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Synthesis of Ag–ZnO composites via ball milling and hot pressing processes

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Ag – 8 wt. % ZnO composites were synthesized by ball milling, heat treating and hot pressing of silver and zinc oxide powder mixtures. The crystalline size and microstrain of the milled powders before and after heat treatment were determined by Debye-Scherrer and Williamson-Hall methods. It was shown that heat treatment resulted in decrease of microstrain and increase in the crystallite size of the milled powders. The effect of uniaxial pressure magnitude and duration of hot pressing at 550 °C on the final density of the powder compacts were investigated. The results showed that both plastic flow and atomic diffusion mechanisms affected densification of the composite powders during the hot pressing process. However, the latter one had more effective role on the density of the hot-pressed samples. The synthesized composites showed homogenous microstructure with relatively high density and hardness.

Keywords: *silver matrix composites; hot pressing; crystallite size; microstrain*

1. Introduction

Metal matrix composites (MMCs) are used in several industrial applications such as aerospace and combustion engine components, brake rotors, drive shafts and heat sinks for microelectronic devices [1–4]. These kinds of composites offer the advantages such as high strength to weight ratio, higher stiffness, higher working temperature and higher electrical and thermal conductivity with respect to unreinforced metals and other composites such as polymer matrix composites (PMCs) [5].

Silver matrix composites are a group of materials which are widely applied as arcing contacts, automotive regulators, relays, controls, etc. The industrial application of metallic composites is determined by the type and volume fraction of the second phase, which can be metal or non-metal. Nickel, tungsten, molybdenum, graphite, tungsten carbide, tin and zinc oxide are some of metallic and non-metallic phases which have been used as the reinforcement in silver matrix composites [6–11]. These kinds of MMCs are mainly fabricated by powder metallurgy methods. Like the other engineering materials, which are manufactured by powder metallurgy processes, the relative density of silver matrix composites has significant influence on the other properties of the composite such as electrical and thermal conductivity. However, other parameters such as homogeneous fine distribution and size of the second phase as well as the type of reinforcement improve the wear resistance of silver matrix composites [12]. Silver matrix composites are produced by powder metallurgy methods. One of the methods is infiltration of porous skeleton of the second phase such as tungsten or graphite. The other one consists in pressing or sintering, or combination of both processes with application of subsequent process of secondary compaction or extrusion of the powders [6, 12].

In this research, synthesis of Ag – 8 wt. % ZnO composites was investigated. These composites are used as low voltage circuit breakers rated to 200 A [7]. The synthesis process included milling, heat treating and hot pressing of silver and zinc oxide powder mixtures. The effect of uniaxial hot pressing parameters such as pressure magnitude

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and duration of the process at 550 °C on the relative density of the obtained compacts was investigated.

Also, the microstrain and crystalline size of the milled and heat treated powders were determined by Debye-Scherrer and Williamson-Hall methods using the X-ray diffraction (XRD).

2. Experimental

Silver oxide (Ag_2O) and zinc oxide (ZnO) were used as starting powders. The premixed oxide powders were ball milled at the milling speed of 330 rpm for 20 h in air atmosphere. The milling vessel and balls were made of stainless steel. The ball to powder mass ratio was 13:1. The milled powders were heat treated in a tube type electrical furnace in argon atmosphere at 600 °C for 90 min. The powders were characterized by XRD technique. The microstrain and crystalline size of the milled and heat treated powders were determined by Debye-Scherrer and Williamson-Hall methods. The heat treated powders were hot pressed at three different conditions (Table 1).

Table 1. Three different hot pressing conditions of the heat treated powders.

