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The effect of ceramic type reinforcement on structure and properties of Cu-Al_2O_3 composites

A. STROJNY-NĘDZA[1*], K. PIETRZAK[1], A. GŁADKI[1], S. NOSEWICZ[2], D.M. JARZĄBEK[2], and M. CHMIELEWSKI[1]

[1] Institute of Electronic Materials Technology, 133 Wolczyńska St., 01-919 Warsaw, Poland
[2] Institute of Fundamental Technological Research Polish Academy of Sciences, 5B Pawińskiego St., 02-106 Warsaw, Poland

Abstract. The purpose of this paper is to elaborate on mechanical alloying conditions for a composite powder consisting of copper and brittle aluminium oxides. Detailed analysis of the Cu-Al_2O_3 powder mixture structure obtained in the mechanical alloying process allows for the study of the homogenization phenomena and for obtaining grains (in composite form) with a high degree of uniformity. The Cu-$5_{\mathrm{vol.}}\%Al_2O_3$ composites were obtained by means of the spark plasma sintering technique. The results presented herein were studied and discussed in terms of the impact of using a different form of aluminium oxide powder and a different shape of copper powder on composite properties. Research methodology included microstructure analysis as well as its relation to the strength of Cu-Al_2O_3 interfaces. It transpires from the results presented below that the application of electrocorundum as a reinforcement phase in composites decreases porosity in the ceramic phase, thus improving thermal properties and interfacial strength.

Key words: metal matrix composites, spark plasma sintering, thermal conductivity, interfacial strength.

1. Introduction

Among others, copper-based composites doped with a ceramic phase show high resistivity to sudden temperature changes, high thermal conduction as well as increased mechanical strength. Because of these superior features, Cu-Al_2O_3 composites have found their use in resistance welding electrodes, accelerators and electrical connectors [1, 2]. Another potential application of Cu-Al_2O_3 composites includes thrusters in turbines, typically employed in the aerospace industry. Parameters such as thermal conduction > 300 W/(mK), increased erosion, oxidation resistance and long lifetime are required in the case of these systems. Up until now, these elements have been produced using Cu-Ag-Zr alloys [3].

Mechanical properties and performance of metal-ceramic composite materials depend predominantly on the mechanical characteristics of the matrix and properties of the reinforcing phase, its shape, and, most of all, the quality of the metal-ceramic interface. A solid coupling enables significant stress transfer from the matrix to the reinforcement, which results in excellent mechanical, tribological and thermal properties. Usually, the matrix has lower mechanical strength but also higher plasticity than the reinforcement material, and when applying a load onto a composite material, stress is transferred from the weaker matrix to the stronger reinforcement. When imposing load onto a composite with poor adhesion, the brittle reinforcing phase is detached from the matrix at the interfacial boundary. Either good or very good adhesion at interfacial boundaries leads to excellent mechanical properties and performance observed for composite materials [4, 5]. Additionally, in the case of these particular materials, it is immensely important for the reinforcing phase to be distributed evenly in the matrix. Agglomerates of ceramic particles in the metallic matrix are likely to lead to matrix inhomogeneity observed in the material, thus resulting in composite properties deterioration. For homogeneous distribution of ceramic particles, it is recommended to use relatively small matrix particles, which may prove costly. Moreover, when mixing matrix particles and reinforcement with a significant density difference, the problem of gravity segregation appears, leading to uneven distribution of ceramic particles in the metallic matrix. Highly homogeneous distribution of reinforcing particles in the matrix can be achieved using mechanical alloying, originally introduced by J. Benjamin [6]. When mixing Cu ductile powder with fragile Al_2O_3, following the mechanical alloying method, one can observe homogeneous distribution of ceramic particles in the plastic matrix. This results from the fact that ceramic powder becomes cracked and highly comminuted during the grinding process, while metal powder is deformed and subsequently fractured. As a result, fragments of brittle powders reach the annealed surfaces of ductile powders and get trapped between them. Consequently, fine, fragile particles end up evenly distributed in the plastic matrix. The mechanical production of composite powders and alloys most often takes place in an attritor, transmitting significant mechanical energy through steel balls and a rotor to the powder mixture. The steel balls can exert percussive, rotary or drumming influence on the powder mixture.

When producing copper-based composites doped with ceramic particles, one comes across a wide variety of physical

*e-mail: agata.strojny@itme.edu.pl

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and chemical constraints, which have to be overcome to obtain a material with negligible porosity and high thermal conductivity. One of the major problems to be faced is the limited temperature range suitable for the production of composites, resulting from the melting temperature of copper (1083°C), which is low when compared with the sintering temperature of ceramics. Consequently, a significant number of pores appear in the material [7, 8]. It is important to obtain a strong coupling at the metal-ceramic interface without the appearance of the third phase, i.e. spinels and oxides that seriously contribute to deterioration of the composites' thermal properties. The coupling should be of adhesive rather than diffusive or reactive nature. This particular coupling type is the weakest among the group mentioned, and therefore other structural defects – expected to considerably influence the composites and their characteristics – should be avoided.

