



AI2O3 PARTICLE SIZE EFFECT ON REINFORCED CU COMPOSITES PRODUCED BY HIGH ENERGY MILLING

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ABSTRACT

Production of copper (Cu) composites with a reinforced Cu matrix using mechanical alloying with Aluminum oxide (Al₂O₃) particles of different sizes was achieved using high-energy ball milling procedure. The initial materials consisted of inert gas-atomized spherical electrolytic Cu powders containing 0.5 wt. % commercial Al₂O₃ powders, with particle sizes ranging from 10 μ m to 1 μ m. Cu powders with different particle sizes of Al₂O₃ were high-energy ball milled at 500 rpm for 3 hours to attain a consistent distribution of Al₂O₃ throughout in the Cu matrix. The powders that were high-energy ball milled were then subjected to cold-pressing at 500 MPa and isothermally sintered for 1.5 hours at 880°C in an Ar atmosphere. The fabricated copper composite materials were characterized using X-ray diffraction analysis (XRD), field emission scanning electron microscopy (FE-SEM), energy-dispersive X-ray spectroscopy (EDXS), density and macrohardness tests. The wear properties and mechanism were investigated through tribological pin-on-disc experiments, which revealed that the reinforcing effect was more significant when finely dispersed Al₂O₃ particles were combined into the Cu matrix compared to coarse Al₂O₃ particles.

*Keywords: Al*₂*O*₃ *particles; Characterization; Copper composite; High energy milling; Powder metallurgy*

YÜKSEK ENERJİLİ ÖĞÜTME İLE ÜRETİLEN Al₂O₃ TAKVİYELİ CU KOMPOZİTLERDE PARÇACIK BOYUTUNUN ETKİSİ

ÖZET

Farklı tane boyutuna sahip alüminyum oksit (Al₂O₃) parçacıklarıyla güçlendirilerek, mekanik alaşımlandırılmış bakır (Cu) matris malzemeler yüksek enerjili öğütme işlemi ile başarılı bir şekilde üretilmiştir. Başlangıç malzemeleri, 10 μm ve 1 μm arasında değişen parçacık boyutlarına sahip ağırlıkça % 0,5 ticari Al₂O₃ tozları ve inert gaz atomize küresel elektrolitik Cu tozlarından oluşmaktadır. Farklı tane boyutlarına sahip Al₂O₃ ve Cu tozları, Cu matrisinde homojen bir Al₂O₃ dağılımı elde etmek için 500 rpm'de 3 saat boyunca yüksek enerjili öğütme işlemine tabi tutulmuştur. Öğütülen tozlar daha sonra 500 MPa basınçta soğuk preslenmiş ve 880°C'de 1,5 saat süreyle izotermal koşullar altında argon atmosferinde sinterlenmiştir. Üretilen bakır kompozit malzemeler, X-ışını kırınım analizleri (XRD), alan emisyonlu taramalı elektron mikroskopu (FE-SEM), enerji dağılımlı X-ışını spektroskopisi (EDXS), yoğunluk ve makro-sertlik testleri kullanılarak karakterize edilmiştir. Aşınma özelliklerini araştırmak için disk üstünde pim tribometre kullanılarak tribolojik testleri yapılmıştır. Deneysel sonuçlar, ince dağılımış Al₂O₃ partiküllerinin Cu matrisine dâhil edilmesinin, kaba Al₂O₃ partiküllerinden daha önemli bir güçlendirme etkisine sahip olduğunu göstermiştir.

Anahtar Kelimeler: Al₂O₃ partikülleri; Bakır kompozit; Karakterizasyon; Yüksek enerjili öğütme; Toz metalurjisi

