Influence of SiC Content on Microstructure, Dislocation Density and Mechanical Behavior of Cu/SiC Composite

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Abstract: The present investigation has examined the impact of micro-SiC on microstructure, dislocation and mechanical behavior of Cu/SiC composite. The micro-composite samples have been fabricated under a constant pressure (480 MPa) and sintered temperature (860oC) for 2 h. The sintering process was performed under argon gas. The microstructure examination was conducted using SEM/EDS and XRD diffraction. The SiC contents were 0, 5, 10,15,20,25 and 30 volume fraction. The outcomes showed that the density was significantly decreased with an increase of silicon carbide content. The relative densities of Cu and Cu/SiC composites was ranged from 91.24% to 83.56% for pure Cu and Cu/30 vol%SiC composites. The copper crystallite size was reduced with growing SiC content while the hardness, ultimate and yield compressive strength increased with increment of SiC volume fraction to 20% vol. The values of hardness, ultimate and yield compressive strength increased to 231 HV,343 and 176 N/mm2, respectively for the composite sample containing 20% SiC particles with a percentage increase of 75%,26.6% and 57.2% compared with pure Cu.

Keywords: Powder metallurgy, Composites, grain size, Dislocation density,

Compressive strength

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1. Introduction:

Because of its outstanding electrical properties, thermal conductivity, corrosion resistance and ductility, copper is considered one of the most important metals in engineering applications [1-3]. The low strength of Cu-matrix, on the other hand, is a severe issue that prevents copper from being used in many fields [4]. Cu-matrix composites have recently been demonstrated to be potential prospects in a number of high-tech applications, including railway overhead current collection systems and homopolar machines [3,4]. The possible reinforcements for the copper matrix include different types such as carbides, oxides, nitride and borides which have thermally stablability at high temperatures [5]. Because of its employment as reinforcement in matrix composites, SiC particles are of tremendous technological value.

Recently, Cu/SiC composites have become a substantial investigation due to their outstanding thermal and electrical conductivity, high hardness values and wear resistance [6,3,7-9]. Effect al [10] found that increasing SiC contents drops the electrical conductivity of Cu/SiC. Somani et al [11] found that increment of SiC to 20 mass % enhances the mechanical properties of Cu due to increasing the interfacing between SiC and Cu particles while the density illustrated a reverse tendency. Metwally et al [7] reported that Cu/SiC corrosion resistance was improved with increment of SiC content due to properties of the protective copper oxide films and reducing the microgalvanic couple between SiC and Cu. Akbarpour et al [12] illustrated that the friction coefficient increased with growing SiC contents. Further they reported that the predominant wear mechanism was flake formation spalling and abrasion. However, with increment of SiC vol% the flake formation spalling wear was reduced. On the other side, Prosviryakov [13] indicated that the increase of SiC concentration above 25 wt. % lowers the hardness of Cu/SiC composites by increasing porosity and decreasing microstructural homogeneity. Unfortunately, few studies have looked into the effects of SiC particles with a high volume % on the physical and mechanical properties of Cu/SiC composites made by powder method. Therefore, this study was devoted to fabricate Cu with different volume fractions of SiC ranged from 0 to 30 SiC (vol. %) and investigate their effects on the microstructure, dislocation density and mechanical properties.

2. Materials and Methods:

2.1. Materials:

A high-purity copper metal with an average particle size of 10 μ m, was used as the matrix, as well as SiC having 99.8 purity and average particle size of 3 μ m, was used as reinforcement material to produce Cu/SiC microcomposites. The powder mixtures were processed by a mixer with 10 cm diameter and speed of 950 rpm for 3 hours and Cu/SiC composites were fabricated with varied SiC particle volume fractions (0 to 30 vol%). The powder mixtures with different volume fractions were compacted in a steel die with a 12 mm diameter at constant load of 400 MPa. SEM micrograph of copper and SiC particles and its EDS analysis were illustrated in Fig 1 (a & b), respectively. After that the cold-pressed specimens were sintered at 850°C (80% of the melting point of Cu) in the argon gas for 2 hours by electrical tube furnace with constant heating and cooling rate of 5 C/min.