As already mentioned above, the interface strength, which depends on the adhesive force between ceramic particles and the metal matrix, is known as a critical parameter, determining the mechanical properties of composites. Therefore, its experimental examination is tremendously important. The present work features the author's own solution for measuring the adhesive force per unit area between the ceramics particle and the metal matrix in composites. Basically, it consists in stretching a wire based on the ceramics-metal coupling by applying force, using a specially designed device [9, 10].

Among other things, the power analysis of phenomena accompanying the formation processes of the Cu-Al_2O_3 composite structures through mechanical synthesis in a high-power attritor mill is presented. The idea behind isotopic production of composite materials employing the above-mentioned method requires understanding of the milling energy dissipation phenomenon. In fact, the key issue here is a conscious choice of the milling process parameters, so that the main energy flux is directed towards the production of the isotropic structure of a composite powder. Additionally, the influence of the form and shape of grains in both copper and aluminium oxide starting powders on formability of the composite powder and on the properties of the composite materials produced based on those was examined.

The outcome composites underwent microstructural examination (i.e. SEM and TEM), the impact of starting powder shape on thermal characteristics and hardness was studied, and strength measurements for the metal-ceramic interface were performed.

2. Experimental procedure

The following commercially available powders were used as the starting materials: copper powder with dendritic (dend.) and spherical (spher.) grains and purity of 99.9% by Sigma Aldrich (Fig. 1) as well as two types of alumina powder, i.e. electrocorundum powder (ED Al_2O_3) with the purity of 99% by KOS (Fig. 2a) and the α-form of aluminium oxide powder – (αAl_2O_3) with 99.99% purity by NewMet (Fig. 2b). The particle size distribution was analysed as a function of the Feret's diameter (d). As a result, the average Feret's diameter

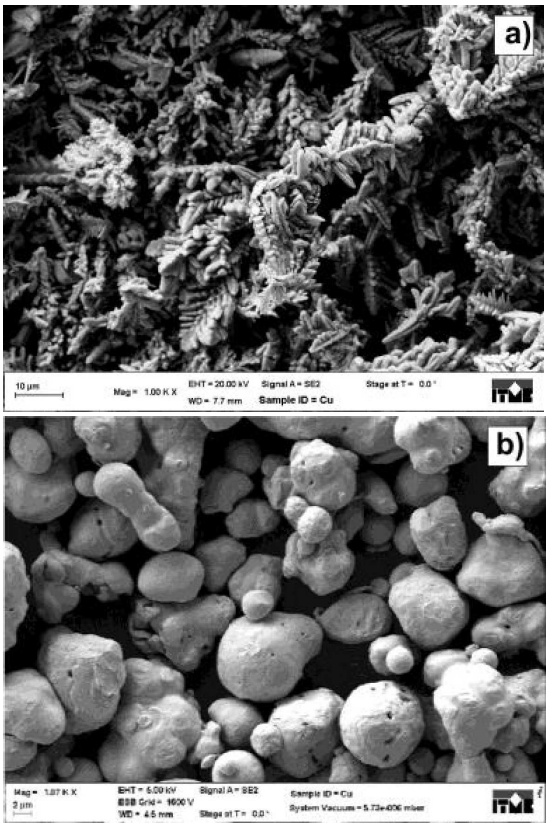

Fig. 1. Morphology of starting copper powders with different shape of grains: a) dendritic, b) spherical

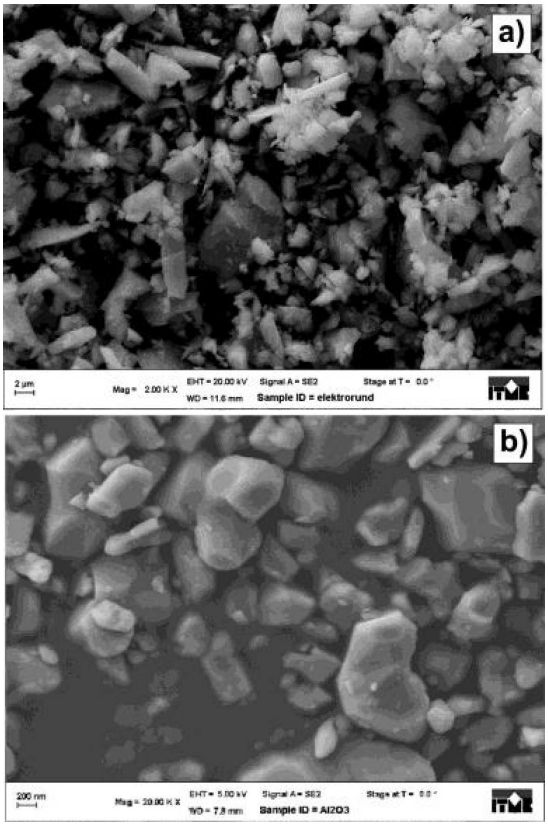

Fig. 2. Morphology of starting alumina powders: a) electrocorundum (ED Al_2O_3), b) α Al_2O_3

(d_A) was calculated. Based on the analysis, mean particle size was found to be 9 μm (between 2 and 11 μm) for Cu_{spher}, 10 μm (between 4 and 12 μm) for Cu_{dend}, 2.4 μm (between 1 and 5 μm) for αAl_2O_3 and 2.5 μm (between 0.9 and 4.5 μm) for ED Al_2O_3.

