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COMPOSITES THEORY AND PRACTICE

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MANUFACTURING AND TECHNOLOGICAL PROPERTIES OF CU-AL2O3 COMPOSITE POWDERS MANUFACTURED BY MECHANICAL ALLOYING PROCESS

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The mechanical alloying process was applied to manufacture Cu based composite powders containing 2.0, 3.5, 5.0 and 10.0 vol.% alumina powders, The electrolytic copper powder and alumina powders were milled in an attritor for 2, 4, 6, 10, 16 and 24 hours. The distribution of the strengthening alumina powders in the copper matrix was investigated by means of light microscopy and evaluated on the basis of earlier elaborated statistical methods. The optimum time ensuring homogeneous distribution of the alumina particles was 24 hours with the attritor axis rotation rate of 120 1/s. The sieve analysis of the manufactured powders, pressability with the application of pressing pressures of 50, 100, 150, 200 and 250 MPa and sinterability at 900°C for 2 hours were investigated. The manufactured powders will be further applied for heat sinks, and contact materials which are characterized by a low wear rate.

Keywords: composite materials, mechanical alloying, copper base materials, composite properties

INTRODUCTION

The mechanical alloying technique was invented in 1974 by J.S. Benjamin and T. E. Volin [1], who produced composite Fe-Cr powders from atomized iron powder and chromium powder obtained by alumino-thermic reduction and grinding. As a result, they manufactured composite iron based powders strengthened with the hard chromium particles. Generally, the process of mechanical alloying makes it possible to manufacture composite powders composed of a relatively soft matrix, mainly powders of metals and metal alloys, combined with hard, fine ceramic powders like metal oxides, metal nitrides or metal carbides. Moreover, this technology can be applied for the production of metastable materials characterized by amorphous or nanocrystalline structures and can be applied to manufacture solid solutions characterized by an extended solubility of components [2].

The technology of mechanical alloying was employed by Chmielewski et al. [3] to manufacture Cu-20 vol.% AlN composite powders, which, after sintering were applied as materials for heat sinks. The powders were manufactured by applying the classical mechanical alloying technique, and the effect of the milling rate, milling time and milling balls to powder ratio on the properties of powders were investigated.

Strojny-Nędza et al. [4] manufactured composite Cu-5 vol.% Al_2O_3 powders by means of the mechanical alloying technique. The powders were further consolidated by the spark plasma sintering technique (SPS). The authors studied the physical properties of the manufactured composite copper based materials containing 5 vol.% alumina particles (density, hardness HV1, thermal conductivity) and performed detailed investigations of the material structure utilizing scanning electron microscopy (SEM), transmission electron microscopy (TEM) and examined the linear distribution of elements – Cu, Al, O – in the sintered materials.

Franczak and Karwan-Baczewska [5], manufactured Cu based composite powders containing 5, 10 and 15 wt% titanium nitride. They researched the following technological properties of the manufactured composite powders: the bulk density, tap density, and flowability, which are influenced by the shape of the particles, their size and surface roughness. The powders containing 5 vol.% TiN were consolidated by means of the spark plasma sintering technique (SPS) [6]. The authors investigated the structure of the composite materials manufactured by the SPS technique and measured their density, hardness as well as electrical conductivity.

Shimada et al. [7] manufactured Cu-Y_2O_3 composite powders by applying the mechanical alloying technique. The composite materials were further processed by hot isostatic pressing (HIP) and the authors investigated the movement of the yttria particles in the copper matrix due to the processes of recrystallization and grain growth during HIP. Rabiee et al. [8] manufactured Cu-Fe composite powders containing silicon carbide nano-powders by the mechanical alloying method. They further processed the composite materials by cold pressing and sintering. They ascertained that the precipitates of iron during sintering were responsible for the relatively small grain growth of the copper matrix. The addition of silicon carbide increased the hardness, which was connected with a strengthening effect in the copper matrix.

