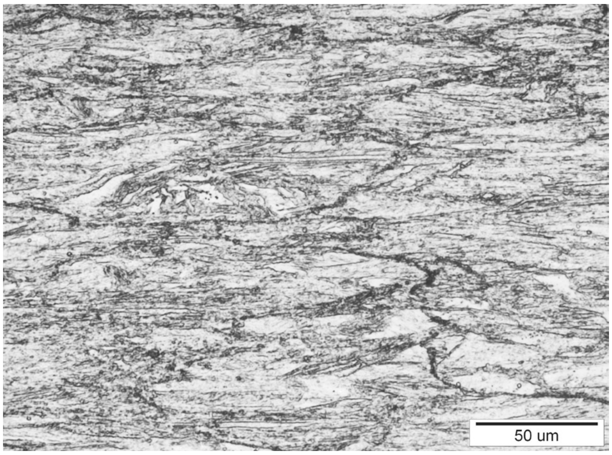

Fig. 8. AgSnBi powder after 8 CEC cycles (φ = 3.4) and hydrostatic extrusion (φ = 1.82) (MO)

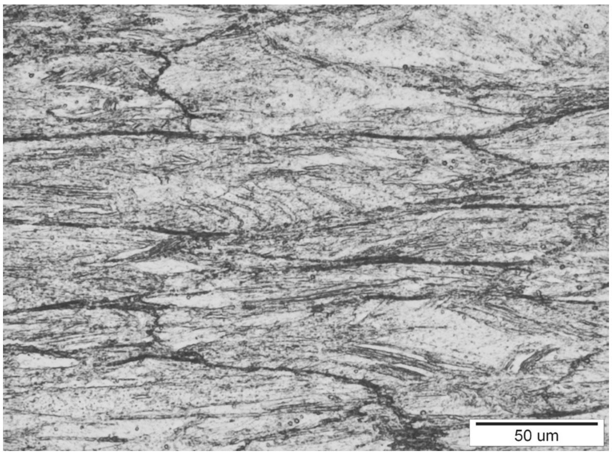

Fig. 9. AgSnBi powder after 16 CEC cycles (φ = 6.7) and hydrostatic extrusion (φ = 1.82) (MO)

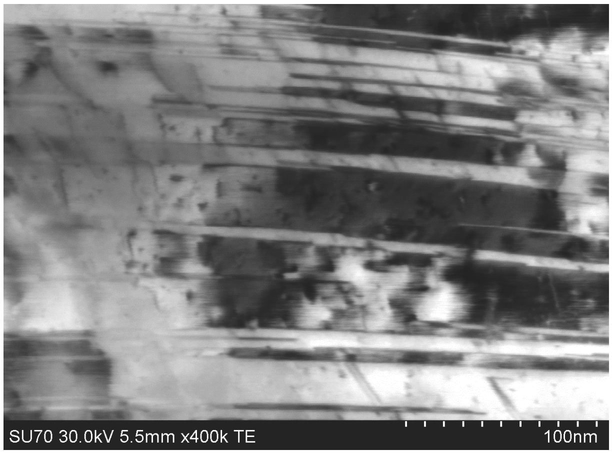

Fig. 10. Twins in the AgSnBi powder after the 8 CEC cycles (φ = 3.4) and hydrostatic extrusion (φ = 1.82) (SEM)

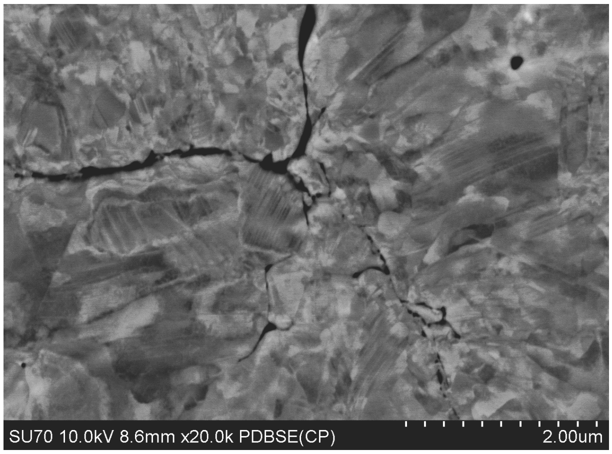

Fig. 11. Consolidated AgSnBi powder after 4 CEC cycles (φ = 1.68) (SEM)