The Cu-$5_{vol.}\%Al_2O_3$ composites were obtained using the mechanical alloying method. The milling process was carried out in a stainless steel attritor (100 mm in diameter and 3000 cm^3 in volume) containing hard steel balls 10 mm in diameter. All milling runs were performed using the following operation parameters: ball-to-powder ratio (BPR) = 3:1 and rotation speed of 100 and 200 rpm. Stearic acid was also used as the process controlling agent to prevent agglomeration of the powder mixture during milling. The Cu-$5_{vol.}\%Al_2O_3$ powders being mixed were milled under nitrogen atmosphere for 10 h. Kinetic studies of the mixing process were sampled every two hours.

Spark plasma sintering (SPS) densification of the obtained powder mixtures was performed in a vacuum chamber (5.0×10^{-5} mbar) using the sintering temperature of 950°C, heating rate of 100°C/min., holding time of 10 min., and pressure of 50 MPa. Both processes were carried out in graphite matrices. As a result, cylindrical samples with the following parameters were obtained: diameter = Ø10.0 mm, height = 3 mm.

The relative density of the sintered samples was measured by the Archimedes method, in accordance with ASTM:B962-08. Theoretical density of the obtained composites was calculated following a simple rule of mixtures, assuming that the theoretical density of copper and alumina is 8.97 and 3.97 g/cm^3, respectively. The microstructure of both powder mixtures and sintered samples was examined with the Auriga CrossBeam Zeiss Workstation scanning electron microscope (SEM) with an integrated EDS microanalysis system.

Further investigation of the interface of Cu-Al_2O_3 composites was conducted using the JEOL 1200 transmission electron microscope (TEM), operating at an accelerating voltage of 120 kV. For this purpose, thin lamellas were extracted from interface regions using the Hitachi NB-5000 system, which combines a scanning electron microscope and a gallium focused ion beam mill (FIB).

Hardness (HV1) was tested by means of DuraScan 10/Emcotests with a Vickers diamond indenter, using a load of 9.81 N applied for 10 s. The hardness results were averaged over 5 indentations per specimen. Each load applied matched the measurement surface.

Measurement of the adhesion force between reinforcement and the matrix was performed following the method described in detail in [9, 10]. Basically, a wire with a particle (or particles) in the middle and copper at both ends was stretched on a custom-made microtensile tester (Fig. 3). The wires were precisely cut from the sample and then electro-etched in a solution of phosphoric acid in order to expose the interface being investigated. Due to the fact that alumina does not conduct electricity, the wires were etched for a few seconds and next their resistance was measured. The process was continued until the wires stopped conducting electricity. Subsequently, they were carefully investigated with an optical and scanning electron microscope. When the interface was exposed, they were put on a custom-made microtensile tester and stretched. It is known that properly prepared wires break at the interface, as a result of which interface strength can be determined. Interface strength is defined as the force needed to break the wire at the interface divided by the interface area:

$$\sigma = \frac{F}{A}, \tag{1}$$

where F is the adhesion force between the metal matrix and a ceramic particle and A stands for the contact area.

The contact area was determined from the images of both ends of the broken wires made with either an optical or a scanning electron microscope.

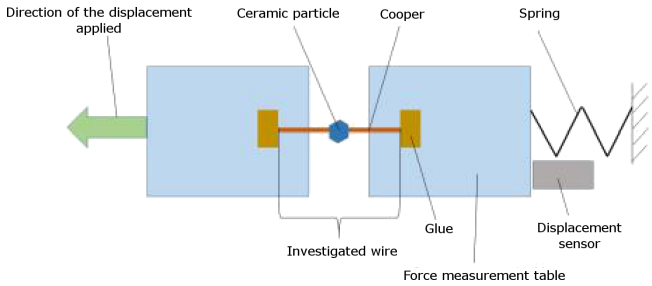

Fig. 3. Scheme of the tensile test of microwires with ceramic particles

Destruction of materials by thermal shock is determined by the thermal stress generated during severe thermal quenching. In general, a material with higher thermal conductivity has higher resistance to thermal shock because of its tendency to reduce both interior temperature gradients and thermal stress. For the purpose of the present investigation into copper-alumina composites, thermal diffusivity was measured at the temperature range of 50–600°C using the LF457/Netzsch Laser Flash Analyser in argon atmosphere. The front face of the sample being measured was homogeneously heated with an unfocused laser pulse. On the rear face of the sample, the temperature increase was measured as a function of time. Thermal conductivity was calculated in similar way as in [11]:

$$\lambda = Cp \cdot \rho \cdot a, \tag{2}$$

where λ – thermal conductivity, Cp – specific heat, ρ – density, a – thermal diffusivity. The specific heat value was determined based on the rule of mixtures.