Hot pressing condition	Temperature (°C)	Pressure (MPa)	Duration (min)
A	550	50	75
B	550	10	75
C	550	50	40

The density of the compacts was determined by geometrical method based on mass and dimensions of the samples. The theoretical density of Ag – 8 wt. % ZnO composites was determined using the following formula [13]:

$$\rho_{Ag-8\%wtZnO} = \frac{\rho_{Ag}\rho_{ZnO}}{\rho_{Ag}W_{ZnO} + \rho_{ZnO}W_{Ag}} \qquad (1)$$

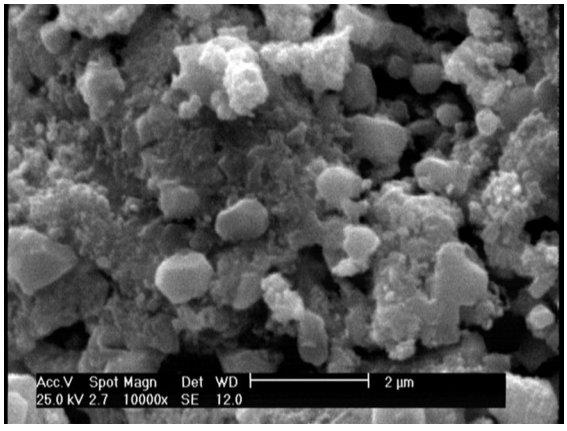
where ρ_{Ag} is density of silver, ρ_{ZnO} is density of zinc oxide and W_{Ag} and W_{ZnO} are mass fractions of silver and zinc oxide, respectively.

The microstructure of the powders and obtained compacts was analyzed by Scanning Electron Microscopy (SEM-PHILIPS-XL30). The hardness of the hot pressed samples was measured by a Rockwell hardness tester.

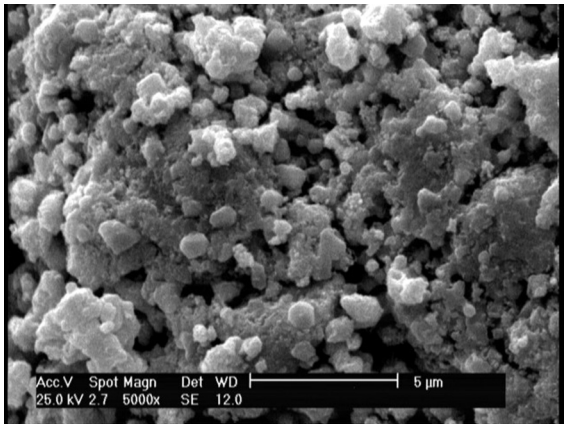
3. Results and discussion

Fig. 1 (a, b) shows the microstructure of the milled powders. The powder particles have semi-spherical and polygonal shapes and their size is mainly below 5 μm. Results of X-ray analysis of both milled and heat treated powders are shown in Fig. 2 and 3. As it can be seen, the milled and heat treated powders include Ag_2O/ZnO and Ag/ZnO, respectively. It was found out that silver oxide was reduced to silver during heat treating of the milled powders.

The crystallite size and microstrain of Ag_2O



(a)



(b)

Fig. 1. SEM micrographs of the milled powders.

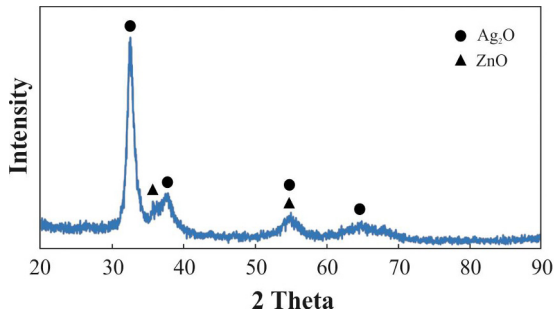


Fig. 2. XRD patterns of the milled powders.

