

# THE INFLUENCE OF THE COMPACTION PROCESS ON THE PERFORMANCE CHARACTERISTICS OF SINTERED COPPER- AI<sub>2</sub>O<sub>3</sub> COMPOSITES

Marcin KARGUL, Joanna BOROWIECKA-JAMROZEK, Marek KONIECZNY

Department 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, EU mkargul@tu.kielce.pl

#### **Abstract**

This paper presents the results of research on the possibilities of producing and using Cu /  $Al_2O_3$  composite material fabricated by powder metallurgy. The starting materials for obtaining the composites were commercial powders: Cu and  $Al_2O_3$ . The quality of the combination of aluminum oxide with the matrix and the influence of aluminum oxide particles on sinter properties were also evaluated. Alumina was introduced into a copper matrix in the amount of 2.5 %, 5 %, 7.5 % and 10 % by weight. Before the sintering process, single pressing was performed with a hydraulic press at a compaction pressure of 620 MPa. The sintering process was carried out for 60 minutes at a temperature of 900 °C. 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 sintered compacts were subjected to the following tests: measurement of density, hardness, electrical conductivity and abrasion resistance. Observations of the microstructure on metallographic specimens made from the sintered samples were also performed using a scanning electron microscope (SEM). The process of compaction of the fabricated composite caused an increase in the hardness, density, electrical conductivity and abrasion resistance in comparison to a composite subjected to only the pressing and sintering process. Both the density and electrical conductivity decreased with the increase of  $Al_2O_3$  content in composites after sintering and after the compaction process.

**Keywords:** Metal composite, sintering, alumina, copper, powder metallurgy

## 1. INTRODUCTION

Composites are a very large and diverse group of construction materials. Among metal composite materials, composites reinforced with ceramic particles deserve special attention. Recently, research has been focused on developing novel composites with a metal matrix reinforced with ceramic particles that show high hardness and good electrical conductivity [1-3]. The literature review shows [4-8] that in recent years sinter-coppermatrix composites in which Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> were commonly used as reinforcing particles. Other metal oxides such as ZrO<sub>2</sub>, Y<sub>2</sub>O<sub>3</sub>, Cr<sub>2</sub>O<sub>3</sub> and Er<sub>2</sub>O<sub>3</sub> work perfectly as the reinforcing particles [9-11].

Copper alloys are widely used for electrical and electronic components such as switches in low-voltage devices, aircraft relays, motor starters and circuit breakers, resistance welding electrodes and also structural elements in reactor technology [12]. They are also used in many other industries, including the automotive industry and hydraulics. It is a well-known fact that introduction of even a small amount of additives lead to a considerable decrease in electrical conductivity of copper. In order to increase the strength of copper without significantly losing its electrical properties, composites based on the effect of particle curing are produced. The introduction of second phase dispersive particles not only increases the mechanical properties, but also allows the use of these composites for operation at elevated temperatures. The introduction of ceramic particles into the matrix increases also the hardness and wear-resistance [13].

In this work an attempt was made to make a Cu-based composite with the participation of Al<sub>2</sub>O<sub>3</sub> particles by powder metallurgy involving mechanical mixing of combined materials, cold compaction, and then heat consolidation and examination of their microstructure and properties.



#### 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  $\mu$ m was used as the matrix while alumina (98.5 % Al<sub>2</sub>O<sub>3</sub>), with an average particle size of less than 45  $\mu$ m was used as the reinforcement. The shapes and arrangements of the powder particles used in the experiments are shown in **Figure 1**.

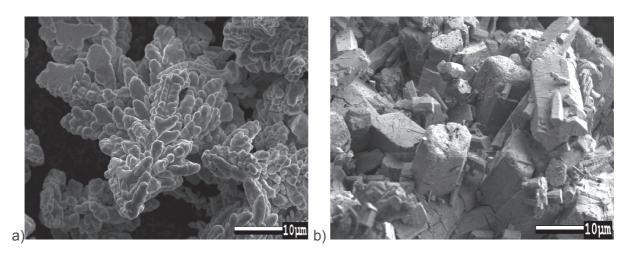


Figure 1 Images of the tested powders: a) electrolytic copper powder, b) alumina powder