2.2.Density determination

According to ASTM B328 Standard, The density and relative density of Cu/SiC composites were determined using Archimedes' principle. The theoretical density of the composites was calculated using a mixture rule as follows:

$$\mathbf{d}_{\mathrm{C}} = \mathbf{d}_{\mathrm{m}} \mathbf{V}_{\mathrm{m}} + \boldsymbol{\rho}_{\mathrm{r}} \mathbf{V}_{\mathrm{r}} \tag{1}$$

where dm, dr and Vm, Vr are the matrix and reinforcement densities and volume fractions, respectively. Also, the ratio of experimental and theoretical densities was used to calculate the relative density.



Figure 1. SEM Micrographs And EDX Analysis of (a) Copper (b) SiC particles.

The schematic diagram of Cu/SiC microcomposites manufactured by powder metallurgy technique is shown in Fig. 2.



Figure 2. Schematic Illustration Of Cu/SiC Microcomposites Produced By Powder Metallurgy Process.

2.3. Microstructural evaluation

The microstructure, crystallite size, shape and distribution of SiC reinforcement were observed by JEOL (model JSM). No – (6330F) microscope at a voltage of 20 keV. All samples were prepared and grounded using 360, 600,1000 and 1200 grit -silicon papers. The specimens were mechanically polished with 6,3 and 1um diamond paste after grinding, then etched in a solution of 5g of ((FeC1₃+25 mL HC1+50 mL H₂O)) to improve contrast. The existing of SiC particles and its dispersion on the copper matrix were seen at different magnifications on the specimens.

The distinct phases and crystallite sizes of Cu and Cu/SiC microcomposite were calculated using Xray diffraction analysis on (Philips Machine). A monochromatic Cu-K radiation with wavelength ((λ = 0.154 nm)) has been used. The experiment was ran from angle of 5° to angle of 90°, at 0.01° step.

2.4. Hardness test

Hardness of Cu/SiC composites was conducted on the polished surfaces without etching using digital metallic Vicker's hardness tester. The Vicker's test was performed on the surfaces of specimen with a 10 kg load applied for 15 seconds. The Vickers hardness values were shown as color-coded contour maps to demonstrate the hardness differences over the sample surface. A hardness indentations were made on each surface of specimen with a 0.5 mm distance between each individual point

2.5. Compression test

Compression specimens were cut from the unrienforced Cu and Cu/SiC composites using a wire cutting machine. Compression testing was performed on 12mm diameter and 10 mm height. The test was performed at an initial strain rate of 0.5mm/min, using a testing machine (com-ten with program software 2.1.25) .The compressive property data are based on the average of three individual tests on the three different samples. The tests were performed on the full disc specimen. The maximum failure load was determined by compressing the sample between two flat platens.

3. Results and discussions:

3.1.Density measurements

Fig. 3. illustrates the effect of SiC vol% on the relative ,theoretical and experimental density of Cu/SiC microcomposites. It is clear that increasing SiC particles declines Cu/SiC microcomposites density. This result is similar to the results of Prosviryakov [13]. Further, Fig.. 3. Indicates that the relative density reduced from 91.3 (pure Cu) to 83.56 % with increasing SiC particles to 30 (vol %). The high reduction in the relative density is attributed to the size and content of SiC according to the conclusion of **Efe** et al [10]. The density of SiC particles (3.2 g/cm³) is substantially less than that of Cu (8.9 g/cm³), which explains this reduction in the density. The Cu/SiC interface is high in composites with a high SiC content, resulting in a high copper atom diffusion barrier. The copper atoms were thus unable to easily disperse and seal the gaps between the SiC particles. Thus, compaction process is hindered [7].

3.2. Microstructure evaluation

Fig. 4 shows SEM images of Cu & Cu/SiC microcomposites sintered at 860°C with various SiC volume fractions. These microstructure images reveal the existence of SiC in the composites, as well as the even dispersal of SiC micro particles within the matrix. Also, when the content of SiC particles is added to 20%, the SiC particles move and concentrate at the grain boundaries, forming a homogenous network and uniformly dispersed inside the matrix as well. Like this behavior was obtained by Efe et.al [10]. Furthermore, Fig. 4 proves that as the SiC content augments, the size of grains for Cu- matrix decreased . The pinning effect of reinforcement, which restricts grain growth in the matrix, causes a decrease in Cu grains. However, at greater volume fractions, non-uniform SiC reinforcement particle distribution and SiC clusters were clearly visible (Fig 4 (f, g)). As the SiC particle volume fraction increased to 25 and 30 vol.%, the clustering became more sharp as shown in (Fig. 4 (f,g)). It's worth noting that the removal of the reinforcing particles during sample preparation contributed to the formation of pores inside the necklace structure of SiC particles (Fig. 4(f, g)).