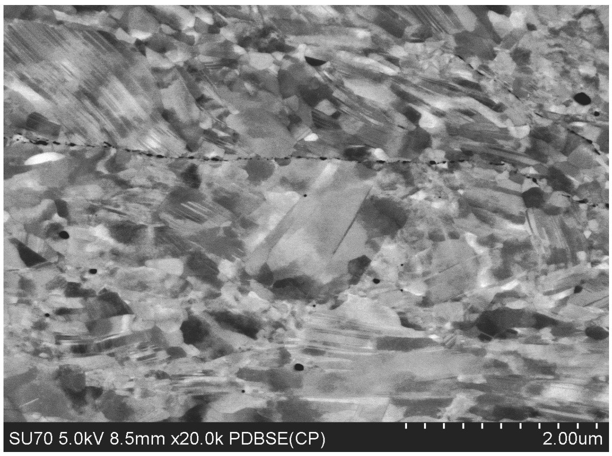

Fig. 12. AgSnBi powder after 16 CEC cycles (φ = 6.7) and hydrostatic extrusion (φ = 1.82) (SEM)

After complexion by CEC the samples were hydrostatically extruded to deformation ε = 1.8. Fig. 6 to Fig. 9. present the typical microstructures of the silver powder consolidated by the combined deformation CEC and HE. The one way direction of the hydrostatic extrusion strongly influenced on the grain shape. The granules are elongated to the extrusion direction and influenced on the structure anisotropy. Characteristic are deformation twins limited to the grain size inclined to the extrusion direction. The effects of twins deflection and mutually crossing twins with the very variable nanometric thickness are visible at the longitudinal sections of hydrostatically extruded samples (Fig. 10). The full densification was not reached, mainly owing to that the powders insufficient connection and shear deformation. The black pores situated generally at granules boundaries, especially in the triple point junctions and also at twins boundaries (Fig. 11). The Vickers microhardness measurements from longitudinal cross-sections of consolidated samples has been presented in Fig. 12.

The CEC and CEC + HE deformed samples are compared. The combined deformation CEC and HE shows higher hardening level in comparison to the level of CEC samples microhardness (Fig. 13). The hardening increase result from shear bands development in the hydrostatically extruded bulk samples consolidated by CEC and lamella twins rotation inside the granules. Shear bands crossing lamella twins considerably increase bulk sample hardening.

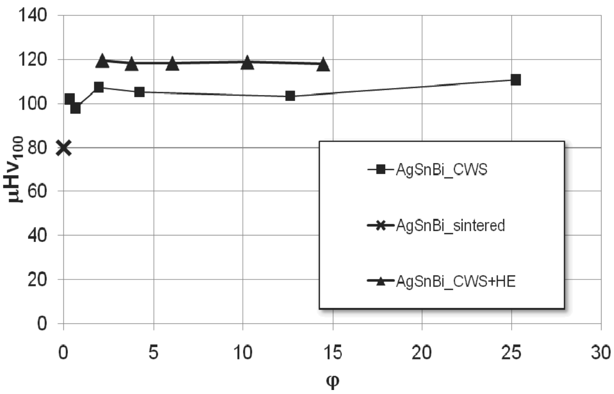

Fig. 13. Microhardness of AgSnBi powder consolidated by CEC, CEC + HE and the level of sintered samples is shown

4. Summary

Bulk material was obtained by cyclic extrusion compression consolidation. The results show almost stable microstructure and microhardness level in the deformed by CEC samples. The noticeable increase of microhardnes was found after the additional deformation of bulk samples by hydrostatic extrusion. It also changes the microstructure, in which the shear bands crossing particles and deformation twins appeared.

The densification of samples was very good however in some places just after the CEC and HE pores and voids were found, especially at granules boundaries and also at boundaries of twins.

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