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1. Introduction

Copper composite materials are developed materials that uniquely combine strength, electrical conductivity, and high thermal stability, making them ideal for various applications. These copper based composites have become popular in various applications such as electrodes for resistance welding [1], semiconductors [2], catalyst [3] and electromechanical devices [4]. For them to be considered superior material, achieving a uniform structure and evenly distributed reinforcement particles within the copper matrix is crucial. Hence, they are considered promising materials for applications that demand high mechanical properties, conductivity, and wear resistance. Copper composite materials strength can be boosted considerably by either age hardening or by introducing insoluble dispersoid particles like carbides, oxides, and borides that remain thermally stable at high temperatures [5, 6]. In recent studies, copper (Cu) alloys with excellent corrosion resistance, electrical conductivity, and thermal conductivity have been explored as matrices for ceramic particle reinforced composite materials. Aluminum oxide particles (Al₂O₃) are frequently used in composite materials to enhance their hardness and ability to resist wear, due to their high levels of these properties. The use Cu and Al₂O₃ together creates various possibilities for applications that require good wear resistance, electrical conductivity and high density [7-9]. In this paper, Cu–Al₂O₃ composites were prepared by mechanochemical route and the surface morphology, particle shape and phases were examined using FE-SEM, EDXS and XRD techniques. An investigation was conducted to study the impact of various sizes of Al₂O₃ powder particles on the phase structure, matrix strengthening, relative density, and wear mechanism of Cu-Al₂O₃ composites.

2. Experimental Study

Starting materials were prepared by mixing a pre-alloyed Cu powder ($\leq 30 \ \mu$ m), with Al₂O₃ powder (10µm and 1µm) at a concentration of 0.5 wt.%, resulting in the production of Cu–10Al₂O₃ and Cu–1Al₂O₃, respectively. Mechanical alloying of the Cu/Al₂O₃ powders was achieved by planetary highenergy ball mill (Retsch PM 100- Germany) at room temperature. In order to achieve adequate blending, a ZrO₂ jar was filled with Cu/Al₂O₃ powders and ZrO₂ balls and secured onto a high-energy milling device. The powders were then milled at 500 rpm for 3 hours (The ball/powder ratio is 10:1). Following the milling process, the powders underwent H₂ treatment at 400°C for 1 hour in order to remove any copper oxides that might have developed on the surface as a result of the milling process. After mixing and milling, the powders were cold-pressed into tablet-shaped samples under 500 MPa, and then cold isostatic pressed (CIP) at 180 MPa. To sinter the specimens, they were placed in a tube-type furnace and subjected to Ar gas control, with the temperature gradually ramped up at a rate of 5°C min⁻¹ until reaching 880°C, and held for 1.5 hours. A schematic representation of the processing steps to manufacture of Cu-Cu/Al₂O₃ composites is shown in Figure 1.

To determine the relative density (%) of sintered compacts, the Archimedes' principle specified in the European Standard EN 99 (ISO 10545-3, 1991) [10] was employed and to assess the Rockwell B hardness of the Cu composite samples, a load of 60 kg-f was applied for a duration of 15 seconds. Hardness and density measurements were taken for each sample at least six times. The pin-on-disc apparatus, which conforms to the ASTM G99 test standard [11], was applied to assess the wear properties of both Cu and Cu-Al₂O₃ composites. Cu composite sample had a diameter and thickness of 18 mm and 2 mm, respectively, with a surface roughness (Ra) of 0.5 μ m after polishing. The wear resistance of the samples was examined by conducting a wear test, which involved subjecting them to varying loads of 10, 30, and 40 N while maintaining a constant speed of 1 m/s and a sliding distance of 90 m on 180 mesh silicon carbide paper. Following the wear test, the tested materials were cleansed and dried using alcohol, and their weight loss was measured by weighing them using a microbalance with an accuracy of 0.0001 g. The experiments were performed at 20-25°C, and to ensure the accuracy and consistency of the test data, a minimum of six wear tests were conducted on the samples under each operating condition. The wear rate was calculated using the average of the measured data. The polished and chemically etched (using FeCl₃+HCl+dH₂O solution) samples were examined using a FE-SEM and EDXS to observe their microstructures. The average grain size of the Cu composite samples was

calculated by examining FE-SEM photographs, using a modified line intercept method. To calculate the grain size (g), the number of grain boundary intersections with an arbitrary line was counted, as shown mathematically in Equation 1. The total length of the test line is L_T , the total number of grain boundary intersections is P, and the magnification level is M.

$$g = \frac{L_T}{PM} \tag{1}$$

A Cu-K α diffractometer (Rigaku D/MAX/2200/PC, Japan) was used to perform XRD analysis. The scan range was 10° to 90° with a step size of 0.01° and a speed of 1 s/step. The data obtained were analyzed using Jade; Materials Data computer software. The crystallite size (D) was estimated by applying the Williamson and Hall method (Equation 2) to analyze the broadening (β) of the main diffraction lines (111), (200), (220) and (311) [12].

$$\beta\cos\theta = \frac{k}{D}\lambda + \frac{k\Delta d}{d}\sin\theta$$
(2)

The shape factor k was set to 0.9, and radiation wave length λ was 0.15405 nm. The relative deviation of the lattice parameters from their mean value was used to define the average lattice distortion, represented as $\Delta d/d$ [13].