Yousuf et al. produced in-situ copper based composite powders strengthened with tungsten carbide by mechanical alloying [9]. As the starting materials, elemental Cu, W and C powders were used. After the sintering process, the presence of two tungsten carbide compounds, namely WC and W_2C were found. WC was formed as the coexisting compound of the reduction of W_2C and unreacted W. The microstructure of the materials and the phase changes were characterized by means of X-ray diffraction, scanning electron microscopy and X-ray photoelectron spectroscopy. Murmu et al. manufactured composite Cu based powders strengthened with TiB_2 [10] from elemental powders of Cu, Ti and B, utilizing the mechanical alloying technique. The in-situ formation of TiB_2 was investigated by means of differential scanning calorimetry (DSC) and X-ray diffraction (XRD). The effect of the various manufacturing parameters on the manufacturing process of the composite pow ders and the in-situ formation mechanism of TiB_2 on the thermo-mechanical route was discussed. From the Cu based composite powder, further bulk composite materials were manufactured and were characterized by the following mechanical and physical properties: ultimate tensile strength (UTS) of 375 MPa, yield strength of 300 MPa, hardness of 150 HV and electrical conductivity of 53% IACS.

Ahmadian et al. [11] manufactured copper based composite materials strengthened with silicon carbide (SiC) nanoparticles and MWCNTs (multi-walled carbon nanotubes) [11]. The SiC nanoparticles were characterized by an average particle size of 30 nm and the MWCNTs were of a diameter of approximately 8-15 nm and length of 3-12 μm. After the mechanical alloying process, consolidation of the obtained composite powders was performed by the SPS process. They ascertained that the hardness of the composite material containing 5 wt% SiC and 1 wt% MWCNTs was 65 HV, although the wear rate under the load of 5 N increased comparing to the unreinforced copper.

Rodrigues et al. [12] produced copper based composite materials from copper, graphite, and alumina powders by carrying out the mechanical alloying process for up to 16 hours. The authors characterized the microstructure of the manufactured powders by means of field emission gun scattering electron microscopy/energy-dispersive spectroscopy and X-ray diffraction. The produced Cu based composite powders were nanostructured with graphite and alumina nanoparticles, which were homogeneously distributed in the copper matrix. It was ascertained that the bonding at the interface of copper and the strengthening elements was very good, which makes it possible to transfer large loads from the Cu matrix to the reinforcement [12]. Sadoun et al. [13] manufactured copper based composite powders strengthened with 2.5, 5.0, 7.5 and 10.0 vol.% alumina particles coated with silver. The powders were further consolidated by the PM method. The microhardness measurements of the composite materials containing 10 vol.% of alumina in the copper matrix exhibited the highest microhardness of 175 HV, whereas a microhardness of 70 HV was obtained for the pure copper matrix. The abrasive wear rate decreased with the increased alumina content in the matrix and the coating of the alumina particles with silver caused a decrease in the coefficient of friction.

Some authors used the mechanical alloying method to manufacture Cu-Cr alloys or Ti-Cu alloys without strengthening particles. Zhao et al. [14]

manufactured by mechanical alloying Cu-Cr powders in a specially designed attritor at the temperature of 325°C for 3 hours applying a ball-to-powder mass ratio of 15:1. As the element reducing metallic oxides, active carbon powder was applied, while the graphite powder did not have such good reducing properties. A detailed microscopic analysis of the manufactured powders was performed and crystal agglomeration phenomenon was ascertained in the alloy powder, which resulted in an increase in the particle size of the Cu-Cr powders. Zhao et al. [15] applied Box-Behnken design (BBD) to manufacture Cu-Cr alloy powder. They investigated the effect of different production parameters like the milling temperature, milling time, in addition to the ball-to-powder mass ratio on the structure and grain size of the Cu-Cr alloy powder. Arkusz et al. [16] produced binary Ti-Cu alloys containing 1.6 and 3 wt% copper by means of the mechanical alloying technique and conventional powder metallurgy. They especially investigated the effect of the copper content in the titanium matrix on the microstructure and properties of the manufactured alloys. The corrosion tests revealed better corrosion resistance comparing to pure Ti, and the manufactured materials are foreseen in biosensing applications [16].