In fact, theoretical value for the composite being studied can be calculated using the following Eq. (3) [10]:

$$\lambda c = \lambda m \frac{1 + 2Vr(1 - \frac{\lambda m}{\lambda r})/(1 + \frac{2\lambda m}{\lambda r})}{1 - Vr(1 - \frac{\lambda m}{\lambda r})/(1 + \frac{\lambda m}{\lambda r})}, \tag{3}$$

where λ_c is the thermal conductivity of the composite, λ_m is the thermal conductivity of the matrix, λ_r is the thermal conductivity of the reinforcement, and V_r is the percentage of the reinforcement.

For the Cu-Al_2O_3 composite containing $5_{vol.}\%$ of the alumina phase, the thermal conductivity value was found to stand

at 349 W/mK (thermal conductivity of copper and alumina was assumed at 384 W/mK and 20 W/mK, respectively).

3. Results and discussion

In general, one can distinguish between two significant dissipation channels in the milling process energy conservation law for powders. Those do not react chemically with one another. The rotary motion energy of the mill, transmitted to the system with both powder mixture and the balls, is the sum of mechanical and thermal energies. Heat effects result from friction between all milling components. Both defragmentation and agglomeration of powder mixture particles follow from mechanical interactions or, to be more specific, collisions. The degree of solid phase reduction is indicated by statistical distribution. Basic estimators include the arithmetic average (Avg) and standard deviation (Std). Analysis of changes over time for these estimators makes it possible to trace concrete steps of both the aggregation and decomposition process of particles in the mixture. Continuous curves in the $Std = f(Avg)$ coordinates clearly show not only the growth, but also the destruction directions observed for powder mixture particles. In addition, they reflect the type of particle decomposition and fusion processes, thus representing a specific hysteresis of milling processes. Energy distribution between mechanical and thermal interactions in the milling process depends on the internal energy of the powder particles. Spherical powder has the smallest surface of all powder shapes with the same volume and is characterized by the lowest internal energy. Therefore, mechanical work, including deformation, defragmentation or consolidation of phases, is more intense for dendritic powder with a developed surface. In addition, milling hystereses will prove dissimilar. Figure 4 depicts hystereses in composite powders obtained for dendritic and spherical copper powders. Cu_{dend} – 5%ED Al_2O_3 and Cu_{spher} – 5%ED Al_2O_3 underwent milling in an attritor at a rotational speed of 100 rpm. In the case of dendritic powder (Fig. 4a), the entire initial milling powder energy is aimed at bonding dendrites and enclosing electrocorundum particles inside them. When reaching the average diameter and standard deviation of around 10 μm, gradual defragmentation of composite powder particles occurs. The process is linear, whereas the initial and final state become very similar in terms of geometry. For spherical powder, the process' nonlinearity (Fig. 4b) stands as proof of mixing energy distribution between plastic deformation of spherical powder and closure of particles. Once the milling process is over, the composite powder arrangement does not return to its original form or anywhere near. On the other hand, when employing the rotational speed of 200 rpm, defragmentation is observed in the case of powder mixtures (Fig. 4c,d). For dendritic copper, one can see the momentary growth of standard deviation in the powder mixture (Fig. 4c), thus proving diversified defragmentation. The spherical powder milling view shows a loop (Fig. 4d). It signifies plastic deformation of spherical copper powder in the form of flattening and, subsequently, further spheroidisation of the already formed composite powder. In theory, copper powders are dissimilar when it comes to internal energy, which results from the varying shape of these particles. A higher degree of energy is observed for the dendritic copper powder due to its shape, differing from the spherical one. SEM images of polished sections mounted in resin, along with final structures of composite powders obtained at 200 rpm, are presented in Fig. 5. In both cases, automorphism is clearly marked when it comes to the shape of the initial powders of the matrix. Composite powder based on the dendritic matrix has a shape of oblong and flat objects (Fig. 5c). In the initially spherical powder, one can clearly see the spherical and flake composite powder (Fig. 5a). Structures inside the final composite granules differ. Roll gaps and pores dominate over granulate with a dendritic matrix (Fig. 5d). Single Cu particles rolled into a composite granule are visible. The spherical powder granulate (Fig. 5b) is characterized by slight porosity and is well-assembled.