Figure 3. Shows The Effect of SiC % on The Relative ,Theoretical And Experimental Density of Cu/SiC Microcomposites.

Distribution maps of the Cu, Si and C elements in a SEM image of Cu/20 vol% SiC microcomposite obtained by EDS analysis and observed in the Fig. 5. As shown in the image with blue color, Cu may be seen covering practically the whole surface. Images with green and red color clearly showed Si and C elements are less than Cu element and that indicates existence of dispersed SiC in the Cu matrix. In addition, EDS analysis map reveals a uniform dispersion of SiC within Cu/20vol%SiC microcomposite.



Figure 4(a-g) SEM Photographs Of Cu And SiC/Cu Microcomposites With Different SiC Contents (0,5,10,15,20,25,30%) Sintered At 860°C.



Figure 5. EDS Elemental Mapping Of The Of Cu/20%SiC Microcomposites a)The Corresponding Elemental Mapping Of Si,C And Cu Respectively. b)EDS Spectrum Of Cu/20%SiC.

3.3. XRD analysis

Fig. 6 shows the XRD spectrum of a Cu/SiC microcomposite with various of volume fraction SiC particles. Due to the JCPDS XRD files, the reflection planes of (111), (200) and (220) at 20 of 43.29, 50.43, 74.12 respectively, assuring the formation of Cu face center cubic. The reflection planes of (100), (002), (101), (102), (110) at 20 of 33.58, 35.50, 38.13 and 49.67 60.05 respectively, confirming the formation of hexagonal SiC particles. In addition, XRD analysis revealed that increasing SiC particles caused reduction in intensity of Cu peaks. According to Akbarpour et al. [12], the reduction in intensity is due to a differential in thermal expansion between the Cu matrix and the SiC particles, resulting in the creation of lattice micro-strain in the matrix. Also the behavior can be explained by the matrix's decreasing grain size.



Figure 6. XRD Pattern Of The Cu/SiC Composite Reinforced With Different Volume Fraction Of SiC Particles

Furthermore, the crystallite size (d) and the lattice strain (η) of α -Cu material can be determined according to Williamson-Hall formula [12,14]:

$$\beta \text{Cos}\theta = \frac{k\lambda}{d} + 4\eta \text{Sin}\theta \tag{2}$$

Where k characterizes the Scherrer constant ((k= 0.9- 1.0)), λ refers to X-ray wavelength and η is the average of lattice strain, β represents the full width at half maximum of the central peaks in radian, θ is Bragg angle. Plotting β cos θ versus sin θ for ((α -Cu)) as shown in fig. 7. Gives a straight line with

slope of (2η) and intercept of $(k\lambda/d)$.

The data which were used in Equation (2) obtained from Fig. 6. The crystallite size (d) and the lattice strain (η) were plotted in the fig. 7. It was discovered that the outcomes had been reversed. The crystallite size of α -Cu matrix is decreased by ~ 36, 62, 66 and 72 % for 5%,10%,15 and 20% vol SiC,

respectively comparing with the pure Cu sample. Increasing SiC volume fraction to 25 and 30% vol, the average lattice strain and the crystallite size increased but still lower than pure Cu. this behavior could be attributed to nonuniform dispersion of SiC within the matrix.