Qin et al. manufactured composite copper based materials strengthened with 3 wt% Y_2O_3, 1 wt% $Y_4Zr_3O_{12}$, 3 wt% $Y_4Zr_3O_{12}$ and 5 wt% $Y_4Zr_3O_{12}$ [17]. The manufacturing process was based on a combined solid-liquid doping process, calcination, mechanical alloying and spark plasma sintering of the composite copper based particles. The composite copper based materials containing 5 wt% $Y_4Zr_3O_{12}$ exhibited strength of 317.5 MPa and the electrical conductivity for this composition was of 77% IACS. Qin et al. [18] manufactured copper based composite materials containing 1, 3 and 5 wt% yttrium oxide (Y_2O_3) based on the process of solid-liquid doping, calcination reduction and spark plasma sintering. The strengthening particles of yttrium oxide after the applied processes were characterized by a diameter of 150 nm. The composite materials containing 3 wt% yttrium oxide (Y_2O_3) showed the best mechanical properties: tensile strength of 290.1 MPa, hardness of 125.7HV and electrical conductivity at the level of 95% IACS.

Su et al. manufactured composite copper/graphite composite materials strengthened with Ti_3AlC_2 hard particles [19]. The composite materials were manufactured by the mechanical alloying process of Ti_3AlC_2 hard particles (5 wt%) with Cu-plated graphite powder (10 wt%) and pure copper powder. The composite powders were consolidated using a vacuum hot press at the pressure of 50 MPa, temperature range of 750-950°C and pressing time of 10 min. A relative density of 98.4% was obtained and a hardness of 75.7 HV was achieved. The manufactured material was characterized by good wear resistance and the lowest wear rate was 1.942×10^{-5} mm^3 N^{-1} m^{-1} for the material sintered at the temperature of 900°C. Meng et al. applied the mechanical alloying technique to manufacture W-Cu coatings on Cu substrates [20]. The studies showed that the manufactured coatings on the Cu substrate exhibited very good bonding at the interface and were characterized by a hardness of 228 HV0.1.

Molina et al. [21] manufactured copper based composite materials containing strengthening particles of intermetallic compounds – CuFeAl, $CuFe_3Al$ and Cu(CuNiAl). As the matrix powder, electrolytic copper powder was applied. Composite powders containing up to 5 wt% strengthening elements were manufactured utilizing the mechanical alloying technique. The composite powders were compacted at the temperature of 350°C under the pressure of 180 MPa. Then the samples were sintered and further subjected to hot extrusion applying the reduction ratio of 2:1. The manufactured composite materials were characterized by relatively good mechanical properties and the additions resulted in a lower corrosion rate investigated in an NaCl solution.

The aim of the presented investigations on manufacturing Cu-Al_2O_3 composite powders is to elaborate the starting composite powder characterized by homogeneous distribution of the strengthening alumina particles in the copper matrix and their minimum damage during the mechanical alloying process. Such powders will be applied for further manufacturing of Cu based composite materials. The composite materials will be manufactured by pressing, sintering as well as hot extrusion and should be characterized by relatively good electrical and heat conductivity, in addition to good resistance to wear. The manufactured Cu-Al_2O_3 composite materials will be applied in sliding contacts and heat sinks.

PROCEDURE AND MATERIALS

Microscopic investigations were performed by means of a scanning electron microscope, HITACHI TM-3000, and the analysis was made utilizing a BSE detector with an accelerating voltage of 15kV.

Sieve analysis was performed using a Multiserw Morek LPzE-2e sieve shaker. Loose powders were placed on the SEM stage, and in addition, metallographic specimens of the composite powders were embedded in the resin, grinded with abrasive papers and finally polished. All the samples (loose powders and metallographic specimens) were observed after vacuum sublimation with graphite utilizing a special system with a Quorum Q150T turbomolecular pump.

As the matrix powder, electrolytic copper powder delivered by EUROMET Skawina was used. The powder was characterized by an irregular shape with characteristic dendrites, as seen in Fig. 1a. The particles were generally smaller than 160 µm and only small fractions of about 3.4% were larger than 160 µm. The sieve analysis of this powder is shown in Fig. 1b.

