

The effect of reinforcement particle size on the properties of Cu-Al₂O₃ composites

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Abstract. The main aim of this paper was to investigate the effect of reinforcement particle size on the properties of Cu-Al₂O₃ composites. The starting materials for obtaining the composites were commercial powders: Cu and Al₂O₃. Experiments were performed using specimens containing 2.5, 5, 7.5 and 10 % of alumina. The average particle sizes of alumina were 3.5, 22.5, and 65 µm. Before the sintering process, single pressing was performed with a hydraulic press at a compaction pressure of 620 MPa. The obtained sinters were subjected to the compaction process at a pressure of 620 MPa, and then again sintered at 900 °C for 60 minutes. The sintering process was carried out for 60 minutes at a temperature of 900 °C. The sintered compacts were subjected to the following tests: measurement of density, hardness and electrical conductivity. Observations of the microstructure on metallographic specimens made from the sintered samples were also performed using a scanning electron microscope (SEM). The density of all the composites increased when the particle size of alumina was reduced. Higher hardness were observed in samples containing finer Al₂O₃ particles. The electrical conductivity of the fabricated composites increased with the reduction of the alumina particle size.

1. Introduction

In recent years, sintered copper matrix composites are increasing interest due to its very high electrical and thermal conductivity. It is well known that the introduction of even a small amount of alloy additives into copper results in a significant reduction in electrical conductivity. In order to increase the strength of copper without a significant decrease in electrical conductivity, among others particles such as metal oxides (Al₂O₃, SiO₂, ZrO₂, Cr₂O₃, BeO, MgO), carbides (SiC, TiC, Cr₇C₃, Cr₃C₂), nitrides and borides (TiB₂, ZrB₂, CrB₂, BN) are used like a reinforcement phases [1-3].

Research is also being carried out on the preparation of composites containing intermetallic phases, the introduction of which results in the increase of strength properties similar to those made with ceramic particles [4]. The composites reinforced with particles combine the properties of the metal matrix with the reinforcing properties of ceramics. Ceramic particles have high hardness and resistance to high temperatures, therefore, the composites reinforced with ceramic particles are also used for working at elevated temperatures. The introduction of ceramic particles into the matrix increases the hardness and abrasion resistance of the composite without a significant decrease in electrical conductivity. For this reason, copper-based composites reinforced with ceramic particles are mainly used in electronics and electrotechnics for relays, switches, elements of electric motors and for the tip of resistance welding electrodes [5-7]. Metal matrix composites reinforced with



ceramic particles are usually made using powder metallurgy techniques [8]. The main advantage of powder metallurgy compared to casting is better control of the distribution of the reinforcement in powder metallurgy compacts. Solid state diffusion, dependent on time and sintering temperature, plays a major role in powder metallurgy in the formation and growth of interparticle bonding. Currently, apart from the basic technique of making composites in powder metallurgy technology consisting in the preparation of mixtures, pressing and sintering, modern technologies such as hot pressing (HIP) and SPS (spark plasma sintering) are also used [9, 10]. The strength properties of composites with a metal matrix reinforced with particles depend, inter alia, on the retention properties of the matrix as well as on the size of the reinforcing particles. For composite materials, the strengthening factor depends on the diameter of the reinforcing particles [11, 12]. The best properties are obtained for dispersion-strengthened composites in which the size of strengthening particles is in the range from 0.01 μm to 0.1 μm [13-15]. In the case of particles larger than 1 μm , composites are reinforced with large particles. The main difference between dispersion and large particles is that in the case of composites reinforced with large particles, the loads are transferred both by the particles and the metal matrix. The aim of this work was to fabricate copper matrix composites reinforced with Al_2O_3 particles using powder metallurgy technology and to analyze the influence of strengthening particle size on their properties.