Figure 1. A schematic representation of processing steps for the manufacture of Cu/Al₂O₃ composites

3. Results and Discussion

3.1. The physical and mechanical characteristics of Cu composites

In Figure 2, the results for relative density and Rockwell B hardness of both the Cu matrix and Cu-Al₂O₃ composites are presented. The decrease in size of Al₂O₃ particles results in an increase in relative density

of the sample, which can be associated to a lower amount of porosity and a higher fraction of reinforced Al_2O_3 . In Cu composites samples, the relative density increases from 84.73% to 87.1% by decreasing the particle size of Al_2O_3 from 10µm to 1µm. When compared to the theoretical density, these values correspond to relative densities of 88.95%, 84.73%, and 87.1%, respectively. The findings suggest that the densification process, which was carried out by cold pressing pre-alloyed powders, was not completely terminated. Insufficient consolidation can be associated with various factors such as the reinforcement of the Cu matrix, the formation of dislocations by Al₂O₃ particles, and the size and shape of the powders [14]. Reducing the size of the hard Al₂O₃ ceramic particles from 10µm to 1µm has led to a notable improvement in the hardness of the Cu matrix, as demonstrated in Figure 2, with an increase from 38.2 to 46.6. As a result of milling and sintering processes, diffusion activities lead to the precipitation of fine Al₂O₃ particles within the Cu matrix and at grain boundaries. Material hardness is a physical property that represents its capacity to resist local plastic deformation. When fine particles are uniformly distributed within Cu matrix, they act as pinning points, obstructing the movement and propagating of dislocations. Consequently, the grain growth mechanism is hindered by the pinning force imposed by the fine Al_2O_3 particles on the grain boundary. By decreasing the particle size of Al_2O_3 , the Cu-1Al₂O₃ composite exhibits a smaller crystal grain size and higher hardness, attributed to stronger dispersion strengthening effects and refined grain structure of the fine Al₂O₃ particles [15].



Figure 2. The macrohardness and relative density results of the Cu, Cu–10Al₂O₃ and Cu–1Al₂O₃ composites

3.2. Tribological Properties

In Figure 3, the wear rates of Cu matrix and Cu-Al₂O₃ composites under sliding conditions are presented for different applied loads (10N, 30N, and 40N) and a sliding speed of 1 m/s. As the load increases, the wear rates for all composites increase as well. This is because the abrasive particle penetrates deeper into the Cu material, leading to more significant wear performance. Besides, the wear rates of Cu-Al₂O₃ composites were determined to be less than those of Cu matrix, and this reduction was associated to a

decrease in the particle size of Al_2O_3 present in the copper matrix. This is due to the to the excellent interfacial bonding and high hardness of the Cu matrix and particles, which in turn leads to a decrease in the actual wear surface area. The distribution of the reinforcement ceramic particles was also important in improving the abrasive wear resistance, as it causes hardness enhancement of the Cu matrix. The probability of fine Al_2O_3 particles in unit contact area gradually increased, resulting in improved wear resistance. Therefore, Cu–1Al_2O_3 composites yielded better results than Cu–10Al_2O_3 composites. Another possible explanation could be that the ceramic nanoparticles have the ability to endure the applied load and can withstand the plastic deformation of the Cu matrix [16,17].