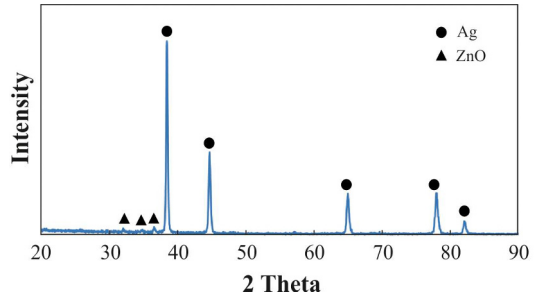


Fig. 3. XRD patterns of the heat treated powders.

and Ag were determined by Debye-Scherrer's formulae [14, 15]:

$$d = \frac{K\lambda}{\beta_S \cos\theta} \tag{2}$$

$$\eta = \frac{\beta}{4\tan\theta} \tag{3}$$

where d is the crystallite size, K shape factor (0.9), λ the wave length of CuK_α radiation (0.15404 nm), θ the Bragg angle, η the microstrain and β_S the sample broadening related to full width at half maximum (FWHM), which can be determined by correcting the measured broadening β_e using the following relation:

$$\beta_S^2 = \beta_e^2 - \beta_i^2 \tag{4}$$

where β_i is the instrumental broadening corresponding to each diffraction peak. Additionally, the crystallite size and microstrain of Ag were determined by Williamson-Hall method [15–17]:

$$\beta_S \cos\theta = \frac{K\lambda}{d} + 2\eta \sin\theta \tag{5}$$

Fig. 4 shows the plot of $\beta_S \cos\theta$ versus $2\sin\theta$ and the linear fit to the data. The crystallite size was determined from the y-intercept and the strain from the slope of the fit. Table 2 shows the microstrain and crystallite size of Ag_2O and Ag. As it can be seen the heat treating process causes a decrease of microstrain and an increase of crystallite size. The XRD patterns of the milled and heat treated samples were correlated with the calculated crystallite size.

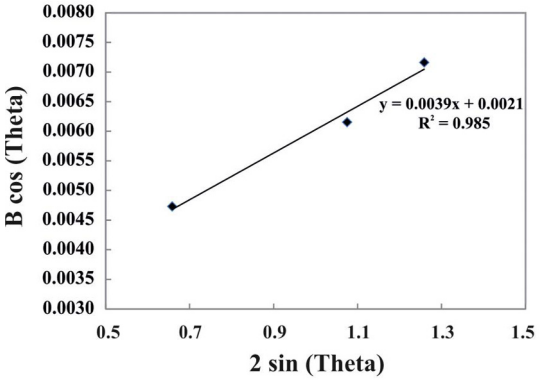


Fig. 4. Plot of $\beta_S \cos\theta$ versus $2\sin\theta$ and the linear fit to the data.

Table 3 shows the density and relative density of the hot pressed samples which were sintered in different conditions. The calculated theoretical density was 9.85 g/cm^3. It can be seen that the relative density of the samples which were hot pressed at 50 MPa for 75 min is the highest one among the others. Furthermore, the relative density of the samples of group (C) is higher than that of group (B). Generally, atomic diffusion and plastic flow are the main mechanisms which are responsible for densification of powder compacts during the hot pressing process. The role of atomic diffusion in densification is enhanced by increasing hot pressing temperature. Also, increasing the uniaxial pressure improves the effect of plastic flow on consolidation of the powder compacts. However, the applied uniaxial pressure should be higher than yield strength of the powders at sintering temperature [18].

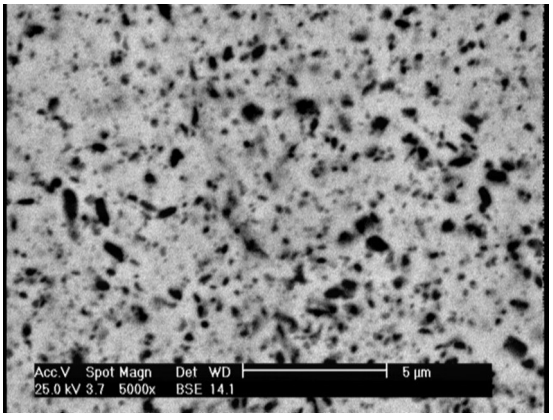
The samples of group (A) were hot pressed at 50 MPa which is higher than the yield strength of silver at 550 °C. So, it can be concluded that

Table 2. Crystallite size and microstrain of Ag_2O and Ag.