Figure 7. Effect Of SiC Volume Fractions On The Crystallite Size And Lattice Strain

3.4. Mechanical properties

3.4.1. Hardness

Hardness is an important mechanical characteristic for understanding composites' overall mechanical behaviour. The hardness of composites can be evaluated by several parameters, such as particles volume fraction, size of particle, distribution, density of reinforcement, and production method [15]. Fig. 8 (a-g) represents the color- contour maps of the hardness on the Cu pure and Cu/SiC composite with different mass fraction of SiC particles. Variation in hardness magnitudes observed in all samples compared with pure Cu sample. The variations in hardness seem to be very low when the SiC mass fraction was 0%. Hardness difference between the maximum value and minimum value was 67,149,151,154,156,141HVin the case of the Cu, Cu/5 vol%SiC, Cu/10 vol%SiC, Cu-15 vol%SiC, 20 vol%SiC, Cu-25 vol%SiC and Cu-30 vol%SiC respectively. The variations in hardness seem to be more significant when the SiC mass fraction was between 25 to 30%. Also, fig 9(a-g) shown that, the hardness value decreased from center to outside of sample. The variation in the value of hardness is due to the variation in the SiC particles distribution in the matrix the difference in internal strain from the sample's core to the outer surface [16,17]. Fig. 9 presented the results of the hardness for the investigated samples. The outcomes revealed that the microcomposites hardness values were higher than the pure Cu specimen. The values became higher as the SiC volume fraction increased, and the hardness increased as well, reaching 231 HV for the composite sample with 20% SiC particles. The hardness was increased from 75% for Cu 20% SiC composite compared with unrienforced Cu. This increase in hardness was caused by the existence of hard SiC particles in the Cu matrix. In comparison to pure Cu, it has been hypothesized that the addition of reinforcements in Cu composites may increase grain refinement even more [18,19]. On the other hand, the hardness results revealed significant decrease with the addition of 25 and 30%-SiC

particles. The average hardness values of microcomposite which contain 25 and 30% SiC (217and 181HV) as shown in Fig. 9 were obviously lower than that of the Cu/20%SiC (231 HV), indicating an inhomogeneous distribution of SiC reinforcement with the Cu matrix. Further, Fig. 4 shows the non-uniform arrangement of reinforcement particles and clusters of SiCp at higher volume fraction.



Figure 8. Show The Contour Map For Hardness Values Of Cu/SiC Composites With Different SiC Contents (0,5,10,15,20,25,30%) Resulted From Hardness Testing With Indentation Distance Of 1mm.

3.4.2. Compressive strength

Fig. 10 illustrated the compression stress-strain graphs for unrienforced Cu and Cu/SiC microcomposites with various volume fractions of SiC reinforcement. Whereas ultimate compressive strength and compressive yield strength which extracted from fig 10 were showed in Fig 11. The results revaled that, the microcomposites had greater yield and ultimate strength than the unreinforced Cu, as shown in Figs 10 & 11. The ultimate compressive and yield compressive strength were increased from ~26.1% for unreinforced Cu to ~57% for the Cu /20 vol% SiC microcomposite, respectively. as demonstrated in Fig. 11, increasing the SiC reinforcement inside the Cu matrix to 25 and 30 percent did not result in substantial gains in yield and ultimate composite strength when

compared to the Cu/20vol % SiC composite. The decrease in yield and ultimate compressive strength of the Cu reinforced with 25 and 30 % SiC composites comparing to 20vol%SiC can be attributed to the presence of certain clusters and nonuniform homogeneity of SiC particles inside the Cu matrix, as indicated in microstructure. This behavior caused a weak interfacial bonding. It is known that, the interface between the hard SiC reinforcement and the Cu matrix plays an important role in the mechanical properties of composites [20,21].



Figure 9 Hardness Of Cu / SiC Microcomposite With Various SiC Content.



Figure 10. Compressive Stress-Strain Curves For The Cu/SiC Microcomposites.

3.4.3. Dislocation density measurements

The following equation was used to determine the effect of SiC volume fraction on the dislocation density (ρ) of α -Cu using an XRD pattern. [22].

$$\rho = \frac{7.75 \, x \, \eta}{b \, x \, t} \tag{3}$$

Where (η) characterizes lattice strain, (b = 0.154nm) represents burger vector of Cu and (t) signifies crystallize size.

