

Fabrication and characteristics of sintered copper-silica composites

M Kargul¹, J Borowiecka- Jamrozek¹, M Konieczny¹

¹Department of Metal Science and Materials Technologies, Faculty of Mechatronics and Mechanical Engineering, Kielce University of Technology, Al. Tysiąclecia P.P. 7, 25-314 Kielce, Poland

E-mails: mkargul@tu.kielce.pl, jamrozek@tu.kielce.pl, mkon@tu.kielce.pl

The paper presents the results of research on the production of a sintered Cu-SiO₂ composites fabricated by powder metallurgy. The starting materials for obtaining the composites were commercial powders: Cu and SiO₂. SiO₂ particles were introduced into a copper matrix in the amount of 2.5 %, 5 %, 7.5 % and 10 % by weight. Finished powders mixtures were subjected to single pressing with a hydraulic press at a compaction pressure of 620 MPa and then subjected to sintering process at 900 °C in an atmosphere of dissociated ammonia. The sintering time was 60 minutes. The sintered compacts were subjected to the following tests: hardness, measurement of density, electrical conductivity and abrasion resistance. The highest density was obtained for a composite containing 2.5 % SiO₂ which was 7.94 g/cm³. The highest hardness was obtained for a composite containing 10 % SiO₂ which amounted to 50 HB. For the composite containing 2.5 % SiO₂ the highest electrical conductivity was obtained which was 47 MS/m. The highest abrasive wear resistance was obtained for a composite containing 10 % SiO₂. Both density and electrical conductivity decreased along with the increase of SiO₂ content in composites.

1. Introduction

Currently, research is being carried out on the production of modern materials in order to achieve the desired properties. A single material generally does not have satisfactory properties, therefore the need for composites with specific properties increases every day. In recent years, sintered copper-matrix composites are finding an increasing interest due to its very high electrical and thermal conductivity [1-6]. It is well known that the inclusion of even a small amount of copper alloying elements results in a significant decrease in electrical conductivity. In order to increase the strength of copper without a significant decrease in electrical conductivity, ceramic particles such as Al₂O₃, SiO₂, ZrO₂, Cr₂O₃, BeO, MgO as well as carbides (SiC, TiC, Cr₇C₃, Cr₃C₂) and borides and nitrides (TiB₂, ZrB₂, CrB₂, BN) are used [7-13]. By using of ceramic particles as a strengthening phase, diffusion phenomenon does not occur between the metal matrix and reinforcing particles. As a result, composites with electrical conductivity similar to pure copper are obtained. 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 [14-16]. The composites reinforced with ceramic 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 metal matrix increases the hardness and abrasion resistance of the composite. Therefore, the copper matrix composites reinforced with ceramic particles are mainly used in electronics and electrical engineering for relays, switches, elements of electric motors and for the tip of electrodes for resistance welding. Metal matrix composites reinforced with ceramic particles are usually made using powder metallurgy. The production of composites by the powder metallurgy method consists in preparing a powder mixture, powder compaction and then sintering. Research is also being carried out on the production of metal matrix composites using the hot pressing method and also using the SPS (spark plasma sintering) technique [17-21]. In this work, an attempt was made to produce a sintered composite with a copper matrix in which SiO_2 powder was used as strengthening particles. The obtained composites were subjected to microstructural investigations and the density, hardness, electrical conductivity and abrasion resistance were examined.

2. Materials and methods

The materials used in the experiment to fabricate the composites were commercial powders. Copper powder (99.5 % Cu) with an average particle size of less than $63\ \mu\text{m}$ was used as the matrix while silica (96.5 % SiO_2), with an average particle size of less than $45\ \mu\text{m}$ was used as the reinforcement. The shapes and arrangements of the powder particles used in the experiments are shown in Figure 1.

