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Effect of Mechanical Alloying Time on Microstructure, Hardness and Electrical Conductivity Properties of Cu-B_4C Composites

Hasaneen Houssain[a, b*], Ahmet Oğuzhan Cengiz[c], Serkan Islak[c]

[a]Institute of Science, Kastamonu University, Kastamonu, Türkiye
[b]Department of Chemical Engineering, Faculty of Engineering, Al Muthanna University, Samawah, Iraq
[c]Department of Mechanical Engineering, Faculty of Engineering and Architecture, Kastamonu University, Kastamonu, Türkiye
***Corresponding Author:** hasanein.mahbuba@gmail.com

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Abstract: This study aims to investigate the effect of mechanical alloying time on the microstructure, hardness, and electrical conductivity properties of copper (Cu) matrix boron carbide (B_4C) reinforced composites. Cu-B_4C composites with 2% B_4C by volume were subjected to mechanical alloying processes for 0, 1, 5, 10, and 20 hours. The microstructure and phase formation of the composites were examined using scanning electron microscopy (SEM) and X-ray diffraction (XRD). Hardness measurements of the composites were conducted using the microhardness measurement method, and density values were determined using the Archimedes principle. The electrical conductivity values of the samples were measured in terms of the international annealed copper standard (%IACS) based on the eddy current principle. SEM images revealed a more homogeneous distribution of B_4C particles in the Cu matrix as the mechanical alloying time increased. Hardness values showed significant increases with the increasing mechanical alloying time, reaching the highest value in the 20 h milled sample with a 90.86 value. The effect on electrical conductivity values was noteworthy, with a measurement of 63% IACS at 0 hours and 25% IACS at 20 hours of mechanical alloying.

Keywords: Cu-B_4C composites, mechanical alloying time, electrical conductivity, microstructure, hardness

Öz: Bu çalışma mekanik alaşımlama süresinin bakır (Cu) matrisli bor karbür (B_4C) takviyeli kompozitlerin mikroyapı, sertlik ve elektriksel iletkenlik özelliklerine etkisinin araştırılmasını amaçlamaktadır. Hacimce %2 B_4C ilave edilen Cu-B_4C kompozitler 0, 1, 5, 10 ve 20 saat mekanik alaşımlama işlemine tabi tabi tutulmuştur. Kompozitlerin mikroyapı ve faz oluşumu taramalı elektron mikroskobu (SEM) ve X-ışınları difraktometresi (XRD) ile incelenmiştir. Kompozitlerin sertlik ölçümleri mikrosertlik ölçüm yöntemi ile yoğunluk değerleri ise Arşiment prensibi ile ölçümü gerçekleştirilmiştir. Numunelerin elektriksel iletkenlik değerleri girdap akım prensibine göre uluslararası tavlanmış bakır standardı (%IACS) cinsinden ölçülmüştür. SEM görüntüleri mekanik alaşımlama süresi arttıkça B_4C tanelerinin Cu matrisinde daha homojen dağıldığını göstermiştir. Sertlik değerlerinde ise mekanik alaşımlama süresinin artmasıyla belirgin artışlar olmuş olup, en yüksek değer 20 saatlik numunesinde 90.86 değeri ile olmuştur. Elektriksel iletkenlik değerleri üzerine ise mekanik alaşımlama süresinin kayda değer seviye olmuştur. 0 saat değerinde %63 IACS iken, 20 saat mekanik alaşımlama değerinde ise %25 IACS ölçülmüştür.

Anahtar Kelimeler: Cu-B_4C kompozitler, mekanik alaşımlama süresi, elektriksel iletkenlik, mikroyapı, sertlik