The obtained results can be presented in Fig. 12. It is obvious that increasing SiC content to 20 (vol. %) increases the dislocation density according to increasing the lattice strain. When the tungsten was 15 vol%, Arsenault and Shi [64] found that the lowest dislocation density within the matrix was $7 \times 10^{11} \text{ (m}^{-2})$, while at the Cu-W interface, the dislocation density was $4 \times 10^{12} \text{ (m}^{-2})$. It was concluded that the variation in coefficient of thermal expansion (about 4:1) between copper and tungsten was shown to be the cause of the increasing dislocations. In our case the difference in the coefficient of thermal expansion between copper and SiC is 8:1 more than twice as great as in the Cu-W system. On the other hand, Fig. 12 reveals that after 20 %SiC, the dislocation density decreased, which can be related to the existence of SiC reinforcement agglomeration in the composites with 25 and 30% SiC, as demonstrated in the microstructure images previously. The dislocation density increased from $12 \times 10^{14} \text{ (m}^{-2})$ for pure copper to $37 \times 10^{14} \text{ (m}^{-2})$ by increasing SiC content to 20 (vol. %). However, the results observed that the dislocation density reduced to 32×10^{14} and $30 \times 1014 \text{ (m}^{-2})$ when SiC content increased to 25 and 30 (vol. %) respectively.



Figure 11. Effect Of The SiC Content On The Yield And Ultimate Compressive Strength Of Cu/SiC Micrcomposites.

Cu/SiC micrcomposites have improved in hardness, compressive yield, and ultimate strength for

many reasons. The first reason may be related to the Apparent strengthening efficiency (Ra). Ra refers to the ratio of yield strength increase of the composite to that of the matrix. The following equation can be used to calculate apparent strengthening efficiency [23].

$$Ra = \frac{\sigma c - \sigma m}{V f \sigma m}$$
(4)

Where $((\sigma m))$ refer to the matrix yield; considering the difficulty to obtain an accurate value of the reference and ((Vf)) is the volume fraction of the reinforcement particles, $((\sigma c))$ represents the composite yield strength,. Ra can characterizes the composite overall strength enhancement as a result of the reinforcement. According to the previous equation, the apparent strengthening efficiency (Ra) increases as the SiC volume fraction increases, accordingly, all mechanical properties will improve. The second reason is rule of mixture. As a simplistic model, the rule of mixture can only be apply roughly to estimate the mechanical characteristics of composites, and it is frequently inconsistent with experimental data. The Rule of mixture [24], can be expressed as follows:

$$\sigma \mathbf{c} = \mathbf{V}\boldsymbol{m}\boldsymbol{\sigma}\boldsymbol{m} + \mathbf{V}\boldsymbol{f}\boldsymbol{\sigma}\boldsymbol{f} \tag{5}$$

According to the rule of mixture, with increasing the SiC volume fraction the strength will be increase.

The third reason is the transfer of load from Cu matrix to SiC reinforcement and interface bonding between matrix and SiC particles which are important to the strengthening of Cu/SiC micrcomposites. When Cu matrix composite was loaded, the load was transferred from the Cu matrix to the SiC reinforcement particle, which increased the composite samples' resistance to plastic deformation due to the variation in thermal mismatch values of the matrix and reinforcement particles. This thermal mismatch caused increasing in dislocation density into the Cu matrix [25]. A higher dislocation density in the composite resulted in a higher degree of internal stress, which improved all of the composites mechanical properties.



Figure 12. Effect of SiC Content on The Dislocation Density of Cu/SiC Micrcomposites.

4.Conclusion

In the current research, Cu matrix reinforced with different volume fraction of micro-SiC particles was fabricated. The crystallite size of copper matrix was decreased with increment of SiC contents to 20 (vol %) beyond this value, it was increased but still lower than the base matrix. SEM results showed a uniform distribution of SiC particles to 20 vol.%SiC .The dislocation density increased from 12×10^{14} (m⁻²) for pure copper to 37×10^{14} (m⁻²) by increasing SiC content to 20 (vol. %). However, the results showed that the dislocation density decreased to 32×10^{14} and 30×10^{14} (m⁻²) when SiC content increased to 25 and 30 (vol. %) respectively. Further, sample of 20 (vol. %) SiC possesses the highest hardness, ultimate and yield compressive strength than that of the other six composites. The values of hardness, Ultimate and yield compressive strength increased to 231 HV,343 and 176 N/mm², respectively for the composite sample containing 20% SiC particles with increasing percentage of 75%,26.6% and 57.2% respectively, compared with pure Cu.

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