2. Experimental procedure

Alumina powder with the particle size of 3.5, 22.5 and 65 μm and 99.3 % purity from 0 to 10 wt. %, along with the copper powder with average 63 μm particle size and 99.5 % purity have been used. The density of copper powder was 8.9 g/cm^3 and the density of alumina powders was 3.95 g/cm^3 . Electron- microscopic images (SEM) of the starting powders are shown in Figure 1.

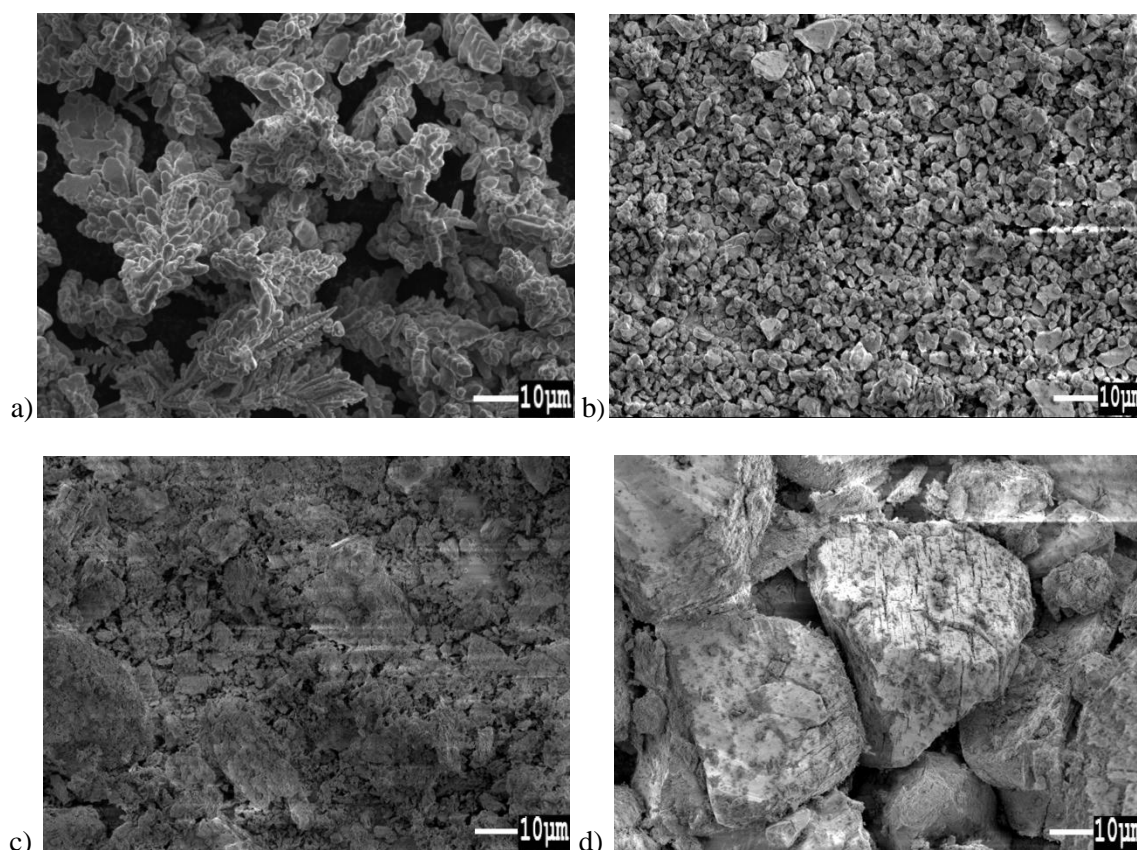


Figure 1. Images of the tested powders: a) electrolytic copper powder, b) alumina (3.5 μm particle size), c) alumina (22.5 μm particle size), d) alumina (65 μm particle size)