1. Introduction

Metal matrix composites (MMCs) are a type of material that combines a continuous metallic matrix with a reinforcement, typically made of ceramic. The matrix can be made of various metals and their alloys, including aluminum, titanium, copper, nickel, and magnesium. The ceramic reinforcement is usually made of materials such as oxide, carbide, nitride, or borides [1-3]. Copper and its alloys are widely used in industrial applications, especially in areas where conductivity plays a significant role due to their unique combination of excellent thermal and electrical properties, low cost, ease of manufacturing, and good corrosion resistance [4, 5]. Nevertheless, pure copper has some significant drawbacks, such as low strength, high coefficient of thermal expansion (CTE), and generally poor mechanical properties. One of the most effective ways to address these limitations is to reinforce copper with ceramic particles to create composites with superior properties without causing any weakening of both thermal and electrical conductivities of copper [6]. Much research produced MMCs with developed properties, such as designing Cu-based alloys reinforced with ceramics. Fathy and El-Kady [6] used powder metallurgy (P/M) technology to fabricate copper-alumina composites and successfully controlled the thermal properties of the produced copper alloy. Shaik and Golla [7] prepared Cu–ZrB_2 composite to improve copper mechanical properties and control its thermal features, they conducted their investigation utilizing the ball milling technique for 20h and consolidating the mixed powder by the hot-pressing method. Understandably, the properties of MMC are affected by milling conditions like milling time, rotating speed, milling environment, etc., however, many researchers studied the influence of milling time on the microstructure, as well as, the forming of the phases of Cu matrix-based alloys [8, 9]. Nevertheless, the effect of milling time on the properties of Cu matrix alloys is still not fully studied. Therefore, in this research, Cu-B_4C matrix composite alloys were prepared and the effect of milling times on microstructure as well as physical and mechanical properties were investigated.

2. Material and Method

In this work, 98% Cu and 2% B_4C powders with a purity of more than 95% and 325 mesh were utilized for fabricating Cu-B_4C composite alloy, the raw powders were supported from Nanografi Nanotechnology Company, as received powders mixed using mechanical mixing for variant mixing times while the other milling conditions remained constant to investigate the effect of mixing durations on produced alloys properties, the process parameters are given in Table 1. Then gained mixed powders were cold pressed into a cylindrical mold of 20 mm in diameter and 5 mm in thickness using a hydraulic press device at 700 MPa pressing load. Then the green alloys underwent a sintering process for 60 minutes at 850 ºC utilizing a tubular furnace with argon gas atmosphere. The schematic of alloys fabricating process illustrates in Figure 1.

Table 1. Powder composition and process parameters

Sample group	Compositions (vol.%)		Mechanical alloying time (Hour)	Sintering Temperature (ºC)	Sintering Time (Minutes)
	Cu	B_4C			
CB0	98	2	0	850	60
CB1	98	2	1		
CB5	98	2	5		
CB10	98	2	10		
CB20	98	2	20		

The samples were prepared by abrasion and polishing process using 320-1500 grade sandpaper and polished using 1um of diamond solution. The etching process was achieved using Kroll etchant to conduct a microstructure investigation test. Microstructure examination and phase analysis were achieved utilizing the scanning electron microscope brand FEI QUANTA 250 FEG and X-ray diffraction (Bruker D8 Advance). The experimental densities of the samples were calculated depending on Archimedes principal meeting with ASTM B 962 [10]. The hardness of the samples was evaluated using the Shimadzu HMV G21 model microhardness device according to the ASTM E92-17 standard ("Standard Test Methods for Vickers Hardness and Knoop Hardness of Metallic Materials," 2017). The hardness load was 2 Kg, and 15 seconds of holding time. Furthermore, to study the effect of B_4C and Mixing duration on electrical properties, the electrical conductivity test was conducted by a Nortec 500 D device utilizing the Eddy current principle according to TS EN ISO 15549 standards.