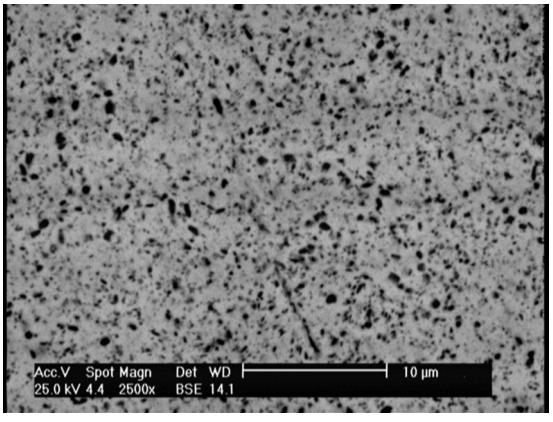
	Microstrain (%)		Crystallite size (nm)	
Constituent	Debye-Scherrer	Williamson-Hall	Debye-Scherrer	Williamson-Hall
Ag_2O	1.62	–	8.2	–
Ag	0.345	0.39	40.6	66.02

Table 3. Density and relative density of different samples.

Sample group code	Density (g/cm^3)	Relative density (%)
A	9.6	97.5
B	8.5	86
C	9.1	92



(a)



(b)

Fig. 5. Backscattered SEM image of hot pressed samples. The matrix phase is silver and the reinforcement phase is zinc oxide.

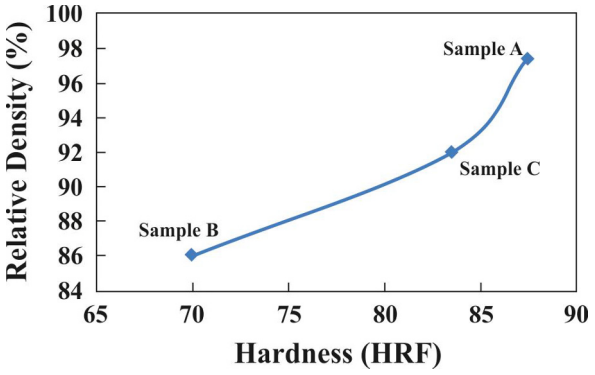


Fig. 6. Relative density versus hardness for the hot pressed compacts.

in these samples both atomic diffusion and plastic flow are responsible for densification of composite powders. On the other hand, the uniaxial hot pressing pressure of the samples of group (B) was 10 MPa which is lower than the yield point of silver at 550 °C [19]. Therefore, it can be assumed that plastic flow did not have an effective role in consolidation of group (B) specimens and atomic diffusion was the dominant densification mechanism in this group. Moreover, the relative densities of group (A) and (B) confirm that atomic diffusion is the main mechanism of densification during hot pressing process.

The difference of relative density between the sintered compacts of groups (A) and (C) reveals that duration of hot pressing is also a significant parameter for densification of the powder compacts and leads to some decrease of the volume percent of porosity within the microstructure of hot pressed samples. However, the higher relative density of the samples of group (C) rather than that of group (B) implies that the magnitude of hot pressing pressure is more effective than its duration for consolidation of the compacts.

A typical backscattered SEM image of the hot pressed powders is shown in Fig. 5. The white phase is silver and the black one is zinc oxide. Fig. 5 shows the fine and uniform distribution of ZnO particles within the silver matrix. This fine dispersion of the reinforcement phase within the matrix increases the efficiency of the electrical contact. Fig. 6 shows the hardness of different samples as a function of relative density for the hot pressed compacts. As it is shown, by increasing the density, the hardness is increased which is due to lower fraction of porosities within the microstructure of the samples with higher densities.

4. Conclusion

Silver/zinc oxide composites were synthesized by ball milling, heat treating and hot pressing of silver and zinc oxide powder mixtures and the following results were obtained:

1. Heat treating of the milled powders led to some decrease of the microstrain.
2. The pressure and duration of hot pressing process are important parameters which have significant influence on the relative density of the hot pressed compacts.
3. The main densification mechanism of A – 8 wt. % ZnO composite powders during hot pressing is atomic diffusion.

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