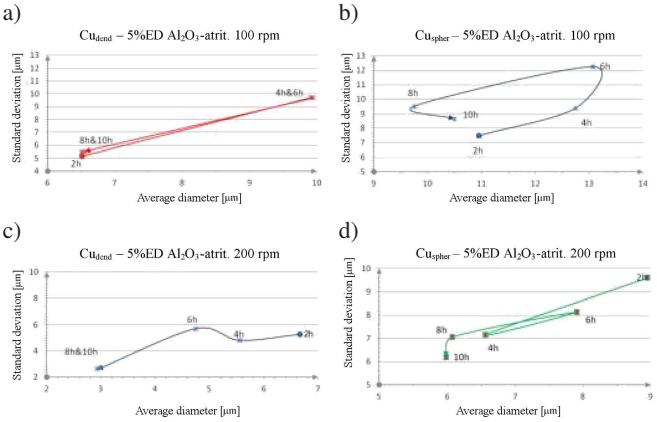

Fig. 4. Time evolution of estimated particle size distribution for composite powders obtained at a speed of 100 rpm: a) Cu_{dend} – 5%ED Al_2O_3, b) Cu_{spher} – 5%ED Al_2O_3 and at a rotational speed of 200 rpm: c) Cu_{dend} – 5%ED Al_2O_3, d) Cu_{spher} – 5%ED Al_2O_3

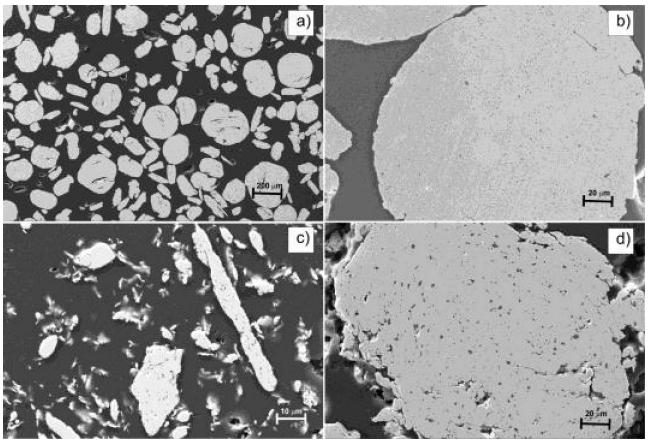

Fig. 5. Flake microstructure resulting from mechanical synthesis of powder mixtures (200 rpm): a–b) Cu_{spher} – 5%ED Al_2O_3 and c–d) Cu_{dend} – 5%ED Al_2O_3

Composite powder structures obtained when using different high-power milling times (200 rpm) are presented in Fig. 6. After 4-hour milling, a significant number of corun-

dum particles in the dendritic Cu matrix remain unbound (Fig. 6a). In the spherical matrix, they are concentrated on the outer layer of the granulate (Fig. 6c). After 10 hours of milling, electrocorundum particles are built into the inside of composite powder objects (Fig. 6b,d). All of those form a homogeneous, powder composite material.

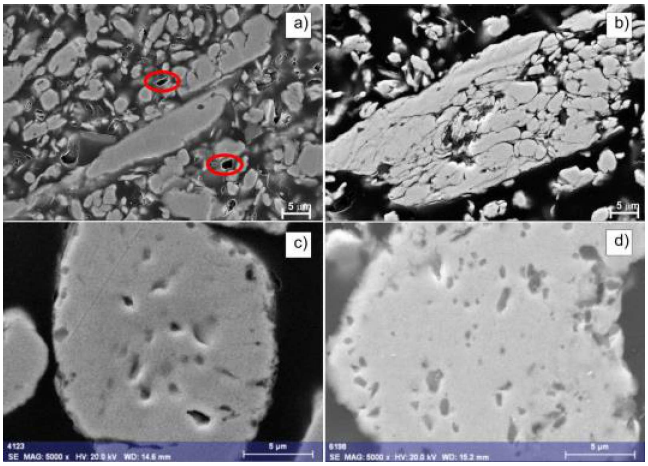

Fig. 6. Microstructure of powder mixture (SEM/EDS): Cu_{dend} – 5%ED Al_2O_3 following a) 4-hour mixing, and b) 10-hour mixing; Cu_{spher} – 5%ED Al_2O_3 following c) 4-hour mixing, and d) 10-hour mixing

During the mechanical synthesis process, the near-surface layers of powder particles get cracked and reveal a clean, active surface of the metal. Their surfaces remain un-oxidized as a result of performing mechanical synthesis in a protective atmosphere of nitrogen. Under the influence of compressive stress, placing two particles having a similar surface onto one another leads to the formation of atomic bonds between them, which results in a compound particle. As the process progresses, particles grow and are simultaneously strengthened in terms of deformation resistance. After reaching a certain, critical size and critical reinforcement, the compound particle cracks and the cycle is repeated. As a result, fragments of brittle powders reach the annealed surfaces of ductile powders and get trapped between them. Consequently, fine, fragile particles end up evenly distributed in the plastic matrix.

Having analysed the phenomena accompanying the formation of composite powder, it can be concluded that the best results were obtained when applying copper powder with grains having a spherical shape and electrocorundum as the reinforcing phase.

Research on the microstructure of sintered composites using scanning microscopy focused on the assessment of the quality of bonding between ceramic grains and the copper matrix as well as the quality of the materials, i.e. distribution homogeneity of the reinforcing phase and absence of any defects which could have a significant influence on mechanical and thermal properties of the composites. SEM images of the composite materials are presented in Fig. 7. Analysis of the microstructure showed rather uniform distribution of the reinforcing phase in the whole volume of the composites.

Physical properties of the composite materials obtained and of pure sintered copper are presented in Table 1.