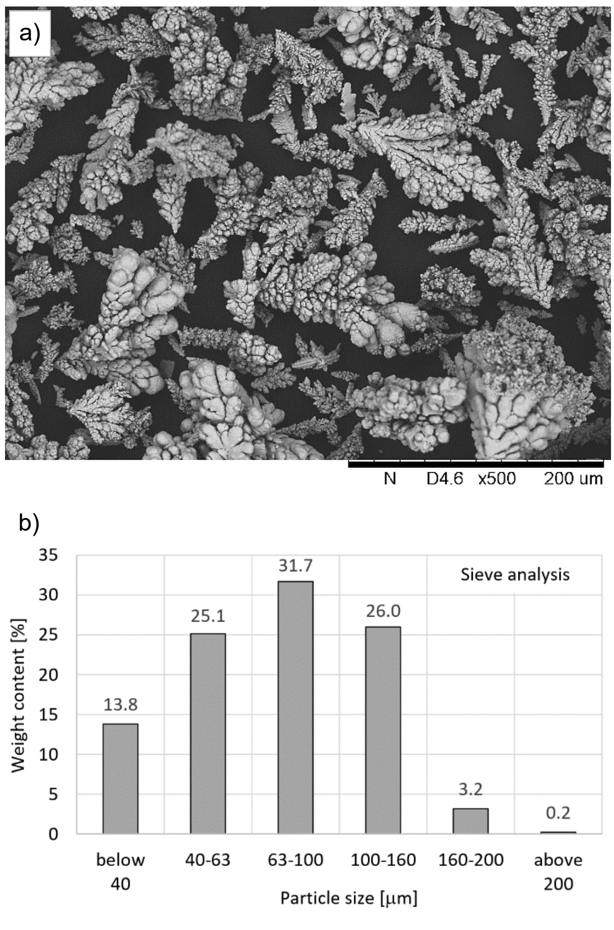


Fig. 1. Electrolytic copper powder produced by EUROMET, Skawina Poland: a) SEM micrograph of as-fabricated Cu powder, b) sieve analysis

The composite Cu based powders containing 2.0, 3.5, 5.0 and 10 vol.% alumina powders were manufactured by means of a classic attritor with a vertical axis and horizontal arms. The rotation rate of the axis was 120 1/s. The view of this attritor designed at the Chair of Lightweight Elements Engineering, Foundry and Automation of Wrocław University of Science- and Technology is shown in Fig. 2. The inner side of the attritor was made from 11G12 Hadfield steel, which has very low wear rate. The mechanical alloying process was carried out by utilizing corundum balls 12 mm in diameter and the volume relation of the ceramic balls to the milled powder was 10:1.

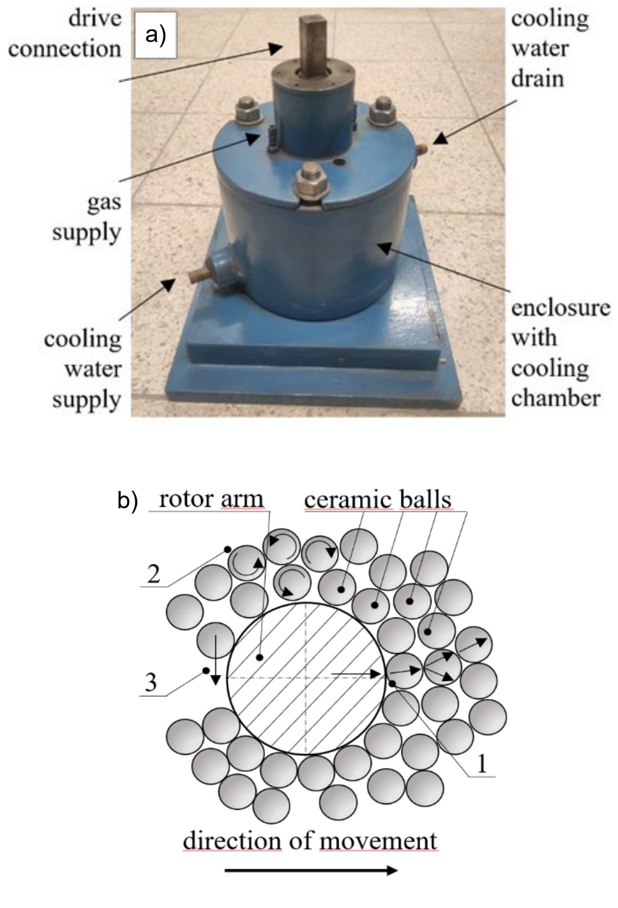


Fig. 2. Attritor designed at Chair of Lightweight Elements Engineering, Foundry and Automation of Wrocław University of Science and Technology: a) general view of attritor, b) interactions of attritor milling elements on matrix and dispersion powder blends: 1-impact, 2- rotational, 3-tumbling

The mechanical alloying process is based on the sequential phenomena of plastic working and welding of matrix materials containing ceramic powders and is schematically shown in Fig. 3 a-d. The starting electrolytic copper powder is schematically illustrated in Fig. 3a and according to the fact that the small ceramic strengthening particles of alumina can be placed between the copper dendrites during preliminary mixing, this kind of electrolytic copper powder is suitable for the MA process.