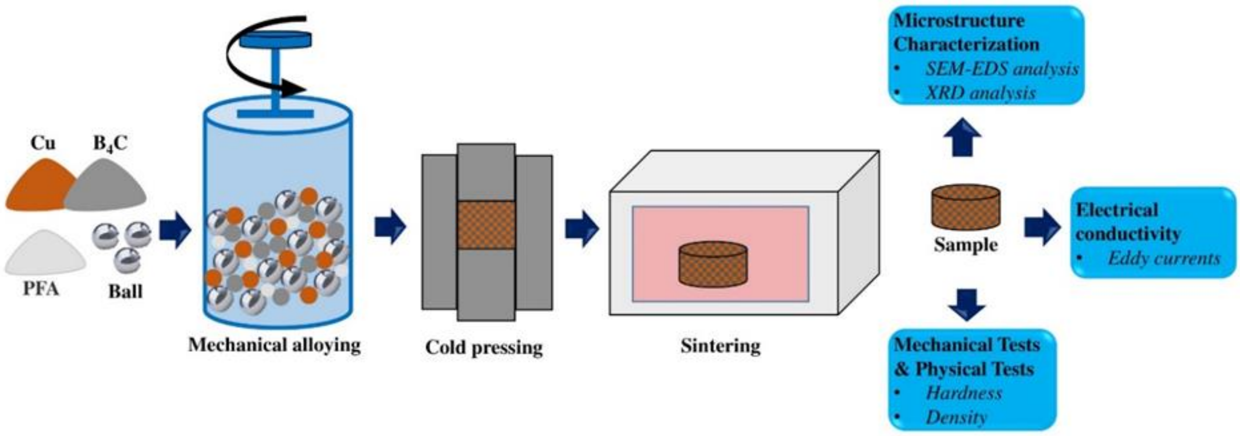

Figure 1. Flow chart of production and applied testing and analysis

3. Result and Discussions

Figure 2 shows the XRD patterns of Cu-B_4C alloys ball milled for 0, 5, and 20 hours. The patterns indicated the existence of the Cu phase only, which can be attributed to the low B_4C content of 2 % in the Cu Matrix. Shik et al. [7] in their work, related the appearance of just the Cu phase in XRD pattern due to low content of ZrB_2 up to 5%. Hamid et al [5] in their study, indicated existing only Cu peaks in the XRD pattern of Cu sample containing 3% wt. of TiC and Al_2O_3; they attributed that to the small amount utilized of both TiC and Al_2O_3. More significantly, as can be seen in (Figure 2), the XRD patterns of the samples milled for variant times showed more sharpness intense, and narrow peaks as the milling time increased which confirmed the finer grain size and more crystallinity. The study of the grain size and investigation of the effect of milling time on particle size is essential in understanding the properties of the Cu–B_4C alloys. The broadening of XRD peaks was employed to estimate the particle size using Debye-Scherrer (Equation 1). The following formula was performed [11]:

$$\boldsymbol{D = \frac{0.9\,\lambda}{\beta\cos\theta}} \qquad \text{Eq (1)}$$

Where D is the particle size, β is the full width at half maximum height, θ is the peak Bragg's angle, λ is the X-ray wavelength.

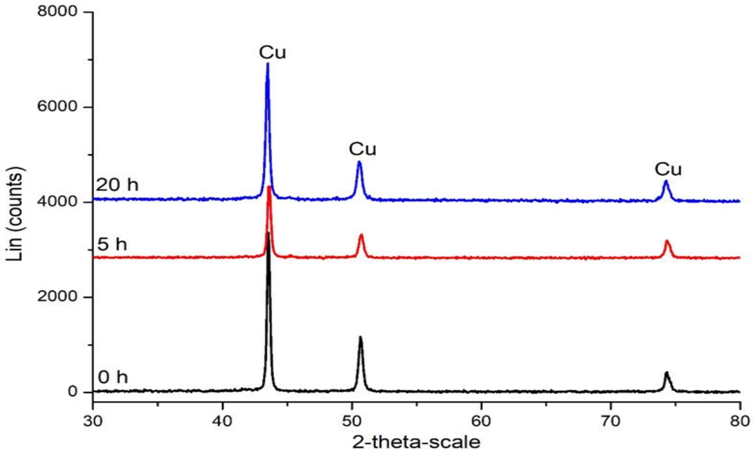

Figure 2. XRD pattern of Cu-B_4C milled for 0, 5, and 20 hours

Figure 3 illustrates the influence of the milling time on the crystallite size. A clear decrease in the crystallite size can be observed with a longer milling time, where the particle size of the alloy produced without mechanical mixing was 36.79 nm, while the alloy milled for 5 hours had a reduction in particle size to 33.37 nm. Furthermore, the crystalline size was reduced to 32.87 nm for the alloy that was milled for 20 hours. This reduction in particle size with longer milling time can be attributed to various factors such as dominant fracture mechanism, severe work hardening, and collisions between the ball-to-wall, ball-to-ball, and powder-to-powder; these factors result in the fracturing of the powder particles into smaller particles and as a result a smaller crystal size of alloy [12]. These outcomes met with other literature; in their work, Liu et al. [13] achieved a smaller particle size of Al composite alloy with a prolonged mixing time of up to 10 hours. In their research, Salur et al. [12] recorded that the average crystallite size decreased as milling time increased.

