

Microstructure and hardness of the Cu-SiC and Cu-diamond composites produced by vacuum hot pressing

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Abstract. In the present investigation, Cu-SiC and Cu-diamond composites with different volume percent of reinforcements were produced by vacuum hot pressing. Copper powder with different amount of SiC and diamond were hot pressed at 1000°C for 0.5 h at an applied pressure of 32 MPa. The achieved sintered density of the composites was in the range of 94-98 %. The sintered density decreased with an increase in the amount of SiC and diamond. Hardness of the composites improved with an increase in the volume percent of the reinforcements. Hardness of the Cu-30 vol.% diamond and Cu-30vol. % SiC composites was 88 VHN and 104 VHN, respectively.

1. Introduction

Copper is one of the metals used for thermal management in electronic packages because of its high thermal conductivity ($400 \text{ Wm}^{-1}\text{K}^{-1}$) [1,2]. However, the coefficient of thermal expansion (CTE) is about 3-4 times higher than most of the semiconductors which leads to premature failure of the packages [1,3]. This problem of mismatch in CTE was solved by the addition of reinforcements with high thermal conductivity and low CTE [3]. SiC, graphene, carbon nanotubes and diamond were used as reinforcements to encounter the problem [1,4–6]. The thermal conductivity of SiC and diamond was 280-400 and 1000-2200 $\text{Wm}^{-1}\text{K}^{-1}$, respectively. At the same time, they possess low CTE compared to copper with a value of 4.8 (SiC) and 1.1 (diamond) ppm K^{-1} [3]. Difficulty with the processing of copper based composites with SiC and diamond as reinforcements is reaction of SiC with copper at elevated temperature [1] and poor wetting of diamond with copper [7], respectively. Wetting of the diamond in copper was improved by coating of diamond particles [1,8] and alloying of copper with strong carbide formers [9–11]. The methods employed for the fabrication of copper based composites were friction stir processing [12], spark plasma sintering [10], infiltration [11], shock wave consolidation [7] and hot pressing [9]. In the present study, an effort has been made to produce copper based composites with different volume percent of silicon carbide and diamond by vacuum hot pressing.

2. Experimental procedure

Copper, silicon carbide and diamond powders used in the synthesis of composites were characterized for shape and size by scanning electron microscopy. Diamond powders were coated with copper by



electroless coating to modify the interface for better wetting with copper. Mixing of the copper and reinforcements (SiC and diamond) powders in different proportions was done in rolling mill using wax as a binder. The mixed powders were consolidated in a 100 T vacuum hot press using graphite die-punch assembly. Trials were conducted in the temperature range of 900-1000°C at a pressure of 32 MPa for holding time of 0.5 h to optimize the cycle. The optimized hot pressing cycle employed was 1000°C with holding time and applied pressure of 0.5 h and 32 MPa, respectively. Vacuum level of about 1×10^{-4} mbar was maintained during the entire hot pressing cycle. The mixed powders were filled in a three inch cylindrical graphite die and subjected to hot pressing. After the hot pressing cycle, the compacts were allowed to cool in the furnace. The hot pressed compacts were sand blasted and all the surfaces were machined to remove 1 mm layer. The density of the composites was measured by using a method based on Archimedes' principle and compared with the theoretical density. Samples for microscopy were prepared by mechanical polishing using emery papers, alumina and diamond paste. The polished samples were etched with a solution of 10 gm potassium dichromate, 5 ml sulphuric acid and 80 ml water, and examined under an optical and scanning electron microscope (SEM). Hardness of the samples was measured using a Vickers hardness tester at a load and dwell time of 5 N and 10 s, respectively.

3. Results and Discussion

3.1.

Raw materials

Atomized spherical copper powder and irregular shaped diamond and SiC powders were used in the study. Fig. 1 shows the SEM images of the copper, SiC and diamond powder. The average size of the copper, silicon carbide and diamond powders was 40 μm , 60 μm and 20 μm , respectively (Fig.1a-1c). Copper coated diamond powders are shown in Fig. 1d.