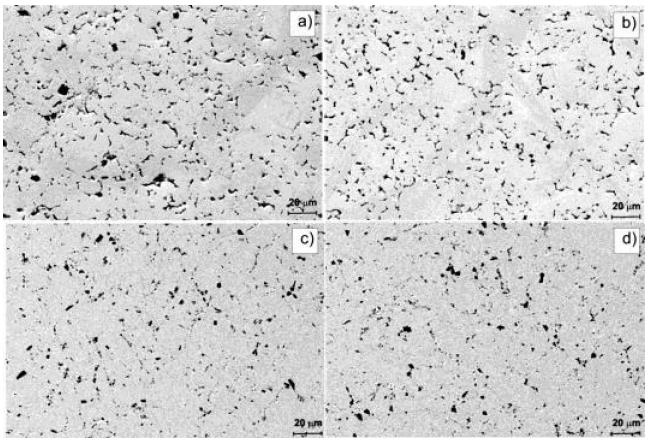

Fig. 7. SEM images of Cu-Al_2O_3 composite materials: a) Cu_{spher} – 5% α Al_2O_3, b) Cu_{dend} – 5%α Al_2O_3, c) Cu_{spher} – 5% ED Al_2O_3, d) Cu_{dend} – 5%ED Al_2O_3

Table 1
Physical properties of copper and copper-alumina composites obtained at different rotational mixing speeds and by SPS method

Materials composition [vol.%]	Measured density [g/cm^3]	Relative density [%]	Hardness HV1	Thermal conductivity [W/mK] at 50°C
$Cu_{spherical}$	8.89	99.1	40.2 ± 3.2	362.4 ± 2.0
$Cu_{dendritic}$	8.92	99.5	44.5 ± 2.6	375.1 ± 4.1
		200 rpm		
$Cu_{spherical}$ – 5% αAl_2O_3	8.44	96.7	51.2 ± 3.2	308.4 ± 0.7
$Cu_{spherical}$ – 5% ED Al_2O_3	8.65	99.2	74.8 ± 1.4	327.1 ± 1.8
$Cu_{dendritic}$ – 5% αAl_2O_3	8.41	96.5	59.9 ± 2.3	310.3 ± 2.5
$Cu_{dendritic}$ – 5% ED Al_2O_3	8.69	99.7	78.5 ± 1.7	342.2 ± 1.4
		100 rpm		
$Cu_{spherical}$ – 5% ED Al_2O_3	8.37	96.2	66.4 ± 2.9	314.7 ± 3.1
$Cu_{dendritic}$ – 5% ED Al_2O_3	8.43	96.7	68.9 ± 3.4	316.3 ± 1.9

The main concept behind the use of electrocorundum as the reinforcing phase in composites was to eliminate porosity and, at the same time, improve density of these materials. Based on research into density of the obtained composites, it was found that the application of electrocorundum as the reinforcing phase enables the production of composites with higher relative density than for the composites in which the αAl_2O_3 type of aluminium oxide was used. Electrocorundum grains do not show a tendency to create agglomerates, which, as proved by images showing their microstructure (Fig. 8), is the predominant reason for the appearance of porosity. Relative density of the Cu-EG Al_2O_3 composites is comparable with the value observed for pure copper. Moreover, the composites the starting powders of which were mixed at a higher rotational speed (200 rpm) were thickened more effectively. It was additionally concluded that density of the composites obtained using mechanical alloying and an attritor mill is higher than in the case of composites produced in a planetary mill under identical mixing conditions [7].

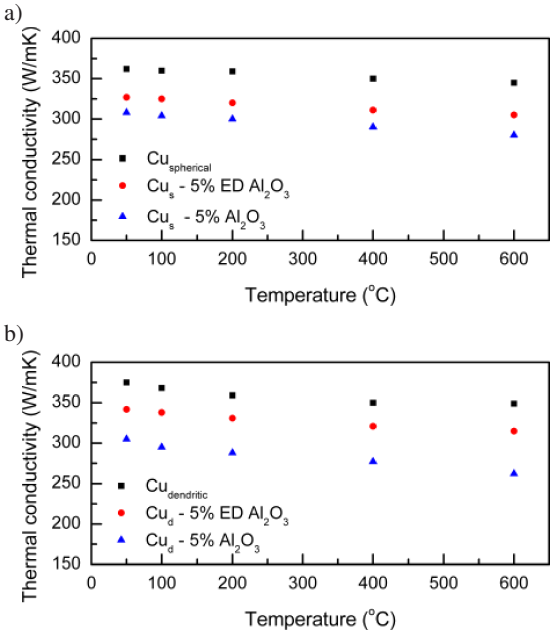

Fig. 8. Thermal conductivity vs. temperature dependence for Cu – ED Al_2O_3 and Cu – αAl_2O_3 composites: a) with spherical copper and b) with dendritic copper

The improved density results bring about the enhancement of both mechanical and thermal properties of the composites being developed. The conducted study, focused on microhardness, proves that the composite hardness is determined by the form of the starting materials used. Incomparably higher hardness values were reached for the composites based on copper with dendritically-shaped grains and electrocorundum.