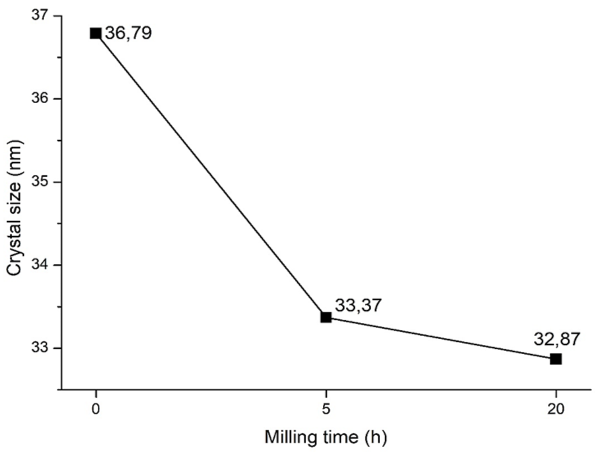

Figure 3. Effect of milling time on crystal size

Figure 4 shows the SEM images of Cu-B_4C sampled milled for 0, 1, 5, 10, and 20 hours. The size and shape of particle and crystal structure of alloys that are processed by the mechanical alloying approach are extremely conditional on the processing parameters. The most influential processing parameters are the milling time, milling environment, ball-to-powder ratio, milling speed, and process control agent [11]. It is seeable from the SEM images in (Figure 4a-f) that there is almost no porose occurring in the samples, indicating that a compact structure has been constructed. Moreover, as can be observable from SEM images, the structure of the alloys is a constituent from the Cu matrix grain with particles of

B_4C distributed through the Cu matrix. EDS analysis (Figure 4f) supports the appearance of Cu, B, and C, confirming the formation of the mentioned structure. However, (Figure 4a) represents the initiated sample without milling, the main view of the matrix structure is a long fragment of matrix particle with almost non-uniform shape and size and the B_4C particles diffused through the matrix. As the milling time is conducted for 1 hour (Figure 4b), the alloy matrix structure becomes a flaky-like form and more uniform compared to the initial alloy due to cold welding during milling [7]. As the milling time progresses for 5 hours (Figure 4c) more flattening and uniform can be observed, again, further mechanical welding effects with a longer milling time become evident. the particles of a relatively larger size which flattened after 1 hour of milling, begin to break gradually after 2 hours of milling. These flattened particles further get fragmented and exhibit an asymmetrical behavior, resulting in a decrease in the average particle size [12]. This observation is supported by crystallite size chart (Figure 3). when 10 hours of milling time is achieved, (Figure 4d), the structure of alloys tends to be more homogenous and the grains become considerably smaller in contrast to lower milling time alloys. Again, the further milling time led to more particle fractions and more cold welding which reflected positively in the diffusing of B_4C through the matrix, which is clearly observable from the SEM image (Figure 4d) which shows the more uniformity distributed of B_4C throughout the Cu matrix. Shaik et al [7] achieved a uniform distribution of ZrB_2 in the Cu matrix when achieving a 10 h milling time. Furthermore, when milling time progressed to 20 h, (Figure 4f), the microstructure becomes clearly defined, as can see, the grain and grain boundary are clearly shown without flaky structure, and the structure becomes more compact and the B_4C distributed with further homogenously in Cu matrix as notable from (Figure 4f) of mapping image for elements distribution. This can be described by when the ball milling process exceeds 10 hours and the mixing process reaches 20 hours, it enters into a steady state. In other meaning, there is a dynamic balance between fracture and cold-welding mechanisms, which occurs in the final stage of ball milling, during this stage, the particles within the system experience approximately the same rate of change, leading to the reduction of larger particles and the growth of smaller particles at a similar rate, this action results in the particles becoming roughly equal in shape [8, 14]. This behaver also helps the reinforcement particles to diffused uniformly throughout the matrix [7].