Before the fabrication of composites, the powders were observed using a JEOL JSM-7100F field emission scanning electron microscope. Powder mixtures with the following content of Al_2O_3 - 2.5 %, 5 %, 7.5 % and 10 % by weight were prepared for the tests. Finished powdered mixtures were subjected to single-track pressing on a hydraulic press at a compaction pressure of 620 MPa. The specimens with a dimension of $\Phi 20 \times 10$ mm were sintered in a silit tubular furnace at 900 °C for 60 minutes in the dissociated ammonia atmosphere. Finally, the material was cooled in the furnace. After the sintering process, the samples were subjected to compaction process at a pressure of 620 MPa and re-sintered at 900° for 60 minutes. The sintered compacts were measured for hardness, electrical conductivity, and density. The hardness of the composites was measured using the Brinell method (with a steel ball 5 mm in diameter at a load of 250 kG) in accordance with the PN EN ISO 6506-1:2014 standard. The electrical conductivity tests were performed using the GE Phasec 3D device using the eddy current method. The density was determined by weighing the specimens in air and water using WPA120 hydrostatic scales in line with the PN EN ISO 2738:2001 standard. Microstructure analysis on the metallographic specimens were conducted using the JEOL JSM-7100F field emission scanning electron microscope fitted with OXFORD INSTRUMENTS EDS X-Max Aztec software for elemental analysis.

3. Results and discussion

3.1. Microstructural investigations

The introduction of powdered alumina particles causes a distinct change in microstructure of obtained composites. Alumina particles of various sizes are very clear in a copper matrix. Differences in a particle size result from the range of the size of the introduced alumina powder. Mixing the powders of copper and alumina led to an even distribution of the particles in the matrix. In certain areas of composites, the alumina particles are bonded into longitudinal agglomerates. Large clusters of alumina particles were not observed. Introduced alumina particles have not dissolved in the matrix in the sintering process, which is associated with high thermal resistance of alumina particles. The examples of microstructures of fabricated materials containing 5% of alumina are shown in Figure 2.

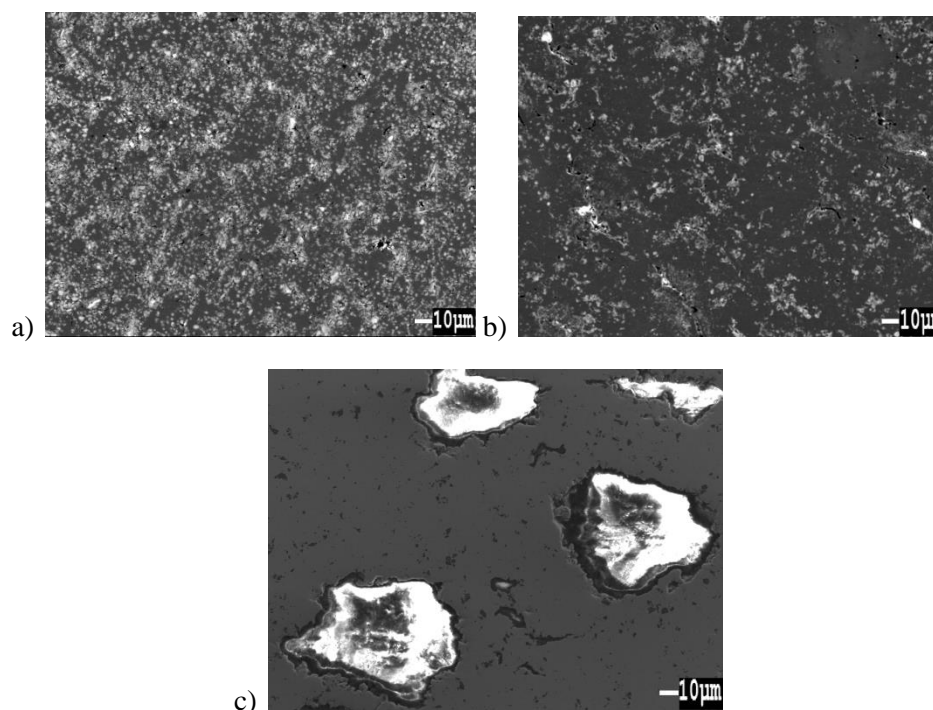


Figure 2. Microstructures of sintered compacts with a SEM obtained for a) Cu+5% of alumina(3.5 μm particle size), b) Cu+ alumina (22.5 μm particle size), c) Cu +alumina (65 μm particle size)

The presence and distribution of elements in a composite containing 5 % of alumina with a particle size of 22.5 μm are presented in Figure 3. From the mapping it is clear that the major elements occurring in a fabricated composites are copper, aluminum and oxygen.