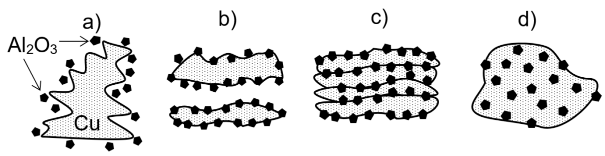


Fig. 3. Stages of mechanical alloying process of copper particles and alumina particles: a) initial stage where alumina particles are distributed around copper particles, b) intermediate stage where alumina particles adhere to plastically deformed copper particle, c) welding of separate copper particles, d) production of equiaxial composite Cu-alumina particles

The pressability of the composite powders was investigated by means of the floating die method utilizing a die characterized by the diameter of 25 mm. The lubricant was not added to the powder and the following pressing pressures were applied: 50, 100, 150, 200 and 250 MPa.

RESULTS AND DISCUSSION

The mechanical alloying process was conducted by applying the relatively low rotation rate of 120 1/s and MA times of 2, 4, 6, 10, 16 and 24 hours (the chosen samples for powder shape control were milled for 22 hours). The distribution homogeneity of the alumina strengthening powder in the copper matrix was examined by means of a light microscopy and statistical methods described in [22]. No fragmentation of the alumina strengthening particles during the MA process was found, which was mainly caused on the one hand by the by the relatively soft copper matrix and on the other hand by the relatively average shear stresses in the matrix during the process. The particle shapes were examined by SEM. An irregular particle shape was observed after the MA process (Fig. 4a). The sieve analysis for the Cu-5 vol.% Al_2O_3 powder after 24 hours of MA is shown in Fig. 4b. Generally, the copper composite particles were smaller than 100 μm, and for instance, in the powder containing 5 vol.% alumina, 93.6% of the powder particles were smaller than 100 μm. The sieve analysis revealed that in the case of Cu-2 vol.% alumina, 97.7% of the particles were smaller than 100μm and only 0.7% were larger than 315 μm. The apparent density of the manufactured composite powders was between 20-24% of the relative density for all the powders.

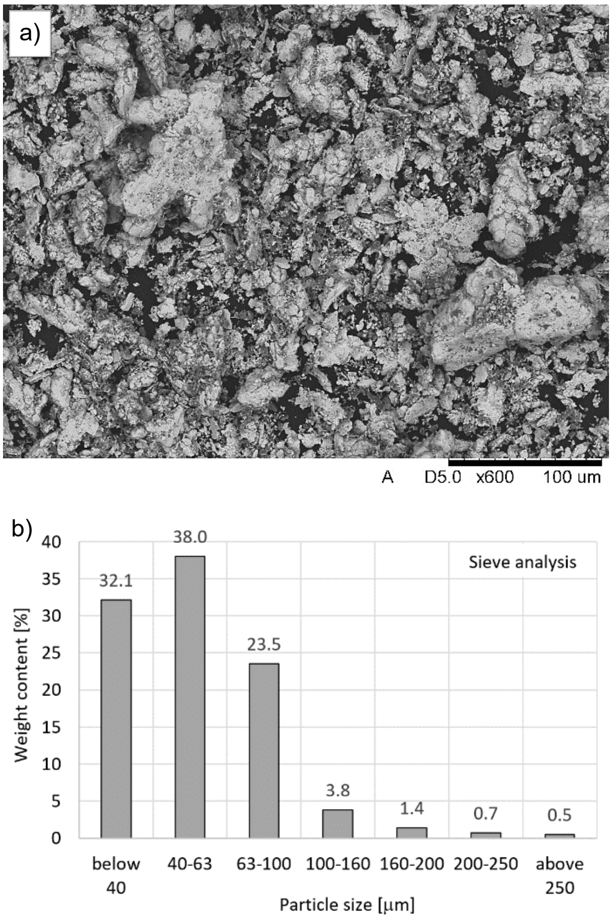


Fig. 4. Composite Cu-Al2O3 powders: a) powder containing 3.5 vol.% alumina particles after 4 hours of MA process (SEM), b) particle size analysis of composite Cu-5 vol.% Al2O3 powder after 24 hours of MA process