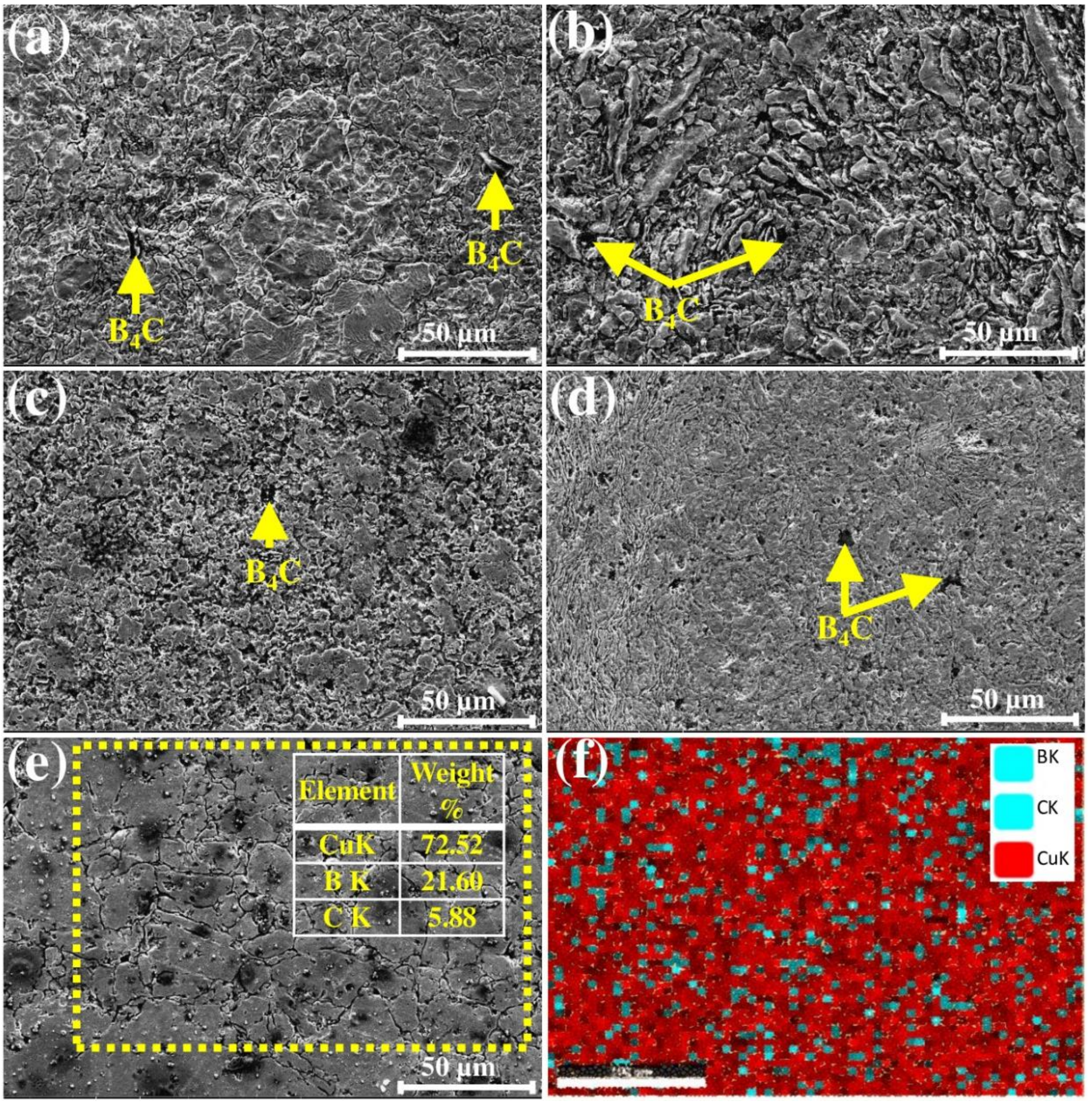

Figure 4. SEM images of samples milled for (a) 0 h, (b) 1 h, (c) 5 h, (d) 10 h, (e) 20 h and EDS analysis, (f) MAP-EDS