Figure 3. The wear rate of the Cu, Cu–10Al₂O₃ and Cu–1Al₂O₃ composites

as a function of applied load

3.3. The morphology and microstructure of Cu composites

The microstructure of a material plays a significant role in determining its properties and performance. The use of mechanical alloying helps to refine the microstructure of the Cu matrix, leading to a more homogeneous and fine-grained structure. This refinement also contributes to the uniform distribution of ceramic particles, resulting in improved mechanical properties such as hardness, toughness, and wear resistance. The enhanced microstructure achieved through mechanical alloying makes it a valuable technique for developing advanced composites with superior performance characteristics. Mechanical alloying can be employed to manufacture composites that have enhanced microstructures and a uniformly distributed Al₂O₃ ceramic particles within the Cu matrix. FE-SEM micrographs of Cu and Cu/Al₂O₃ powders, following mechanical alloying for 3 hours at 500 rpm, are displayed in Figure 4. The shape of the Cu and Cu/Al₂O₃ powders changes from spherical to more flake-like granular morphology due to the shearing effect of the ZrO₂ balls during mechanical alloying, as shown in Figure 4 and FE-SEM images of the starting powders can be seen in Figure 1. During the milling process, a layered microstructure is developed by the Cu powder particles, and a highly consistent distribution of Al₂O₃ particles can be observed in both Cu–10Al₂O₃ and Cu–1Al₂O₃ composites. In Figures 4b and 4c, it is evident that the Al₂O₃ particles are largely embedded at the interfaces between the clashing Cu

particles without any distortion. The microstructure/morphology of the Cu composite and the distribution of elements in its surface morphology were examined using FE-SEM. Figure 5 shows the results of this investigation, as presented in the EDXS mapping images. The microstructure analysis suggests that the Cu samples have undergone adequate sintering and densification.



Figure 4. FE-SEM micrographs of (a) Cu, (b) Cu–10Al₂O₃ and (c) Cu–1Al₂O₃ powders after high energy ball milling

Figures 5(b) and 5(c) display the results of EDXS analysis at two specific points (+1, +2) in the Cu-10Al₂O₃ and Cu-1Al₂O₃ composites. The images show the presence of a second dispersed phase comprising well-bonded Al₂O₃ grains, and a fairly consistent distribution of Al₂O₃ particles within the Cu matrix. The brighter area (marked as +1) is identified as the Cu matrix, while the dark region (marked as +2) is presumed to be Al₂O₃. It is observed that the produced Cu and Cu composites have a nonporous structure and exhibit a more homogeneous structure with a decrease in the size of Al₂O₃ particles in the Cu matrix. The embedding of Al₂O₃ nanoparticles into the Cu matrix is due to their high atomic diffusivity during high-energy milling. This allows for a homogeneous distribution of the ceramic reinforcement phase within the metal matrix, leading to improved physical and mechanical properties as well as wear resistance properties of the metal matrix composites obtained with the presence of second-phase particles [18]. The scanning results of the surface composition indicate that the secondary ceramic phase particles within the metal matrix are uniformly distributed and that the Al₂O₃ particles are covered by the Cu matrix.

The analysis detected elementary points of Cu, O, and Al, which corresponded to the composition of the Cu matrix and Al_2O_3 particles, respectively. The surface scanning also revealed the presence of the $Cu_xAl_yO_z$ phase, [19] as interlapping of all the three elements was noticeable. The Al_2O_3 particles possess characteristics such as high hardness, thermal stability, corrosion resistance, and chemical inertness, along with a high melting point that protects them from melting or coarsening during annealing with Cu. As a result, these particles effectively accelerate the grain refinement mechanism within the Cu matrix. By impeding the motion of dislocations at certain slip planes along the grain and sub-grain boundaries, they enhance the strength of the composite material when exposed to high temperatures [19,20].

In all FE-SEM photographs, the dark regions correspond to the Al_2O_3 matrix/particles, while the brighter regions indicate the Cu matrix. The grain size of the Cu and Cu composite samples was measured by adapting the line intercept method, which involves relating the length of a line placed randomly across the width of the FE-SEM image to the total number of grain boundaries intercepted, and the magnification applied, as shown in Equation 1. After sintering, the grain size of the Cu matrix without reinforcement phase was between 20 and 24.5 μ m, while that of the Cu–10Al₂O₃ composite was between

9-12 μ m, and that of the Cu–1Al₂O₃ composite was between 6-8.5 μ m. The smaller particle size of 1 μ m in the Cu-Al₂O₃ composite may be attributed to the higher deformation experienced by smaller powder particles during high energy milling than coarser particles. These observations were further verified by the FWHM results of all Cu samples. (see Figure 7a).