Thermal conductivity of the Cu-Al_2O_3 composite materials, derived from the measurements of thermal diffusivity, is presented in a graphical form in Fig. 8. The graphs show the changes of thermal conductivity as a function of the measured temperature, depending on the type of starting powders. The presented research indicates that the form of the powders used has a significant impact on the thermal properties of the composites produced. The best results were obtained for composites with ED Al_2O_3 and dendritic copper. In the case of the copper-alumina composite containing $5_{vol.}\%$ of the alumina phase, the thermal conductivity value calculated as per (3) was 349 W/mK. The highest measured thermal conductivity of 342.2 W/mK was observed for the $Cu_{dendritic}$ – ED Al_2O_3 composite. It is very close to the theoretical value of thermal conductivity for composites with $5_{vol.}\%$ of the ceramic phase.

Preliminary observations of the composite obtained with the use of transmission electron microscopy showed that essentially the Cu-Al_2O_3 boundaries are "clean", i.e. no clear traces of presence of the third phase are observable (Figs. 9 and 10). On the other hand, the thermodynamics of interaction between copper and alumina suggests that formation of a new phase is feasible in the presented processing conditions [12]. The formation of $CuAlO_2$ in the Cu-Al_2O_3 system was reported by many research groups, including Trumble

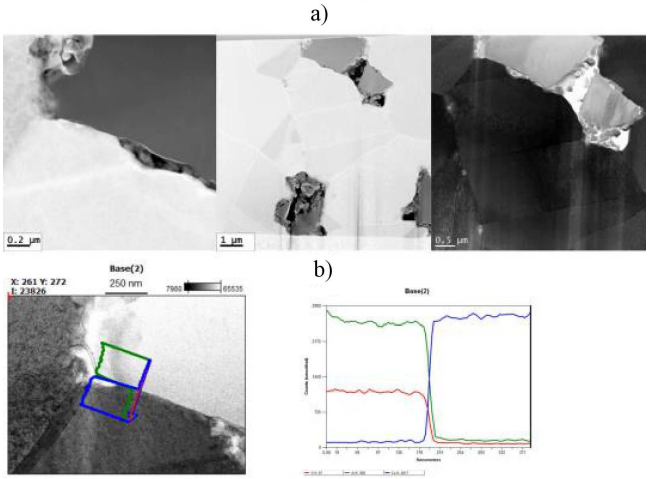

Fig. 9. a) TEM images, and b) linear distribution of elements (Cu, Al, O) across the metal-ceramic interface of the $Cu_{dendritic}$ – α Al_2O_3 composite (200 rpm)

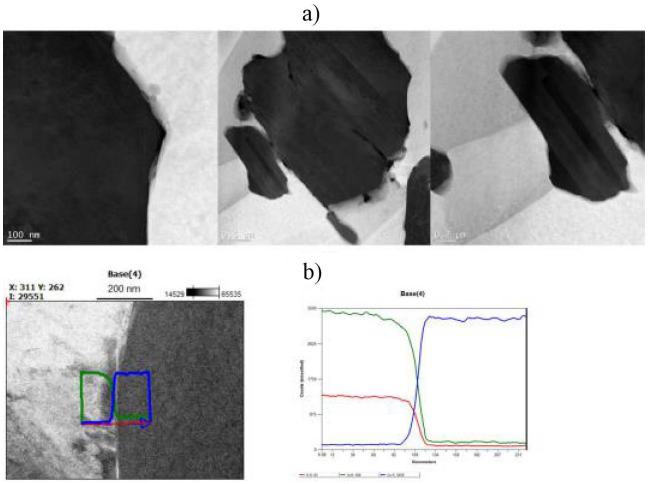

Fig. 10. a) TEM images. and b) linear distribution of elements (Cu, Al, O) across the metal-ceramic interface of the $Cu_{dendritic}$ – ED Al_2O_3 composite (200 rpm)

et al. [13], Seager et al. [14], Kim et al. [15] and Fathy et al. [16]. Furthermore, Dash pointed out that following SPS Cu-Al_2O_3 compacts exhibit annealing twins and sub-grain boundaries for both micro- and nanocomposites that might have occurred during the high temperature sintering stage. Suitable conditions for twin formation are achieved when a large number of obstacles is formed in the crystal, thus blocking the dislocation movement. TEM micrographs performed by Dash suggested formation of a third phase, i.e. $CuAlO_2$ (copper aluminate) around the alumina particles.

Furthermore, due to having used the SPS technique, it was possible to reduce the amount of Cu_2O, which emerges as a direct consequence of sticking to conventional sintering methods, e.g. hot pressing in a nitrogen atmosphere [17]. Formation of Cu_2O during sintering reduced the extent of bonding of copper with alumina, which considerably affects both density and mechanical properties. Application of the SPS method instead of conventional ones reduces the threat of vaporization, minimizes grain growth and renders the grain boundaries more defined [18, 19].