SEM analysis of the structures of the particles revealed that after short times of MA (Fig. 5a) the alumina particles were mainly distributed at the outer layers of the copper particles. The homogeneous distribution of the strengthening particles in the matrix was achieved after 24 hours of conducting the MA process (Fig. 5b).

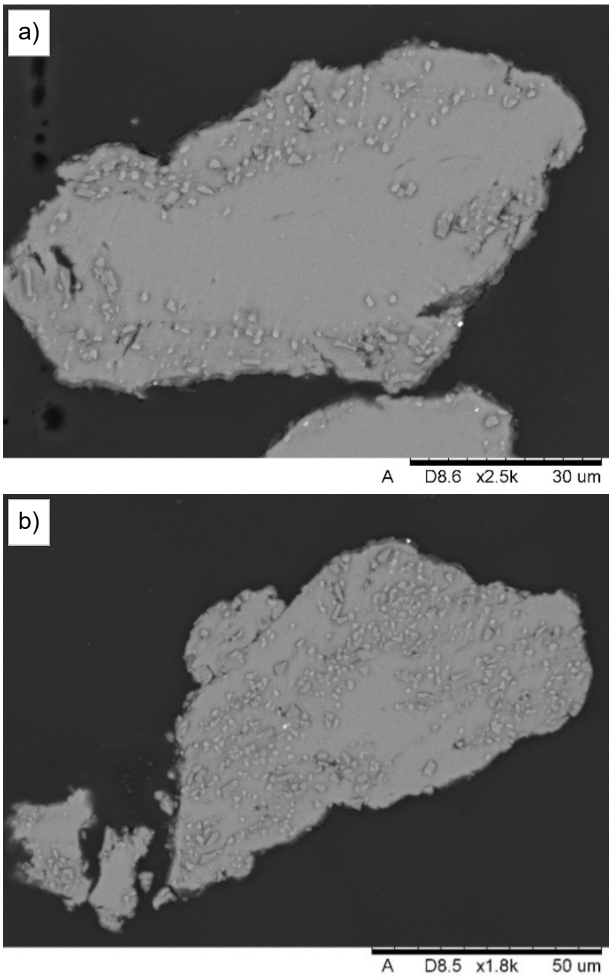
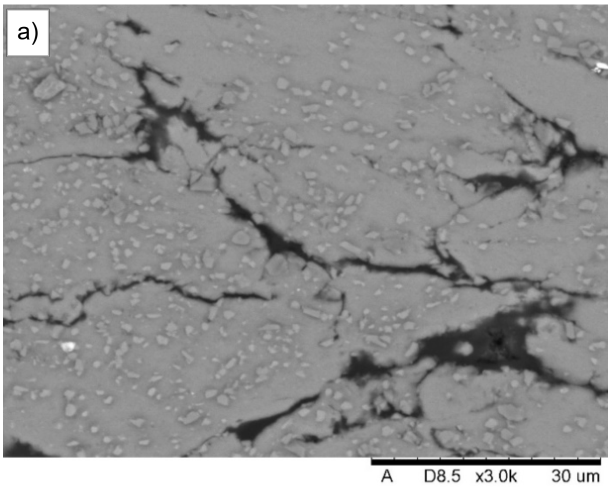


Fig. 5. SEM microstructure of cross-sectioned Cu-Al2O3 composite particles after MA process: a) 3.5 vol.% of Al2O3, time 2 h, b) 5 vol.% of Al2O3, time 24 h

The composite powders formed agglomerates, which is shown in Fig. 6a and 6b.