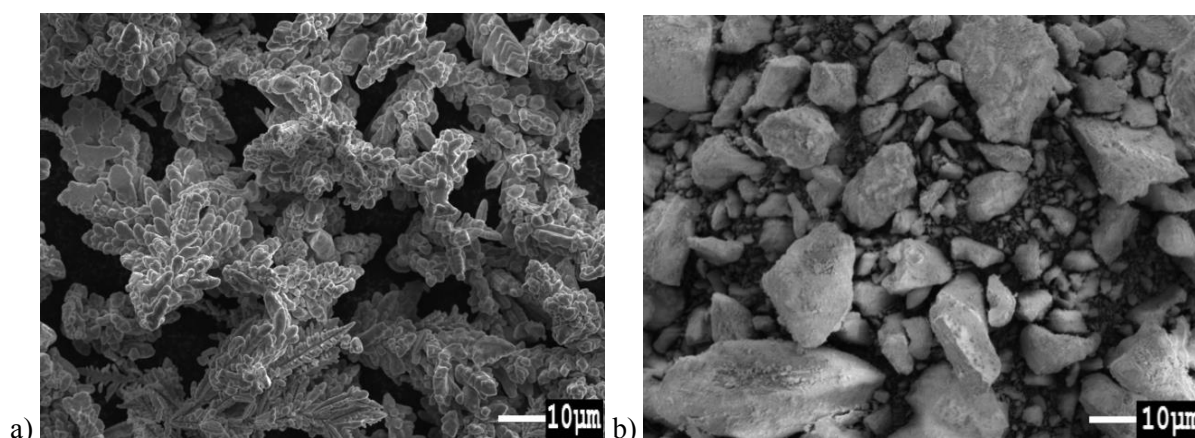


Figure 1. Images of the tested powders: a) electrolytic copper powder, b) silica powder.

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 SiO_2 - 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 $\Phi\ 20 \times 10\ \text{mm}$ were sintered in a silit tubular furnace at $900\ ^\circ\text{C}$ 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 $900\ ^\circ\text{C}$ for 60 minutes. The sintered compacts were measured for hardness, electrical conductivity, density and abrasion resistance tests. 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. The abrasion resistance tests of the fabricated composites was conducted using device with a roll on block configuration. The roller was made of hardened 100Cr6 steel with a hardness of

62 HRC. The blocks with a dimension of 6.35x10x15 mm were made of Cu/SiO₂ composites. The test was performed with a rotational speed of 300 rpm with a load of 5 N, while the friction path was set as 3000 m. The blocks were weighed before and after the tests to determine the loss of weight. 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 characterization

The introduction of powdered silica causes a distinct change in microstructure of obtained composites. Silica 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 silica powder (<45 µm). Mixing the powders of copper and silica led to an even distribution of the particles in the matrix. In certain areas of composites, the silica particles are bonded into longitudinal agglomerates. Large clusters of silica particles were not observed. Introduced silica particles have not dissolved in the matrix in the sintering process, which is associated with high thermal resistance of silica. The microstructure of fabricated materials is shown in Figure 2.