The dark areas in both images correspond to the Al_2O_3 phase, whereas the bright ones represent Cu. However, many nano-cracks and voids were observed in the interfacial areas between Cu and Al_2O_3 phases for the composite with the αAl_2O_3 ceramic phase. The observation process also revealed that the αAl_2O_3 powder grains are arranged in agglomerates (Fig. 9) and empty spaces appear between grains forming these agglomerates. When investigating the metal-ceramic coupling obtained in the composites, it is easily noticeable that a decidedly better coupling is observed for the composites in which electrocorundum was used. In these particular composites, no discontinuities are found at the interfaces.

In Fig. 11a an exemplary image of a wire following a tensile test is shown. It was taken with an optical microscope in the multi-focus mode and with polarized light in order to enhance the contrast between copper and the ceramic particle. Figure 11b and 11c present the 3D reconstruction image of the particle. The particle at the top of the broken wire can be clearly seen. There is no copper on it due to the fact that the wire was broken exactly at the interface. These images were further used to determine the contact area between the particle and copper.

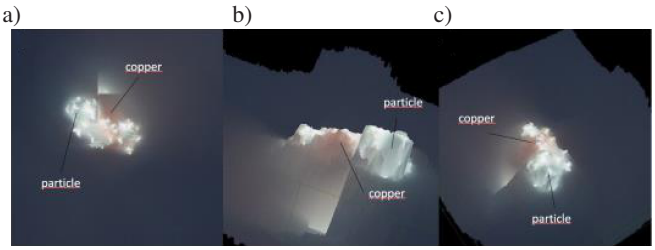

Fig. 11. Optical microscopic images of a particle at the end of a broken wire in polarized light for the sample prepared from Cu_{dend} – ED Al_2O_3: a) top view; b), c) 3D reconstruction from multi-focus image

Results of interface strength evaluation for the four samples are shown in Fig. 12.

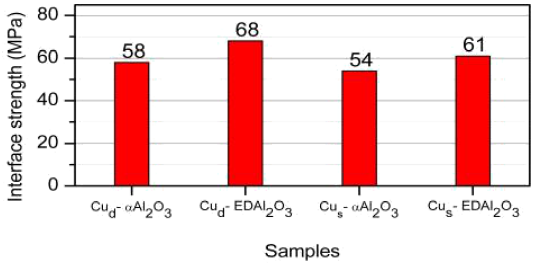

Fig. 12. Values of interface strength as depending on the form of copper and alumina phase

The form of the ceramics has a detrimental impact on the mechanical properties of the materials. The interface strength is higher for the sample with electrocorundum powders, and equals 68 $\pm$ 2 MPa for dendritic copper and 61 $\pm$ 3 MPa for spherical copper. It is worth noting that the interfacial strength value is an average of 10 measurements performed for each sample. The measurement error is based on standard deviation observed for experimental results and the inaccuracy of the contact area evaluation. Although the shape of the particles is irregular, the results are repeated (low standard deviation) because only microwires with relatively flat interface were analysed. Therefore, it can be assumed that the normal stress (tension) is dominant.

The results for strength evaluation of the ceramics-metal coupling demonstrate that it is significantly lower than the tensile strength of pure copper (227 MPa). Composite wires were found to always crack where the ceramics-metal coupling was situated. Having compared the strength values achieved for the transitory layer in the composites being examined, it can be concluded that for the composites based on copper with grains that have a dendritic shape, the outcome coupling is stronger. However, it seems that the coupling is of a rather adhesive-diffusive nature.

4. Conclusions

This paper presents analysis of the powders involved in the phenomena accompanying formation processes of Cu-Al_2O_3 composite structures through mechanical synthesis in a high-power attritor mill. Additionally, influence of the form and shape of grains in both copper and aluminium oxide starting powders on the formability of composite powder and properties of the composite materials produced is examined. Copper matrix composites with 5vol.% of the ceramic phase were obtained using the spark plasma sintering method. Application of the SPS method instead of the conventional ones reduces the threat of vaporization, minimizes grain growth and renders grain boundaries more defined.

The use of high energy mill (of the attritor type) allowed for supplying a large amount of energy to the powder mixture, which contributed to the formation of the Cu-Al_2O_3 powder composite with uniform distribution of the ceramic phase in the metal matrix. The composites thus obtained were characterized by higher density and improved thermal and mechanical properties as compared to composites whose starting powder had been mixed in a planetary mill [8, 20].

Having analysed the phenomena accompanying the formation of composite powder, it can be concluded that the best results were obtained when applying copper powder with grains having a spherical shape and electrocorundum as the reinforcing phase. As a result of using electrocorundum in copper-based composites, it was possible to limit porosity in the ceramic phase, thus contributing to improvement in thermal properties and interface strength.

Detailed analysis of influence of the reinforcement type used on formatting the transition layer and of its impact on the physical and mechanical properties of materials is of cognitive importance from the point of view of development of metal-ceramic composites. It will allow for fully controllable production of materials meeting practical requirements related to their future application.

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