Figure 5. FE-SEM micrographs and EDXS analysis of (a) Cu, (b) Cu–10Al₂O₃ and (c) Cu–1Al₂O₃ composites after sintering *ADYU Mühendislik Bilimleri Dergisi 20 (2023) 125-134*

3.4. Phase Identification (XRD)

The identification of phases presents in the sintered Cu samples and their estimated crystallite size from FWHM data were carried out using XRD. The sintered samples were ground clean and analyzed using XRD to ensure that no trace phases appeared in the composite samples. Figure 6 depicts the XRD pattern obtained for the Cu samples following the sintering process. The sharp peaks in XRD patterns are associated with the Cu phase, whereas the Al_2O_3 phase is characterized by low-intensity peaks. Previous literature researches [2,21] indicates that there is a low-intensity peak present in the structures of Cu/Al_2O_3 composites. This peak corresponds to the Cu_xAl_yO_z phase, which is formed due to the eutectic reaction between Cu and Al_2O_3. It is thermodynamically feasible for the second phase to form when there is contact between surfaces of Cu and Al. Cu and Al_2O_3 are joined through eutectic reaction, heating up to the eutectic temperature causes the eutectic point to enlarge and react with Al_2O_3, resulting in the formation of Cu_xAl_yO_z phase. This compound is capable of being compatible with both phases present at the inter-surface.



Figure 6. X-ray diffraction patterns of the sintered composites at 880°C for 1.5 h (a) Cu matrix (b)

Cu-10Al₂O₃ and (c) Cu-1Al₂O₃

The XRD analyses of the Cu composites examined explain that decreasing the size of Al₂O₃ particles leads to an improvement in line broadening, as evidenced by FWHM. Figure 7 shows the FWHM as well as estimated crystallite size values of Cu matrix, Cu–10Al₂O₃, and Cu–1Al₂O₃ composites after sintering at 880°C for 1.5 h. According to Figure 7a, the FWHM of the pre-alloyed Cu powder, which has been reinforced with 1 μ m Al₂O₃ particles, is greater than that of the Cu-Al₂O₃ composite that has an average particle size of 10 μ m. This implies that the initial size of Al₂O₃ particles greatly affects the FWHM during the milling process. The FWHM values of the Cu-10Al₂O₃ composite are slightly higher compared to Cu matrix. This suggests that the impact of the Al₂O₃ powder particles (10 μ m) used in the initial stages has a greater influence on the FWHM. According to Figure 7b, the estimated crystallite sizes for the sintered composites of Cu matrix, Cu-10Al₂O₃, and Cu-1Al₂O₃ were 538±13 nm, 485±9 nm, and 456±7 nm, respectively. The lattice distortion and crystallite size refinement resulting from high-energy planetary milling and a lower second-phase particulate lead to progress in FWHM broadening, which are crucial factors affecting powder compacting throughout the sintering procedure and characteristics of the Cu matrix reinforced by fine dispersoids [14,22]. In Cu composites, the

existence of Al₂O₃ particles that are finely dispersed and uniformly distributed hinders the growth of grains [23,24].



Figure 7. (a) Full width at half maximum (FWHM) and (b) estimated crystallite size values of sintered Cu matrix, Cu–10Al₂O₃ and Cu–1Al₂O₃ composites

4. Conclusions

In conclusion, the copper based Cu/Al₂O₃ composites were produced via a process that involved highenergy milling and cold pressing. Physical and mechanical property tests were performed on the Cu composites and it was concluded that there was a correlation between the reduction in particle size of Al₂O₃ and the increase in relative density and macrohardness of the composites. The crystal grain size of the Cu-1Al₂O₃ composite was smaller and its hardness was higher than that of the Cu-10Al₂O₃ composite, which is associated to the stronger dispersion and grain refinement effects of the fine Al₂O₃ particles. The experimental investigation also explored the tribological characteristics of the composites, the wear rates decreased. The arrangement of Al₂O₃ reinforcement particles within the copper matrix was a crucial point in enhancing the capacity of the material to withstand abrasive wear. Finally, the microstructure analysis showed that the Cu samples were sufficiently densified and sintered, and the Al₂O₃ particles were mostly embedded at the interfaces between the Cu particles. Overall, the highenergy ball milling and cold pressing method proved to be an effective method for producing Cu-Al₂O₃ composites with improved properties.

Conflict of Interest Statement

The author of the article declare that they do not have any personal or financial conflicts of interest with any institution, organization, or person.

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