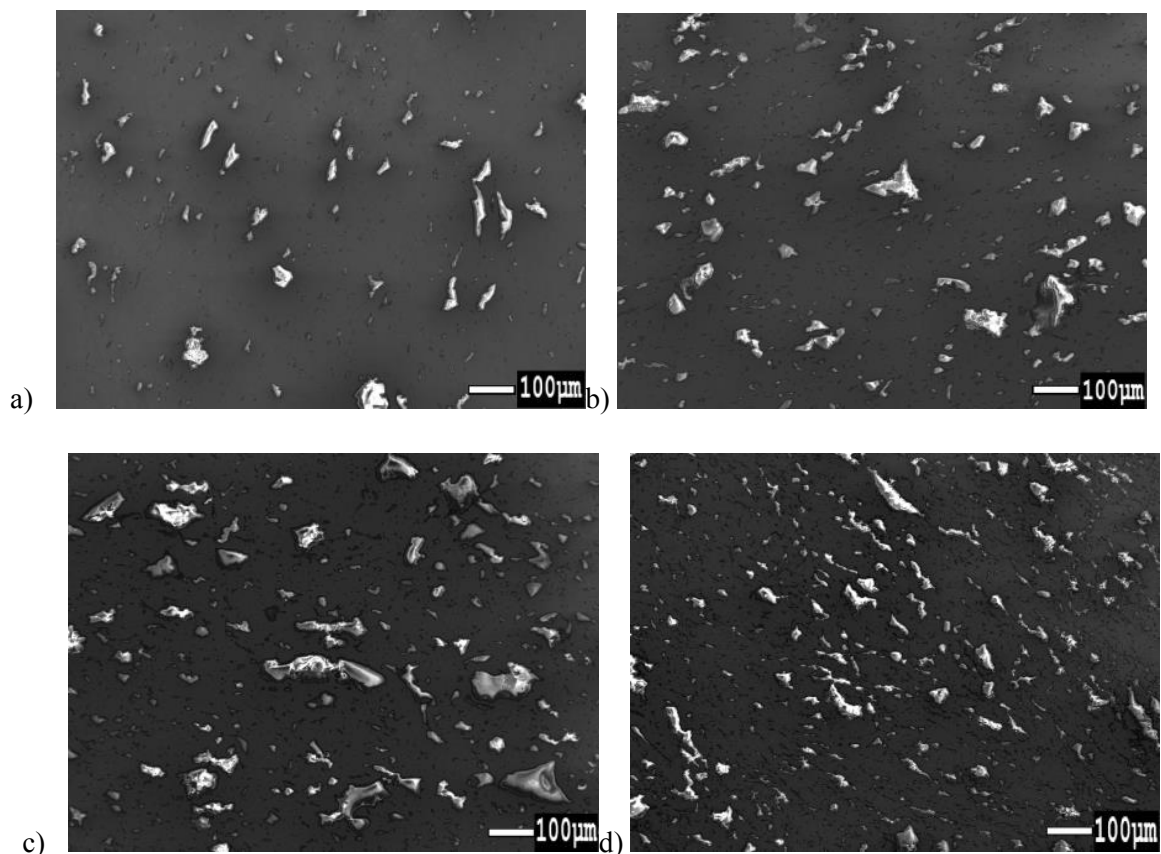


Figure 2. Microstructures of the sintered compacts observed with a SEM obtained for a) Cu+2.5 % silica, b) Cu+ 5 % silica, c) Cu+7.5 % silica d) Cu+10 % silica

An example of the distribution of elements in a composite containing 10 % SiO₂ is shown in Figure 3. From the mapping it is clear that the composite contains only copper, silicon and oxygen.

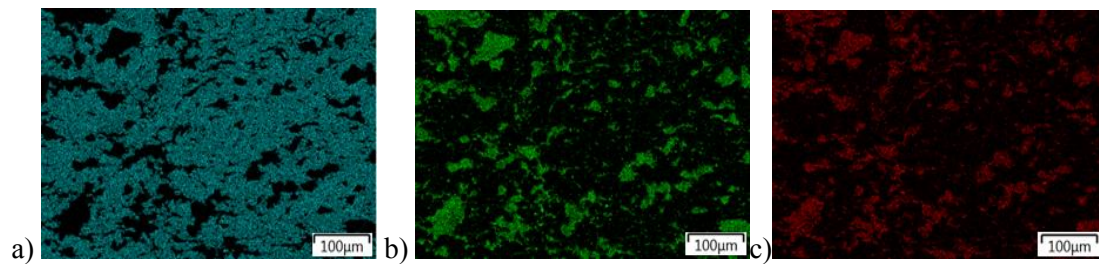


Figure 3. Distribution of elements in the micro-area of the composite containing 10% SiO₂, a) copper, b) silicon, c) oxygen

3.2. Density and hardness measurements

The results of density and hardness measurements are presented in Table 1.

Table 1. Results of the density and hardness measurements

Material	Density (g/cm ³)	Relative density (%)	HB
Cu	8.19± 0.02	92.01	36.65± 1.5
Cu + 2.5 % of SiO ₂	7.95± 0.04	90.88	44.61± 1.8
Cu + 5 % of SiO ₂	7.29± 0.05	85.78	45.56± 1.3
Cu + 7.5 % of SiO ₂	7.23± 0.03	84.93	48.59± 1.7
Cu + 10 % of SiO ₂	6.73± 0.07	81.31	49.66± 1.8

The examination showed that, the introduction of silica particles into the copper matrix increases hardness of the composites, while reducing the density. This is due to the high hardness and low density of silica particles. The highest hardness was obtained for the composite containing 10 % of silica which amounted 50 HB (36% higher than composite made of pure copper). The same phenomenon was also noticed by other researchers [2, 4, 7, 9, 12]. The highest density was obtained for a composite made from pure copper which amounted 8.19 g/cm³. Addition 10 % of silica particles caused decrease in density to 6.73 g/cm³.