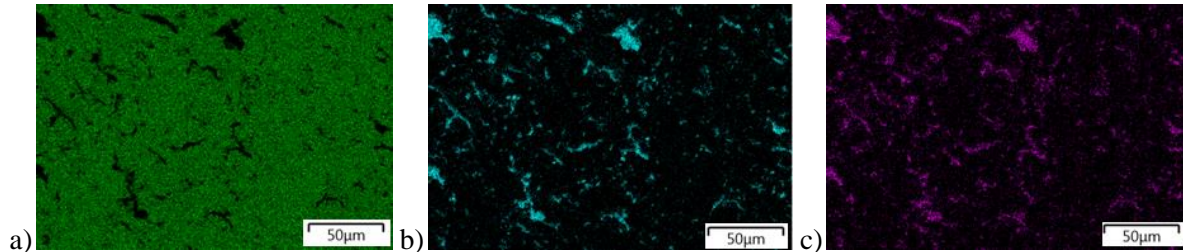


Figure 3. Distribution of elements in the micro- area of composite containing 5% of alumina with a average particle size of 22.5 μm , a) copper, b) aluminum, c) oxygen

3.2. Density and hardness measurements

The results of density and hardness measurements are presented in Tables 1-3.

Table 1. Results of the density and hardness measurements for a composites with a alumina average particle size of 3.5 μm

Material	Density (g/cm^3)	Relative density (%)	HB
Cu	8.19 \pm 0.02	92.01	36.65 \pm 1.5
Cu + 2.5 % of Al_2O_3	8.35 \pm 0.04	95.26	51.14 \pm 1.8
Cu + 5 % of Al_2O_3	7.98 \pm 0.05	93.66	51.90 \pm 1.3
Cu + 7.5 % of Al_2O_3	7.92 \pm 0.03	91.57	53.08 \pm 1.7
Cu + 10 % of Al_2O_3	7.73 \pm 0.07	92.03	54.29 \pm 1.8

Table 2. Results of the density and hardness measurements for a composites with a alumina average particle size of 22.5 μm

Material	Density (g/cm^3)	Relative density (%)	HB
Cu	8.19 \pm 0.02	92.01	36.65 \pm 1.5
Cu + 2.5 % of Al_2O_3	8.04 \pm 0.01	91.62	46.53 \pm 1.6
Cu + 5 % of Al_2O_3	7.97 \pm 0.04	92.23	48.58 \pm 1.3
Cu + 7.5 % of Al_2O_3	7.84 \pm 0.03	92.05	50.76 \pm 1.5
Cu + 10 % of Al_2O_3	7.51 \pm 0.06	89.49	51.90 \pm 1.2

Table 3. Results of the density and hardness measurements for a composites with a alumina average particle size of 65 μm

Material	Density (g/cm^3)	Relative density (%)	HB
Cu	8.19 \pm 0.02	92.01	36.65 \pm 1.5
Cu + 2.5 % of Al_2O_3	7.97 \pm 0.02	90.91	45.56 \pm 1.2
Cu + 5 % of Al_2O_3	7.68 \pm 0.05	88.87	47.54 \pm 1.7
Cu + 7.5 % of Al_2O_3	7.60 \pm 0.04	89.18	49.66 \pm 1.9
Cu + 10 % of Al_2O_3	7.41 \pm 0.01	88.23	50.76 \pm 1.5