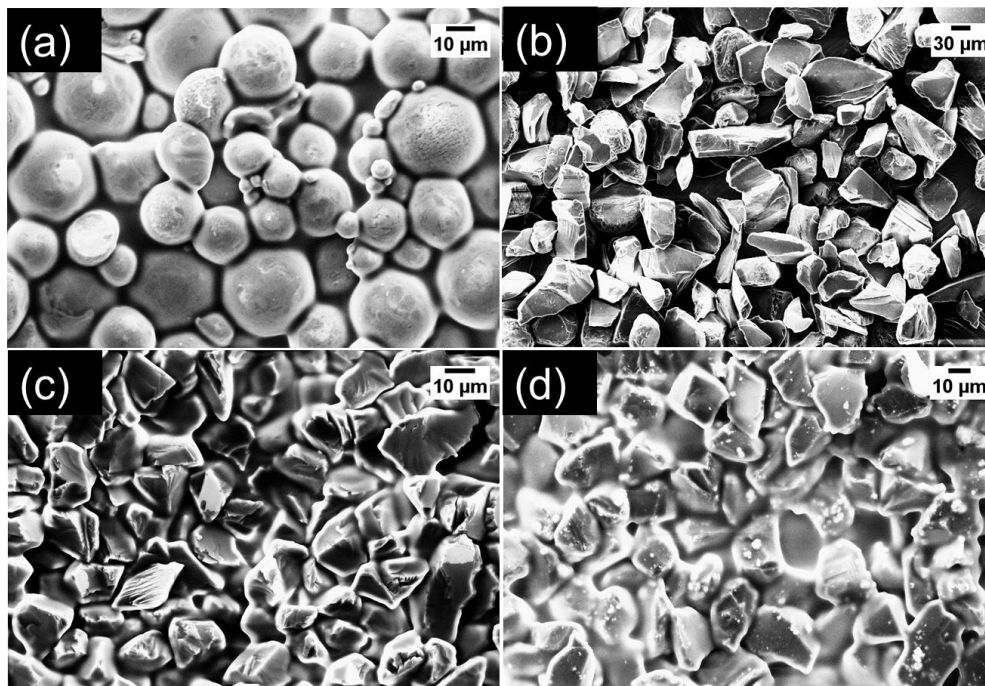


Figure 1. SEM image of the powders: (a) copper, (b) silicon carbide, (c) diamond and (d) copper coated diamond

3.2. Optical microscopy

3.2.1. Cu-SiC

The distribution of the reinforcement and porosity in Cu-SiC composites is shown in Fig. 2. SiC particles were seen as a dark feature in the grey matrix. Composites with 10 and 20 volume percent of SiC were free from porosity as shown in Fig. 2a-2b. In addition, distribution of SiC in copper matrix was uniform. At 30vol.%SiC, necklace structure was prevalent with all the grain boundaries decorated with the SiC as shown in Fig. 2c. A small amount of porosity was observed in the Cu-30%SiC composite.

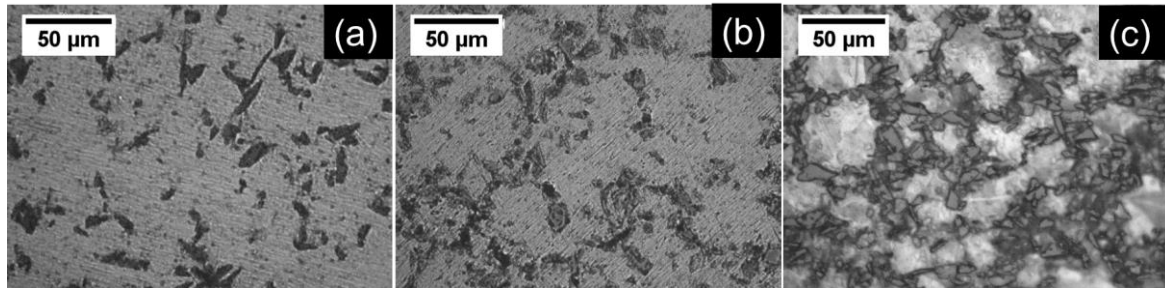


Figure 2. Optical micrographs of the Cu-SiC composites: (a) Cu-10vol.%SiC, (b) Cu-20vol.%SiC, and (c) Cu-30vol.%SiC

3.2.2. Cu-diamond

Fig. 3 shows the optical micrographs of the Cu-diamond composites with different volume percent of reinforcement. Diamond particles are seen as dark particles in the grey copper matrix. The distribution of the diamond particles was uniform in Cu-10vol.% diamond (Fig. 3a) and Cu-20vol.% diamond (Fig. 3b) composites. At higher volume percent (30%) agglomeration was observed as shown in Fig. 3c. It was rather difficult to distinguish between diamond particles, porosity and pull-out as all of them appear dark in bright field. To distinguish them, optical micrographs were recorded in both bright field and dark field mode at higher magnification as shown in Fig. 4a and Fig. 4b, respectively. In the dark field, diamond particles appear bright in the dark copper matrix. Whereas, pull-out can be identified by the depth as indicated by the black arrow and porosity is indicated by white arrow (Fig. 4b).