3.3. Electrical conductivity test

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

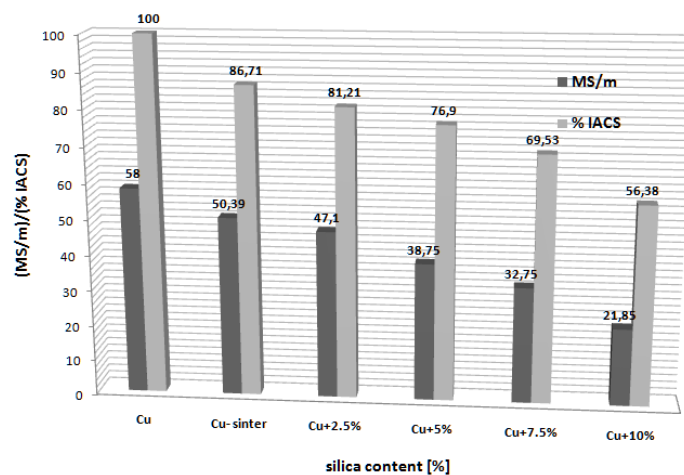


Figure 4. The results of electrical conductivity measurements of copper-silica composites

The examination showed that the introduction of silica 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. The highest electrical conductivity was obtained for a composite made from copper powder which amount 50.39 MS/m (14 % less than pure copper in a solid state). Similar results were received by other researchers [2, 4, 5]. The addition of 2.5 % of silica particles caused decrease in electrical conductivity to 47.1 MS/m but addition of 10 % of silica particles caused decrease in electrical conductivity to 21.85 MS/m.

3.4. Wear characterization

Weight loss of composites after tribological test are shown in Figure 5.

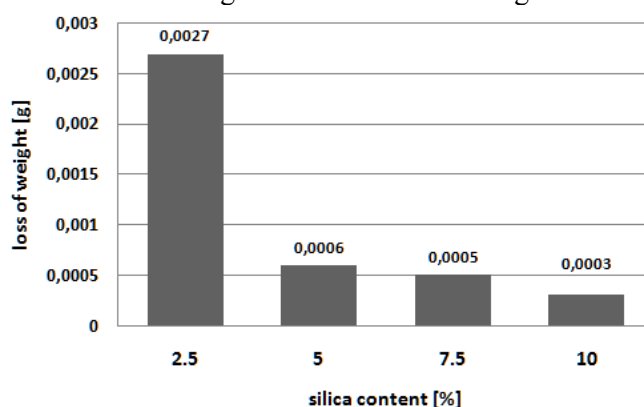


Figure 5. Weight loss of composites depending on the content of silica

Tribological tests has shown that the addition of silica particles increasing wear resistance of composites. The smallest loss of weight was observed for the composite containing 10% of silica.

4. Conclusion

The analysis of the microstructure of the composites showed that the silica 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 silica particles with the copper matrix was obtained, without voids, only the pores occurring in sintered metals were visible on the micrographs. Silica particles were clearly visible in the form of irregular precipitates. The introduction of silica particles resulted in an increase in the hardness of the composites and a decrease in the density and electrical conductivity. Tribological tests have shown that the introduction of silica particles increases abrasion resistance.

5. References

- [1] Gao F, Fu R, Song B Y, Xu Z Y and Berthier Y, 2009, Effects of speed sequence on friction properties of sintered Cu-SiO₂, *Tribology- Materials, Surfaces & Interfaces*, **vol. 3:1**, pp 37-40.
- [2] Mikula J, Łach M and Kowalski J S, 2015, Copper matrix composites reinforced with volcanic tuff, *Metallurgija*, **vol. 54**, pp 143-146.