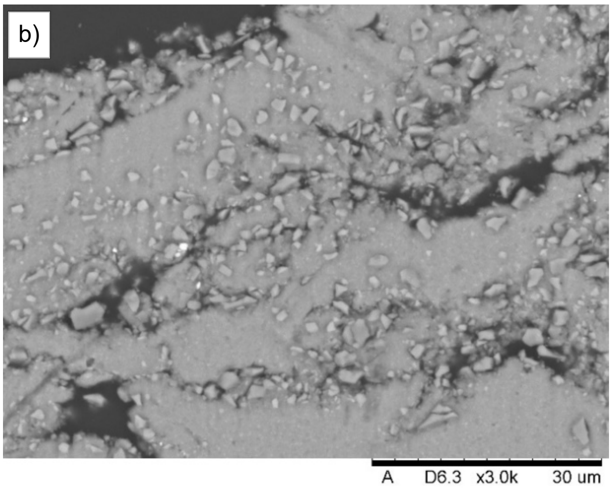


Fig. 6. SEM microstructure of cross-sectioned Cu-Al2O3 agglomerates of composite particles after MA process: a) 5 vol.% Al2O3, time 16 h, b) 10 vol.% Al2O3, time 10 h

The mechanical alloying process makes it possible to manufacture composite Cu-Al_2O_3 powders containing 2.0, 3.5, 5.0 and 10.0 vol.% alumina particles, although during the future investigations the rotational speed of the rotor should be increased in order to shorten the MA time.

The pressability of the Cu-Al_2O_3 composite powders was dependent upon the alumina strengthening powder content in the copper matrix and it is presented in Fig. 7.

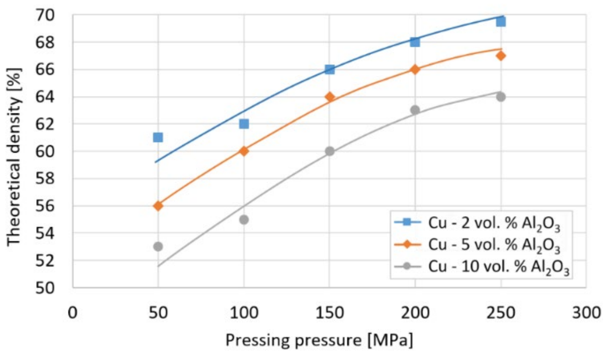


Fig. 7. Pressability of Cu-alumina composite powders containing 2, 5 and 10 vol.% alumina particles

The application of a pressing pressure of 250 MPa allowed a theoretical density of 69.5% to be attained in the case of the composite powder containing the smallest amount (2 vol.%) of strengthening particles. On the other hand, the density of the powder containing 10 vol.% strengthening powders was much smaller and at the pressing pressure of 250 MPa the compacts made of composite powder containing 10 vol.% alumina reached only a theoretical density of 64%.

The highest relative density was achieved for the samples made of pure copper powder and by applying the pressure of 250 MPa, it was 75%. The hard alumina particles decreased the relative density during pressing, according to the friction forces acting on the outer surface of the copper particles, and on the other hand, the hard strengthening alumina particles located in the copper matrix considerably limit the plastic deformation of the copper particles due to strengthening of the copper matrix.

Sinterability tests were performed on the samples pressed with the pressure of 200 MPa and sintered at the temperature of 900°C during 2 hours in the protective gas atmosphere of dry hydrogen. After sintering the density was measured, then the theoretical density was calculated and the results are shown in Fig. 8.

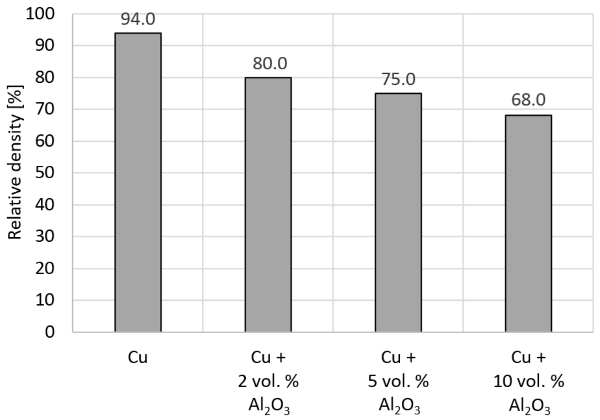


Fig. 8. Relative densities of compacts made from pure electrolytic copper powder and from powder containing 2 vol.% alumina pressed with pressure of 200 MPa and sintered in hydrogen at temperature of 900°C for 2 hours.