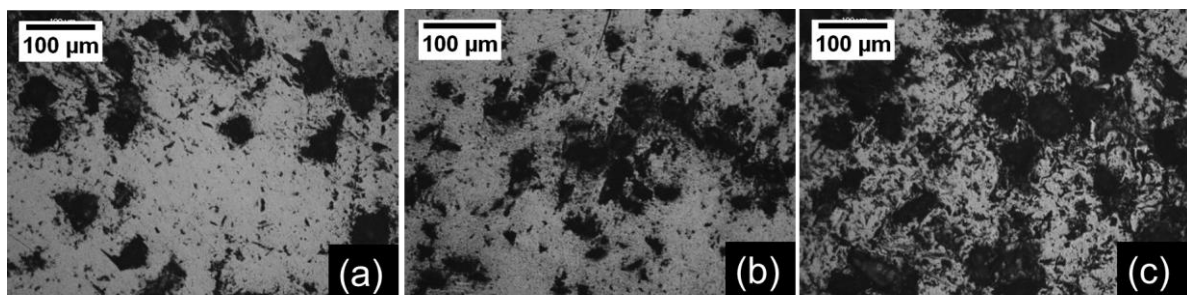


Figure 3. Optical micrographs of the Cu-diamond composites: (a) Cu-10vol.% diamond, (b) Cu-20vol.% diamond, and (c) Cu-30vol.% diamond

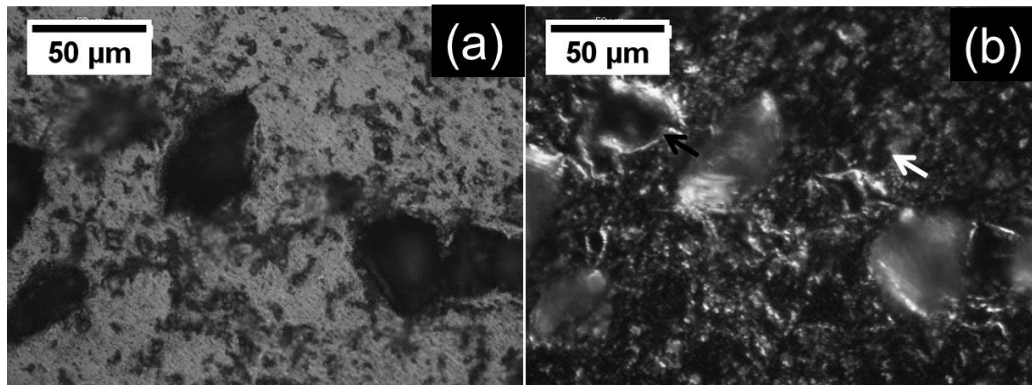


Figure 4. Optical micrographs of Cu-10vol.% diamond at higher magnification: (a) bright field (bf) and (b) dark field (df)

3.3. Scanning Electron Microscopy (SEM)

Fig. 5 shows the SEM micrographs of the Cu-10vol.% SiC and Cu-10vol.% diamond composites. The distribution of the SiC and diamond particles was uniform as shown in Fig. 5a and Fig. 5c, respectively. The interface between SiC and copper was good without any porosity (Fig. 5b). In the case of Cu-diamond, porosity was observed at the interface as indicated by the arrows in Fig. 5d.