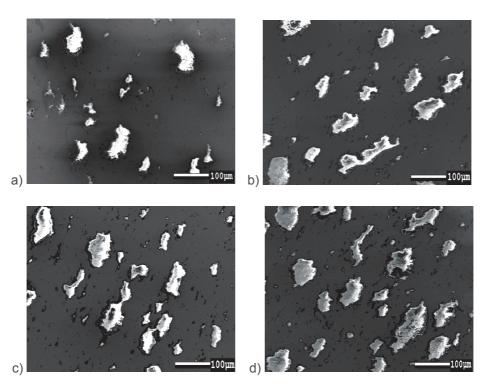
Powder mixtures with the following content of Al<sub>2</sub>O<sub>3</sub> - 2.5 %, 5 %, 7.5 % and 10 % by weight were prepared for the tests. Finished powdered mixtures were subjected to single-track pressing on a hydraulic press at a pressure of 620 MPa. The tests were conducted on cylindrical specimens with dimensions of Φ20x10 mm. The first batch of samples was subjected to a sintering process in a sillit tubular furnace at 900 °C in the dissociated ammonia atmosphere. The sintering time was 60 minutes. The samples were cooled in the furnace. The second batch of samples was made using the same parameters as before, however, after the sintering process, the samples were subjected to compaction and re-sintering. The fabricated composites were measured for density, hardness, electrical conductivity and abrasion resistance tests. Density measurement was carried out with the air and water weighing method using a WPA 120 hydrostatic scales in accordance with the PN-EN ISO 2738:2001 standard. The hardness of the obtained composites was measured using the Brinell method (with a steel ball 5 mm in diameter at a load of 250 kg) according to PN-EN ISO 6506-1:2014 standard. Next, microstructure investigations on the metallographic specimens were carried out using the JEOL JSM-7100F field emission scanning electron microscope fitted with OXFORD INSTRUMENTS EDS X-Max AZtec software for elemental microanalysis. The electrical conductivity tests were carried out using the GE Phasec 3D device using the eddy current method. The abrasion resistance test of the produced composites was carried out using T-05 Tester with a roll on block configuration. The roller was made of hardened C45 steel with a hardness of 52 HRC, while the blocks were made of Cu/Al<sub>2</sub>O<sub>3</sub> composites. The test was carried out with a rotational speed of 200 rpm and a load of 196.2 N. The friction path was set at 200 m. The samples were weighed before and after the tests to determine the loss of weight.

## 3. RESULTS AND DISSCUSSION

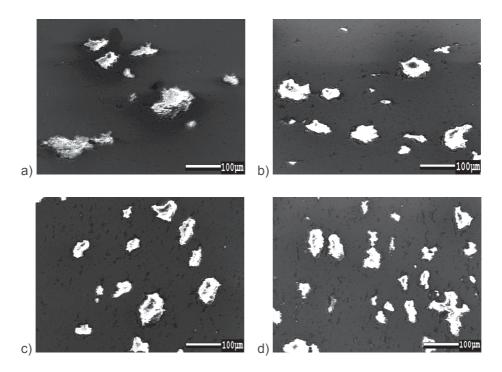
# 3.1. Microstructural characteristics

The optimal parameters of the sintering process were selected on the basis of the authors previous findings and similar data available in the literature [1-3]. The microstructure of the composites obtained after the pressing and sintering process is shown in **Figure 2**, and after the pressing, sintering and compacting process in **Figure 3**.





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

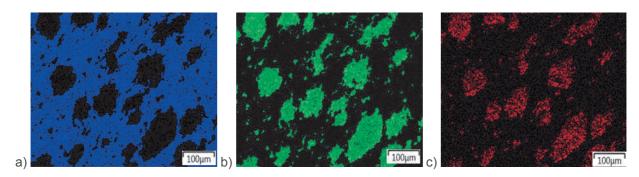


**Figure 3** Microstructures of the sintered compacts after compacting observed with a SEM obtained for a) Cu + 2.5 % alumina, b) Cu + 5 % alumina, c) Cu + 7.5 % alumina d) Cu + 10 % alumina

The distribution of the reinforcing particles is uniform and the quality of bonding with the matrix is relatively high. Analysis of the microstructure (**Figures 2** and **3**) of the obtained composites showed that the Al<sub>2</sub>O<sub>3</sub> powder particles combine into agglomerates. Before sintering, powders should be mixed thoroughly to break up the agglomerates and obtain a homogeneous mixture. Uniform distribution of Al<sub>2</sub>O<sub>3</sub> particles in the matrix



will result in the same properties in the entire volume of the composite. An example of the distribution of elements in a composite containing 10% Al<sub>2</sub>O<sub>3</sub> is shown in **Figure 4**. From the mapping it is clear that the composite contains only copper, aluminum and oxygen.



**Figure 4** Distribution of elements in the micro-area of the composite containing 10% Al<sub>2</sub>O<sub>3</sub>, a) copper, b) aluminum, c) oxygen

# 3.2. Density and hardness measurements

The results of density and hardness measurements are presented in **Tables 1** and **2**.