The largest theoretical density of 94% after conducting sintering (temperature of 900°C for 2 hours) is characterized by the copper compacts pressed with the pressure of 200 MPa. An increase in the volume content of strengthening alumina particles in the copper matrix lower the relative density, which is caused by the alumina particles that were located inside and at the surface the copper particles. The diffusional transport of matter in the case of sintering of copper takes place mainly by grain boundary diffusional transport of matter having an effect on shrinkage and neck growth. On the other hand, the surface and volume diffusional transport of matter only have an impact on neck growth [23]. The grain boundary and volume/surface diffusional transport of matter is hindered by the alumina particles, which results in the lower density of the samples containing the larger amounts of strengthening particles. At the critical contents of alumina particles in the matrix, they can stop the diffusional transport of matter completely during sintering.

Authors working on MA of Cu based composite powders generally applied particles of hard and stable materials like aluminum oxide (Al_2O_3) [4],[13], yttria (Y_2O_3) [7], yttria (Y_2O_3) and compounds of $Y_4Zr_3O_{12}$, [17], aluminum nitride (AlN) [3], titanium nitride (TiN) [5],[6], silicon carbide (SiC) [8], tungsten carbide (WC) [9], silicon carbide nanoparticles (SiC) together with MWCNTs [11], titanium diboride (TiB_2) [10], graphite and alumina nanoparticles [12]. Moreover the copper particles were strengthened with compounds like titanium aluminum carbide (Ti_3AlC_2) [19], and intermetallic compounds – CuFeAl, $CuFe_3Al$ and Cu(CuNiAl) [21]. The application of alumina strengthening powder in present investigations fulfills the condition of the application of hard and stable ceramic particles to strengthen the copper matrix. Moreover, the wettability of alumina with copper is very good, which results in good bonding at the interface alumina/copper.

On the basis of the conventional sintering of Cu composite powders, due to hinderance of the diffusional transport of matter by the hard strengthening particles, it is not possible to manufacture composite materials characterized by very high relative densities. The following techniques were applied in order to obtain Cu based composite materials characterized by a minimum theoretical density of 95%: spark plasma sintering (SPS) [4],[6],[11],[18], hot isostatic pressing (HIP) [7], vacuum hot pressing [19] and hot extrusion [21]. Some authors applied the conventional PM technique consisting of pressing and sintering and they inform on the attainment of high theoretical densities [13],[16]. Further investigations of the present authors will be focused on the manufacturing of Cu based composite materials by conventional pressing, sintering and hot extrusion.

The performed investigations demonstrated the possibility of manufacturing of Cu-Al_2O_3 composite powders characterized by a homogeneous distribution of strengthening alumina particles in the copper matrix and no damage of the alumina particles was observed during the MA process. The irregular shape of the Cu based composite powders was suitable for the pressing operations. The MA processing of copper and alumina particles was possible even for the relatively high contents of alumina in the copper matrix reaching 10 vol.%.

CONCLUSIONS

1. The mechanical alloying process of composite Cu based powders containing 2.0, 3.5, 5.0 and 10 vol.% alumina powders in the matrix makes it possible to manufacture composite powders characterized a homogeneous distribution of alumina particles in the matrix and an irregular shape, suitable for the pressing process.
2. The majority of the composite particles (generally over 93%) are smaller than 100µm and the long times of mechanical alloying process resulted in the production of equiaxied Cu-alumina composite particles.
3. On the basis of the microscopic observations, it was found that the mechanical alloying process causes no damage to the strengthening alumina particles.
4. The pressability of the composite powders containing alumina is much smaller in comparison with the non-strengthened copper powders and the increasing content of strengthening alumina particles in the copper matrix results in a smaller density of the compacts.
5. The sinterability of the compacts made of the composite particles was evaluated on the basis of the obtained relative density (for Cu-2 vol.% Al_2O_3 the relative density after sintering was 80%, for Cu-5 vol.% Al_2O_3 it was 75% and for Cu-10 vol.% Al_2O_3 it was 68%). Such low densities comparing to unreinforced sintered copper compacts (94%) are caused by the hinderance of the diffusional transport of matter by the alumina particles during the sintering process. In order to achieve the large densities of the sintered materials exceeding 98%, which will be suitable for the practical applications, the process SPS or hot plastic working, like hot extrusion, should be applied.

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