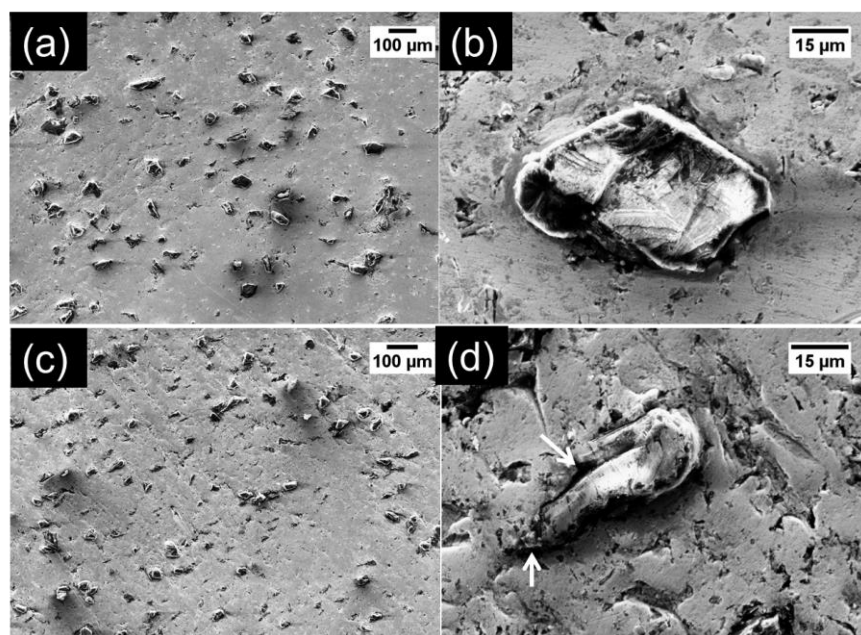


Figure 5. SEM micrographs of the Cu-10vol.% SiC and Cu-10vol.% diamond composites: (a) Cu-10vol.% SiC (lower magnification), (b) Cu-10vol.% SiC (higher magnification), (c) Cu-10vol.% diamond (lower magnification) and (d) Cu-10vol.% diamond (higher magnification)

3.4. Sintered density

Temperature, pressure and time are the main parameters that dictate the densification behavior of the powders during hot pressing [13]. Densification rate depends on the temperature, time and pressure. However, temperature shows profound effect on the densification rate and increase in temperature leads to faster densification rate. Copper was hot pressed at temperature more than 0.6 times of melting temperature where the creep mechanisms become active. Dislocation creep dominates the deformation process at a lower temperature and grain boundary sliding takes over at high temperature. In the present case, reinforcements (SiC and diamond) are very hard and do not deform even at the

temperature as high as the melting point of copper [3]. Yield strength of pure copper at 700°C is about 20 MPa and it is expected to further decrease with the temperature [14]. Therefore, hot pressing temperature of 1000°C and applied pressure of 32 MPa was selected for the hot pressing experiments. Fig. 6 shows the sintered density of Cu-SiC and Cu-diamond composites with different volume percent of reinforcements. Sintered density of pure copper was measured to be about 98%. The 2% porosity observed may be attributed to the presence of oxide on the powder surface and trace impurities which retard the diffusion during hot pressing. Sintered density of the composites decreased with an increase in the volume percent of the SiC and diamond. This reduction in the sintered densities compared to the copper could be associated with adverse effects of the reinforcements on the sintering. The possible mechanism could be entrapment of fine porosity at the interface between the matrix and reinforcement which prevents the densification [13]. In addition, the difference in the melting point and density of the copper and reinforcements (SiC and diamond) could be responsible for the lower sintered density [6,15]. The sintered density of Cu-SiC composites reduced from 96% to 94% with an increase in the volume percent of SiC from 10 to 30. Cu-diamond composites showed sintered density in the range of 98-96% for volume percent of 0-30.