The examination showed that, the introduction of alumina particles into the copper matrix increases the hardness of the materials, however, these changes are small. The increasing of Al_2O_3 content (hard

ceramic particles) with its homogeneous dispersion are the main reason for the increasing of hardness [5-8]. The highest hardness was obtained for the composite containing 10 % of alumina with average particle size of 3.5 μm which amounted 54 HB (47% higher than composite made of pure copper). Along with the increase in particle size, the hardness of composites decreases. The difference in hardness between composites containing 10% aluminum oxide with an average particle size of 3.5 μm and 65 μm was almost 4 HB. Analyzing the obtained density results, it should be stated that the introduction of alumina particles decreases density of materials. Along with the increase in the content of alumina and the increase in the size of strengthening particles the density decreases. The highest density was obtained for a composite containing 2.5 % of alumina with average particle size of 3.5 μm which amounted 8.35 g/cm^3 . The obtained result is caused by filling the voids of the metal matrix with fine particles of alumina. Similar results were received by other researchers [1-3].

3.3. *Electrical conductivity of the sintered compacts*

The results of electrical conductivity test are shown in Figure 4.

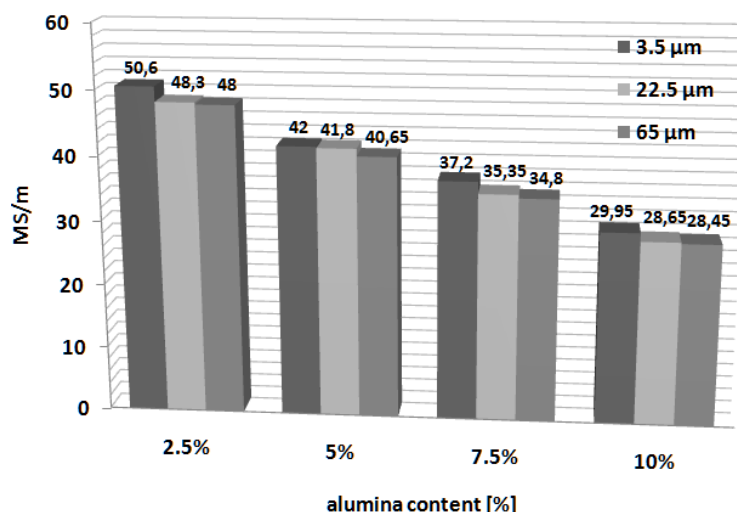


Figure 4. The results of electrical conductivity measurements of copper-alumina composites depending on amount and average particle size of alumina

The examination showed that the introduction of alumina particles into copper matrix caused decrease in electrical conductivity. This phenomenon is caused by the fact that even a small amount of alloy additives in copper causes a reduction of their electrical conductivity. Many researchers postulated that the electrical conductivity is affected by impurities. By taking into account that Al_2O_3 acts as impurities in copper matrix, so the electrical conductivity might decrease [13]. The highest electrical conductivity was obtained for a material containing 2.5 % of alumina with a average particle size of 3.5 μm which amounted 50.6 MS/m (13% less than pure copper in a solid state). Along with the increase in the content of alumina and the increase in the size of strengthening particles the electrical conductivity decreases. These results agree, to a good extent, with those reported in [14,15].

4. Conclusion

The analysis of the microstructure of the composites showed that the alumina powder particles combine into agglomerates. Before sintering, powders should be mixed thoroughly to break up the agglomerates and obtain a homogeneous mixture.

The sintering parameters were chosen correctly based on previous research and compared with the results available in the literature. The microstructural examinations showed that there were no discontinuities at the interface between the matrix and the ceramic particles. A very good bonding of alumina particles with the copper matrix was obtained, without voids, only the pores occurring

in sintered metals were visible on the micrographs. Alumina particles were clearly visible in the form of irregular precipitates. The introduction of alumina particles resulted in an increase in the hardness of the composites and a decrease in the density and electrical conductivity. Higher hardness and density were observed in samples containing finer Al_2O_3 particles. An addition of Al_2O_3 and increasing the particle size of alumina as well as the amount of reinforcement increase porosity. The best properties were obtained for composites reinforced with particles of average size $3.5\ \mu\text{m}$.