Figure 5 illustrates the impact of mixing time on the experimental density, relative density, and porosity of Cu/B_4C composites. As per the graph, both experimental density and relative density reduce, and porosity increases with an increase in mixing time. The experimental densities of alloys mixed for 0, 1, 5, 10, and 20 hours were 7.48, 7.29, 7.04, 6.63, and 6.50 g/cm^3, respectively. Similarly, the relative densities were 84.72%, 82.59%, 79.76%, 75.15%, and 73.63%, respectively. This reduction in density is due to the low density of the B_4C reinforcing element (2.52 g/cm^3) compared to the Cu matrix (8.94 g/cm^3). Zhou and Duszczyk [15] added SiC to Aluminum alloy type AA2014 and fabricated Al/SiC alloy. They reported a decrease in densities when adding SiC. Similarly, Kriewah and Islak [16] reported that reinforcement material with low density compared to the matrix reduces the density of the composite. The decreasing density with increasing mixing duration may related to that the longer mixing time means a more homogenous distribution of ceramic particles through the matrix, moreover, also attributed to B_4C particle fragmentation which restricts the rearrangement of matrix metal powder particles during sintering [17]. The reduction in relative densities is due to two reasons. Firstly, the fact that the ceramic particles added to the metallic matrix negatively affect the sintering ability and prevents the matrix particles from necking. Secondly, related to the difference in melting temperature between the matrix and the reinforcement elements which means more porose in the final alloy microstructure [18]. However, the porosity rates were estimated as 15.27%, 17.4%, 20.23%, 24,84%, and 26.36%, for 0, 1, 5, 10, and 20 hours of mixing duration, respectively. The increase in porosity can be associated with the decrease in relative density.

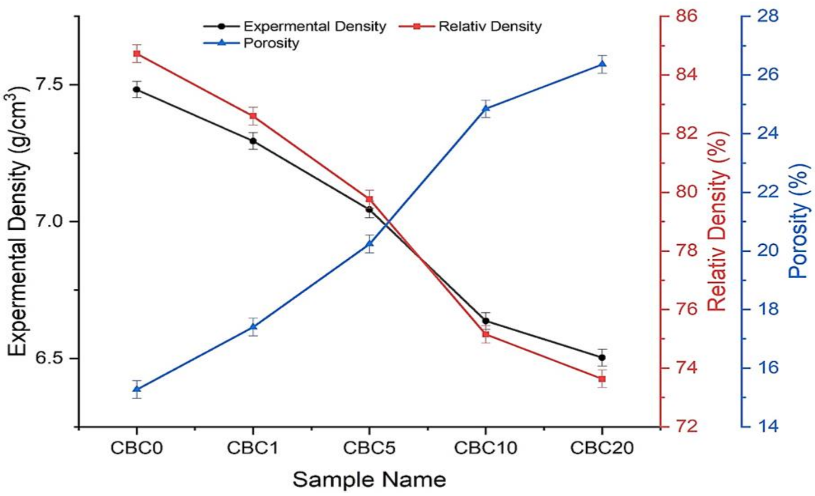

Figure 5. Effect of milling time on Experimental density, relative density, and porosity

Figure 6 shows the effect of mixing duration on hardness values, which is ceramic alloy composites' most important mechanical property. Vickers hardness values of samples mixed for (0, 1, 5, 10, and 20) hours were (56.82, 67.22, 80.58, 87.62, 90.86) respectively. The increase in hardness clearly can be noted as the longer mixing duration. The hardness increase was about 59.9% for the sample mixed for 20 hours compared to a sample without mixing. The increase in hardness with increased milling time attributed to the longer time means more fine powder particles and decreasing in primary grain. Also, more mixing duration leads to a more homogenous distribution of B_4C, and the B_4C may behave as an obstacle to primary grain growth, causing smaller grain as a result [13]. Some researchers attributed that the finer particles mean the greater interfacial area between reinforcement and matrix phases [19]. Moreover, the Hall-Petch (Equation 2) indicated that the hardness is inversely relation to grain size.

$$\boldsymbol{H = H_0 + KD^{-1/2}} \quad \text{Eq (2)}$$

Where H is hardness, H_0 and K is a constant related to value of hardness and D is crystalline size [20]. The prolonged mixing time leads to excessive plastic deformation of alloyed powders and exposure to the work hardening mechanism, which leads to raises the hardness and brittleness of powders and causes them to show higher hardness values [21]; furthermore, the B_4C particles with longer mixing time dispersed homogeneously through the matrix, which means more diffusion of hardening powders, and also increased the density of dislocation, as a results increment of hardness values of final alloys [12]. Lokesh and Karunakara [22] obtained the same results, where they found that the hardness of Al-Cu/B_4C alloys increased with a prolonged milling time.