Table 1 Results of the density and hardness measurements after sintering

| Material                                  | Density (g/cm³) | Relative density (%) | НВ          |
|---|-----------------|----------------------|-------------|
| Cu  | 7.98 ± 0.02     | 89.67                | 36.65 ± 1.5 |
| Cu + 2.5 % Al <sub>2</sub> O <sub>3</sub> | 7.89 ± 0.03     | 89.95                | 46.87 ± 1.8 |
| Cu + 5 % Al <sub>2</sub> O <sub>3</sub>   | 7.54 ± 0.01     | 87.28                | 40.23 ± 1.3 |
| Cu + 7.5 % Al <sub>2</sub> O <sub>3</sub> | 7.40 ± 0.02     | 86.88                | 37.63 ± 1.7 |
| Cu + 10 % Al <sub>2</sub> O <sub>3</sub>  | 7.36 ± 0.04     | 87.68                | 34.36 ± 1.8 |

Table 2 Results of density and hardness measurements after compacting

| Material                                  | Density (g/cm³) | Relative density (%) | НВ          |
|---|-----------------|----------------------|-------------|
| Cu  | 8.19 ± 0.03     | 92.01                | 36.72 ± 1.4 |
| Cu + 2.5 % Al <sub>2</sub> O <sub>3</sub> | 8.00 ± 0.04     | 91.22                | 47.55 ± 1.8 |
| Cu + 5 % Al <sub>2</sub> O <sub>3</sub>   | 7.68 ± 0.03     | 88.78                | 50.76 ± 1.6 |
| Cu + 7.5 % Al <sub>2</sub> O <sub>3</sub> | 7.52 ± 0.02     | 88.21                | 54.29 ± 1.3 |
| Cu + 10 % Al <sub>2</sub> O <sub>3</sub>  | 7.48 ± 0.02     | 89.12                | 58.17 ± 1.4 |

The examination showed that the compaction process following the initial sintering increased the properties of Cu /Al<sub>2</sub>O<sub>3</sub> composites. Analyzing the results of tests of composites after pressing and sintering (**Table 1**) and subjected to the compacting process (**Table 2**), it can be concluded that the compacting process causes a slight increase in the density of composites and a significant increase in hardness compared to only presintered composites. The hardness of pre-sintered composites decreases with the increase of Al<sub>2</sub>O<sub>3</sub> content, which may be caused by the significant porosity of the composites. As a result of the compaction process, the pores inside the material are closed, which confirms the increase in density of composites after the compaction process in relation to pre-sintered composites. The highest density of material was obtained for a composite made from pure copper after the compaction process which amounted 8.19 g/cm<sup>3</sup>. Addition 2.5 % of alumina



caused decrease in density to 8 g/cm $^3$ . The highest hardness was obtained for the composite containing 10% Al $_2$ O $_3$  after the compaction process which amounted to 58 HB (70% higher than the pre-sintered composite). Similar results were received by Lee et al. [4].

# 3.3. Electrical conductivity tests

The results of electrical conductivity measurements are presented in **Table 3**.

**Table 3** The results of the electrical conductivity measurements

| Material                                  | Electrical conductivity (MS/m) |              |  |
|---|--------------------------------|--------------|--|
|   | Sintered                       | Compacted    |  |
| Cu  | 50.39 ± 1.63                   | 50.80 ± 0.34 |  |
| Cu + 2.5 % Al <sub>2</sub> O <sub>3</sub> | 45.05 ± 0.44                   | 47.26 ± 1.13 |  |
| Cu + 5 % Al <sub>2</sub> O <sub>3</sub>   | 34.90 ± 1.01                   | 39.73 ± 1.05 |  |
| Cu + 7.5 % Al <sub>2</sub> O <sub>3</sub> | 29.41 ± 1.04                   | 34.34 ± 0.54 |  |
| Cu + 10 % Al <sub>2</sub> O <sub>3</sub>  | 24.83 ± 1.25                   | 30.86 ± 0.40 |  |

The electrical conductivity of the tested composites decreases with increasing content of  $Al_2O_3$ . The same phenomenon was also noticed by other researchers [1, 2, 4, 9]. As a result of the compaction process, the value of the electrical conductivity of composites is increased in comparison to pre-sintered composites (**Table 3**). The increase in electrical conductivity of composites after compacting process is caused by higher density and lower porosity.

# 3.4. Wear characterization

The results of the abrasion resistance test are shown in **Figure 5**.