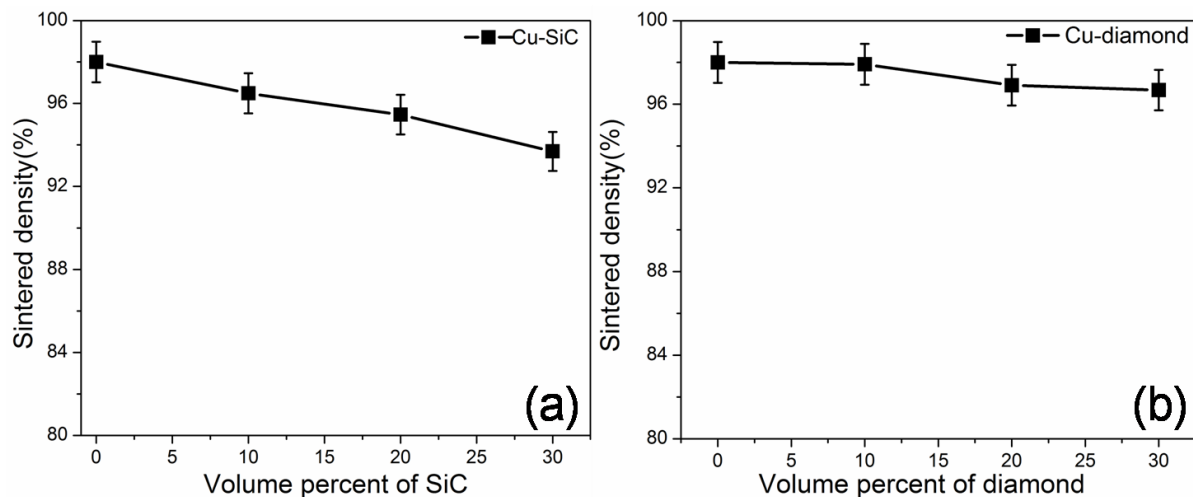


Figure 6. Sintered density of copper based composites with different volume percent of reinforcements: (a) SiC and (b) diamond

3.5. Hardness

The effect of reinforcement content on the hardness of the composites is shown in Fig. 7. The hardness of Cu-SiC composites was in the range of 47 to 105 VHN. Hardness was found to increase with the amount of SiC and maximum hardness of 105 VHN was observed for Cu-30vol.%SiC. Cu-diamond composites showed hardness in the range of 47-88 VHN. Maximum hardness of 88 VHN was observed in the Cu-30vol.% diamond composite. The difference in the hardness of Cu-30vol. % SiC and Cu-30vol. % diamond is due to variation in the powder size, distribution and interface as shown in Fig. 3-6. An increase in the hardness of composites is due to dispersion strengthening by diamond and SiC particles. Reinforcement particles added to the matrix will impede the plastic flow and result in higher hardness than pure metal. With an increase in SiC and diamond, the obstacles to the plastic flow increased which led to improvement in the hardness [6]. On the other hand, porosity has adverse effect on the hardness of the composites as reported in the literature [15]. A similar trend for hardness was reported in copper based composites with an increase in the reinforcements at lower volume percent [6,13,15].

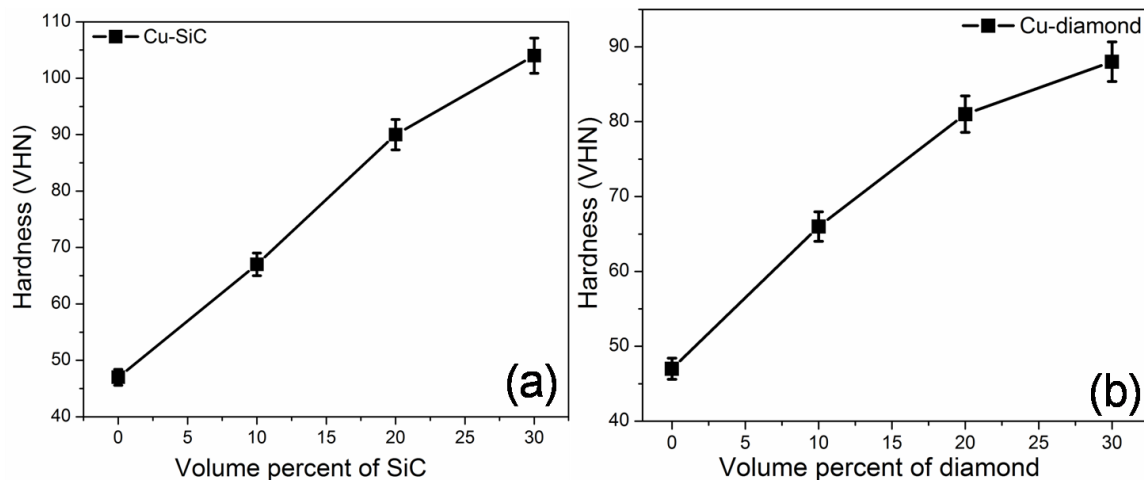


Figure 7. Influence of SiC and diamond content on the hardness of the composites: (a) Cu-SiC and (b) Cu-diamond

4. Conclusions

Dense composites of Cu-SiC and Cu-diamond were successfully prepared by vacuum hot pressing. All the composites synthesized showed high sintered density (98-94%). Sintered density showed a decreasing trend with an increase in the volume percent of the SiC and diamond. Hardness of the composites increased with the volume percent of SiC and diamond. Hardness of 104 and 88 VHN was achieved in Cu-30vol.% SiC and Cu-30vol.% diamond composites, respectively.

5. References

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