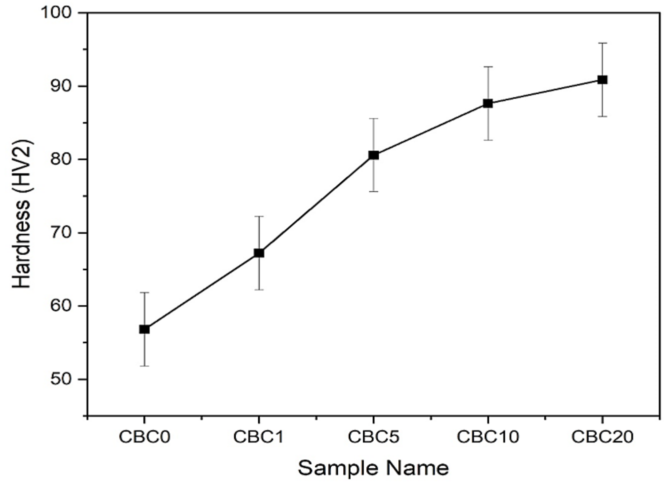

Figure 6. Effect of milling time on hardness

Figure 7 presents the effect of mixing time on the electrical conductivity of Cu-B_4C alloy. As can be seen from the charts, the electrical conductivity decreased from 63% IACS to 25% IACS. The electrical conductivity of Cu-B_4C alloy lowered by about 2.5 times compared to sample without mixing. This can be related to the increasing in porosity which means more void that filled with air inside microstructure which act as isolators of electrical conductivity, also the lattice strain and presence of more-fine ceramic (B_4C) particles due to prolonger mixing time [23]. And as the B_4C has extremely low conductivity, the Cu matrix conductivity continuity broken as B_4C particles networks are formed as a result of more homogenous diffusion through the Cu matrix which is occur with prolonging mixing time [24]. These results align with the literature of Guo et al [25], where they succeeded in decreasing the electrical conductivity of Cu-CrB_2 alloys with longer mixing times. Altinsoy et al [26] observed a decrease in the electrical conductivity of copper when B_4C was added, they attributed this result to the increase in porosity within the copper matrix, which led to a decrease in both relative density and overall density. Thus, this higher porosity acted as an insulation barrier, hindering the passage of electrons between the grains of the copper matrix.

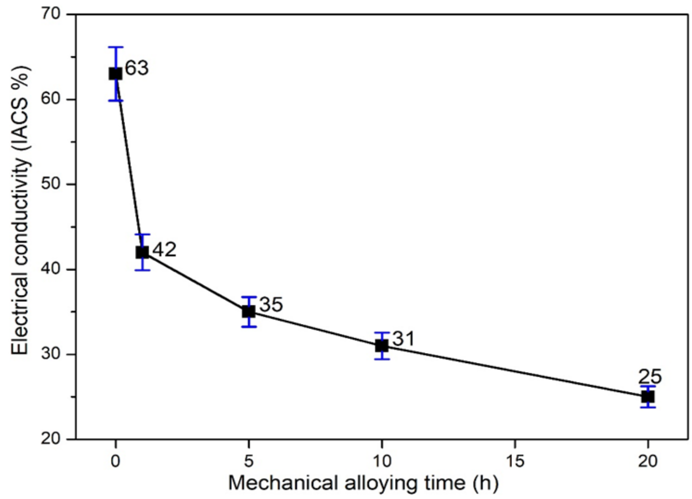

Figure 7. Effect of mechanical alloying time on electrical conductivity

4. Conclusion

The following outcomes were obtained when investigating the effect of milling time on the properties of a Cu matrix composite reinforced with B_4C, fabricated by powder metallurgy approach.

Microstructure inspection showed that as milling time progressed, the microstructure became more homogeneous and the grain became finer due to the work welding effect. At 20 hours of mixing, a steady-state was achieved, where the work welding and fraction effect were almost at the same level. This resulted in a more uniform microstructure and better diffusion of reinforcement particles throughout the matrix. These results were also supported by EDS analysis and mapping images in addition to the calculation of crystallite size.

With an extended milling time, a reduction in both experimental and relative densities occurred, along with an increase in porosity. However, there was a considerable improvement in hardness values. The electrical conductivity gradually decreased with increasing milling time.

Conflict of Interest

All authors certify that they have no affiliations with or involvement in any organization or entity with any financial interest or non-financial interest in the subject matter or materials discussed in this manuscript.

Ethics Committee Approval

Ethics committee approval is not required.

Author Contribution

Conceptization: HH, SI, AOC; methodology and laboratory analyzes: HH; writing draft: HH, SI, AOC; proof reading and editing: HH, SI, AOC. Other: All authors have read and agreed to the published version of the manuscript.

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