5. References

- [1] Konieczny M, 2012, The effect of sintering temperature, sintering time and reinforcement particle size on properties of Al- Al_2O_3 composites, *Composites Theory and Practice*, **vol.12:1**, pp 39-43.
- [2] Taha M A and Zawrah M F, 2017, Effect of nano ZrO_2 on strenghtening and electrical properties of Cu-matrix nanocomposites prepared by mechanically alloying, *Ceramics International*, **vol. 43**, pp 12698-704.
- [3] Shojaeepour F, Abachi P, Purazrang K and Moghanian A H, 2012, Production and properties of Cu/ Cr_2O_3 nano-composites, *Powder Technology*, **vol.222**, pp 80-84.
- [4] Konieczny M, 2013, Mechanical properties of Ti-($\text{Al}_3\text{Ti}+\text{Al}$) and Ti- Al_3Ti laminated composites, *Composites Theory and Practice*, **vol.13**, pp 102-106.
- [5] Rajkovic V, Bozic D, Stasic J, Wang H and Jovanovic M T, 2014, Processing, characterization and properties of copper-based composites strengthened by low amount of alumina particles, *Powder Technology*, **vol. 268**, pp 392-400.
- [6] Rajkovic V, Bozic D, Devecerski A and Jovanovic M T, 2012, Characteristics of copper matrix simultaneously reinforced with nano- and micro-sized Al_2O_3 particles, *Materials Characterization*, **vol. 67**, pp 129-137.
- [7] Rajkovic V, Bozic D, Popovic M and Jovanovic M T, 2009, The influence of powder particle size on properties of Cu- Al_2O_3 composites, *Science of Sintering*, **vol.41**, pp 185-192.
- [8] Borowiecka- Jamrozek J, Kargul M, Borowiecki S, Engel Z, 2018, Wpływ dodatku naturalnego zeolitu na mikrostrukturę i własności mechaniczne spiekanego kompozytu o osnowie żelaza, *Rudy i metale nieżelazne*, **vol. 63:7**, pp 12-17.
- [9] Martinez C, Briones F, Rojas P, Aguyilar C, Guzman D and Ordonez S, 2017, Microstructural and mechanical characterization of copper, nickel and Cu-based alloys obtained by mechanical alloying and hot pressing, *Materials Letters*, **vol.209**, pp 509-512.
- [10] Zhou D, Wang X, Muransky O, Wang X, Xie Y, Yang C, Zhang D, 2018, Heterogeneous microstructure of an Al_2O_3 dispersion strengthened Cu by spark plasma sintering and extrusion and its effect on tensile properties and electrical conductivity, *Materials Science and Engineering A*, **vol.730**, pp 328-335.
- [11] Rahimian M, Parvin N and Ehsani N, 2010, Investigation of particle size and amount of alumina on microstructure and mechanical properties of Al matrix composite made by powder metallurgy, *Materials Science and Engineering A*, **vol. 527**, pp 1031-38.
- [12] Rahimian M, Ehsani N, Parvin N and Baharvandi H R, 2009, The effect of particle size, sintering temperature and sintering time on the properties of Al- Al_2O_3 composites, made by powder metallurgy, *Journals of Materials Processing Technology*, **vol. 209**, pp 5387-93.
- [13] Hussain Z and Keong K H, 2005, Studies on alumina dispersion-strengthened copper nanocomposite through ball milling and mechanical alloying method, *J. Teknol.*, **vol. 43**, pp 1-10.
- [14] Shi Z and Yan M, 1998, The preparation of Al_2O_3 - Cu nanocomposite by internal oxidation, *Appl. Surf. Sci.*, **vol. 134**, pp 103-106.
- [15] Sable M J and Tripathi V K, 2011, Fabrication and study of optimum properties of copper alumina metal matrix nanocomposites for high temperature applications, *Ind. Eng. Technol.*, **vol.3:2**, pp 143-154.