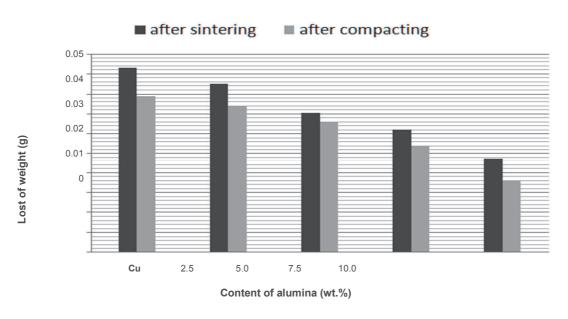


Figure 5 Weight loss of composites depending on the content of Al<sub>2</sub>O<sub>3</sub>

Tribological tests indicate that the compaction process of composites caused a slight increase in abrasion resistance (**Figure 5**). Al<sub>2</sub>O<sub>3</sub> particles in both sintered and compacted composites reduce abrasion-wear of the material. Increase in wear resistance with growing content of alumina has also been observed by Shi et al. [5].



## 4. CONCLUSION

The analysis of the microstructure of the composites showed that the  $Al_2O_3$  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 our previous own 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  $Al_2O_3$  particles with the copper matrix was obtained, without voids, only the pores occurring in sintered metals were visible on the micrographs. Aluminum oxide particles were clearly visible in the form of irregular precipitates. The composites fabricated by pressing, sintering and compacting were characterized by properties superior to those of the materials obtained using pressing and sintering. The compaction process caused an increase in density, hardness, electrical conductivity and abrasion-resistance of composites containing  $Al_2O_3$  particles.

## **REFERENCES**

- [1] MIKUŁA, J., ŁACH, M. and KOWALSKI, J.S. Copper matrix composites reinforced with volcanic tuff. *Metalurgija*. 2015. vol. 54, pp. 143-146.
- [2] ŁACH, M. Structure of metal matrix composites with an addition of tuff. *Archives of Foundry Engineering*. 2010. vol. 10, special issue 3, pp. 135-140.
- [3] BOROWIECKA-JAMROZEK, J. and DEPCZYŃSKI, W. The effect of the addition of zeolite on the properties of a sintered copper- matrix composite. In *METAL 2017: 26rd International Conference on Metallurgy and Materials*. Ostrava: TANGER. 2017. pp. 1652-1657.
- [4] LEE, D.W. and KIM, B.K. Nanostructured Cu-Al<sub>2</sub>O<sub>3</sub> composite produced by thermochemical process for electrode application. *Materials Letters*, 2004, vol. 58, pp. 378-383.
- [5] SHI, Z. and YAN, M. The preparation of Al<sub>2</sub>O<sub>3</sub>-Cu composite by internal oxidation. *Applied Surface Science*. 1998. vol. 134, pp. 103-106.
- [6] KACZMAR, J.W., PIETRZAK, K. and WŁOSIŃSKI, W. The production and application of metal matrix composite materials. *Journal of Materials Processing Technology*. 2000. vol. 106, pp. 58-67.
- [7] DASH, K., RAY, B.C. and CHAIRA, D. Synthesis and characterization of copper-alumina metal matrix composite by conventional and spark plasma sintering. *Journal of Alloys and Compounds*. 2012. vol. 516, pp. 78-84.
- [8] KRUGER, C. and MORTENSEN, A. In situ copper-alumina composites. *Materials Science and Engineering* A. 2013. vol. 585, pp. 396-407.
- [9] TAHA, M.A. and ZAWRAH, M.F. Effect of nano ZrO<sub>2</sub> on strengthening and electrical properties of Cu-matrix nanocomposites prepared by mechanical alloying. *Ceramics International*. 2017. vol. 43, pp. 12698-12704.
- [10] FATHY, A., ELKADY, O. and ABU-OQAIL, A. Synthesis and characterization of Cu- ZrO<sub>2</sub> nanocomposite produced by thermochemical process. *Journal of Alloys and Compounds*. 2017. vol. 719, pp. 411-419.
- [11] SHOJAEEPOUR, F., ABACHI, P., PURAZRANG, K. and MOGHANIAN, A.H. Production and properties of Cu/Cr<sub>2</sub>O<sub>3</sub> nano-composites. *Powder Technology.* 2012. vol. 222, pp. 80-84.
- [12] RAJKOVIC, V., BOZIC, D., DEVECERSKI, A. and JOVANOVIC, M.T. Characteristics of copper matrix simultaneously reinforced with nano- and micro-sized Al<sub>2</sub>O<sub>3</sub> particles. *Materials Characterization*. 2012. vol. 67, pp. 129-137.
- [13] KONIECZNY, M. The effect of sintering temperature, sintering time and reinforcement particle size on properties of Al<sub>2</sub>O<sub>3</sub> composites. *Composites Theory and Practice*. 2012. vol. 12, no. 1, pp. 39-43.