- [3] Xuehui Z, Chenguang L, Shun C, Zengde L, 2016, Characteristics of nano-alumina particles dispersion strengthened copper fabricated by reaction synthesis, *Rare Metal Materials and Engineering*, **vol.45:4**, pp 893-896.
- [4] Lee D W and Kim B K, 2004, Nanostructured Cu-Al₂O₃ composite produced by thermochemical process for electrode application, *Materials Letters*, **vol.58**, pp 378-383.
- [5] Shi Z and Yan M, 1998, The preparation of Al₂O₃-Cu composite by internal oxidation, *Applied surface science*, **vol.134**, pp 103-106.
- [6] Kruger C and Mortensen A, 2013, In situ copper–alumina composites, *Materials Science and Engineering A*, **vol.585**, pp 396-407.
- [7] Borowiecka- Jamrozek J, Kargul M, Borowiecki S, Engel Z, 2018, Wpływ dodatku naturalnego zeolitu na mikrostrukturę i własności mechaniczne spiekane go kompozytu o osnowie żelaza, *Rudy i metale nieżelazne*, vol. 63:7, pp 12-17.
- [8] Li J F, Chen B, Tang H, Zhang S and Li C, 2017, Tribological behavior of Cu- based composites with NbSe₂ and TiB₂, *Digest Journal of Nanomaterials and Biostructures*, **vol.43**, pp 1205-14.
- [9] Taha M A and Zawrah M F, 2017, Effect of nano ZrO₂ on strengthening and electrical properties of Cu-matrix nanocomposites prepared by mechanical alloying, *Ceramics International*, **vol.43**, pp 12698-704.
- [10] Samal C P, Parihar J S and Chaira D, 2013, The effect of milling and sintering techniques on mechanical properties of Cu-graphite metal matrix composite prepared by powder metallurgy route, *Journal of Alloys and Compounds*, **vol. 569**, pp 95-101.
- [11] Li J F, Chen B, Tang H, Zhang S and Li C, 2017, Tribological behavior of Cu- based composites with NbSe₂ and TiB₂, *Digest Journal of Nanomaterials and Biostructures*, **vol.43**, pp 1205-14.
- [12] Taha M A and Zawrah M F, 2017, Effect of nano ZrO₂ on strengthening and electrical properties of Cu-matrix nanocomposites prepared by mechanical alloying, *Ceramics International*, **vol.43**, pp 12698-704.
- [13] Samal C P, Parihar J S and Chaira D, 2013, The effect of milling and sintering techniques on mechanical properties of Cu-graphite metal matrix composite prepared by powder metallurgy route, *Journal of Alloys and Compounds*, **vol. 569**, pp 95-101.
- [14] Konieczny M and Mola R, 2007, Sintered copper matrix composites containing aluminium-ferrie intermetallic phases, *Composites Theory and Practice*, **vol.7:2**, pp. 109-113.
- [15] Konieczny M, 2013, Mechanical properties of Ti-(Al₃Ti+Al) and Ti-Al₃Ti laminated composites, *Composites Theory and Practice*, **vol.13**, pp 102-106.
- [16] Karakulak E, 2017, Characterization of Cu-Ti powder metallurgical materials, *International Journal of Minerals, Metallurgy and Materials*, **vol.24:1**, pp 83-90.
- [17] 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.
- [18] Zhou D, Wang X, Muransky O, Wang X, Xie Y, Yang C, Zhang D, 2018, Heterogeneous microstructure of an Al₂O₃ 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.
- [19] Oanh N T H, Viet N H, Kim J S, and Junior A M J, 2017, Characterization of in-situ Cu-TiH₂ and Cu-Ti-C nanocomposites produced by mechanical milling and spark plasma sintering, *Metals*, **vol.7:4**, pp 117.
- [20] Borowiecka-Jamrozek J, 2013, Engineering structure and properties of materials used as a matrix in diamond impregnated tools, *Archives of Metallurgy and Materials*, **vol. 58:1**, pp 5-8.
- [21] Borowiecka-Jamrozek J and Konstanty J, 2014, Microstructure and Mechanical Properties a New Iron-base Material Used for the Fabrication of Sintered Diamond Tools, *Advanced Materials Research*, **vol. 1052**, pp 520-523, doi:10.4028/www.scientific.net/AMR.1052.520.