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Microstructural, electrical, thermal and tribological studies of copper-fly ash composites through powder metallurgy

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Abstract. This research work address the fabrication of copper (Cu) matrix composites, reinforced with fly ash (FA) particulates with 3, 6, 9 and 12 wt.% using the powder metallurgy route. The microstructural, physical, electrical, thermal, mechanical and tribological properties of thus fabricated Cu-FA composites have been studied. Optical microstructural characterization of the composites exposed persuasively uniform distribution of FA reinforcement with minimum porosity. The mixed powder SEM images revealed the homogeneous dispersion of fly ash particulates in the copper matrix. The hardness values showed improvement with increase in the weight percentage of FA in the Cu matrix. Electrical conductivity was measured using the four-point probe method at room temperature. Thermal conductivity was measured with a thermal diffusivity analyzer at room temperature. The fly ash addition leads to weakening the conductivity of Cu-FA composites. The tribological properties of Cu-FA composite specimens were investigated using a Pin-on-disc tribo testing machine against an EN81 steel contour disc. The specific wear rate of the composites tended first to decrease, which was attributed mainly to the formation of a mechanically mixed layer on the worn surface. Then it would increase as the FA content increased because of reduction in ductility and brittle oxide cracks associated with adding more FA particulates. It seems that composites with FA percentages below 9wt.% have optimum properties of microstructure, hardness and wear resistance, which is suitable for applications such as electrical sliding contacts, electrical discharge machining and spot welding electrodes.

Key words: copper composites, fly ash, electrical and thermal conductivity, wear test.

1. Introduction

Copper possesses several desirable properties for usage in electrical and thermal industries. Those include high electrical conductivity, thermal conductivity, good formability, high melting point and good corrosion resistance [1, 2]. Cu has found numerous applications such as material for nozzles of gas turbines, rocket engines, liners of combustion chamber walls, contact breakers, rotating neutron targets etc., [2, 3]. Nevertheless, its low hardness, poor wear resistance together with poor arcing resistance cause massive concerns in the case of electrical contact applications where sliding wear occurs. Wear resistance can be improved by either age hardening mechanisms or reinforcing copper with hard ceramic particles [4, 5].

Recent experience has shown that the mechanical response of copper metal matrix composites can further be improved if submicron- and Nano-sized particles are used as the reinforcing phase. Copper based metal matrix composites have high prospective properties such as improved antifriction and wear resistance characteristics [6]. Copper metal matrix composites (CMMCs) play a vital role in electrical and thermal properties, particularly in the field of transportation applications such as those for aerospace, marine industry, etc., [7]. In addition,

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CMMCs have high potential properties such as enhanced antifriction and wear resistance characteristics [8].

Many researchers have investigated the electrical, mechanical and tribological properties of copper composites which were reinforced with hard ceramics for the achievement of better properties in a copper matrix [9–13]. So far, many attempts have been made to improve the mechanical and tribological properties of the copper matrix by means of different reinforcements [13–16], i.e. SiC, Al₂O₃, B₄C, TiC, TiO₂, Gr, MoS₂, CNT, etc. The choice of ceramic materials is mainly influenced by their high hardness and good wear resistances, refractory nature as well as relative ease of use [17–20]. Fly ash is abundantly offered as a waste by-product lightweight material from coal-fired thermal power stations, which is used effectively to form hard reinforcement particulates in copper composites. Therefore, FA provides excellent reinforcement particulates for a copper matrix as well [21].

The literature survey showed that CMMCs were produced using various processing methods, including powder metallurgy, stir casting, squeeze casting, in-situ, friction stir processing, coating and laser sintering. However, those CMMCs always manifested a variety of production defects, which were not limited to porosity, aggregation, segregation, inhomogeneous distribution and interfacial reactions.

It is a precondition to distribute the reinforcement particles homogenously into the copper matrix to acquire better properties of CMMCs. Those defects tend to affect the mechanical and tribological properties in an adverse manner [22]. Among

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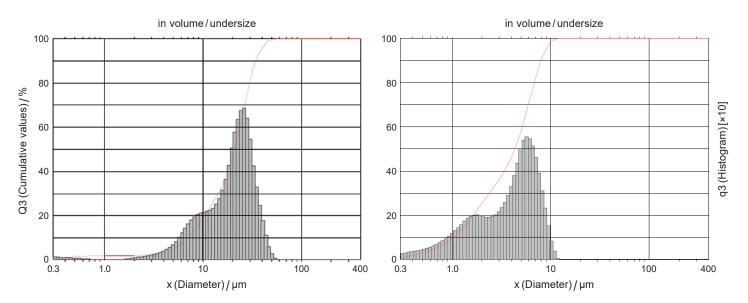


Fig. 1. Particle size distribution of as-received (a) Cu powders and (b) fly ash particulates

the above-mentioned methods, powder metallurgy (PM) is found to be a promising method for producing CMMCs. PM is one of the best approaches for manufacturing CMMCs with a huge difference in density of the elemental matrix and reinforcement materials, which is not conceivable in casting [23]. It is a more economical method to be used on a massive scale, even though the wettability of reinforcement particles is poor.

There are no systematic investigations on the mechanical and tribological properties of FA-reinforced copper composites. In the present investigation, copper composites fabricated with varying wt.% of fly ash as reinforcements through the PM technique are discussed. In addition, the effects of FA content such as the microstructure, density and hardness are studied. Furthermore, electrical, thermal and wear properties are also studied.

2. Materials and methods

In this study, copper powder with average particle size of 20 μm ($<35~\mu m$ and 99.7% purity) was used as the matrix material supplied from M/s. Metal Powder Company (P) Ltd., Tirumangalam, Madurai, Tamilnadu, India. Fly ash having less than 10 μm of particle size and chemical composition of SiO $_2$ – 67.23%, Al $_2$ O $_3$ – 29.64%, Fe $_2$ O $_3$ – 0.47%, CaO – 1.34% and MgO – 1.86%, collected from the Thermal Power Station in Tuticorin, India, was used as the reinforcement material.

The particle sizes of the Cu and FA powders were analyzed using a particle size analyzer and scanning electron microscopy, respectively. Fig. 1a, b shows the size distribution of Cu and FA powder particles. The SEM micrographs of the as-received powder particles is shown in Fig. 2a, b. The Cu particles appear in flake shapes and fly ash particulates take the form of

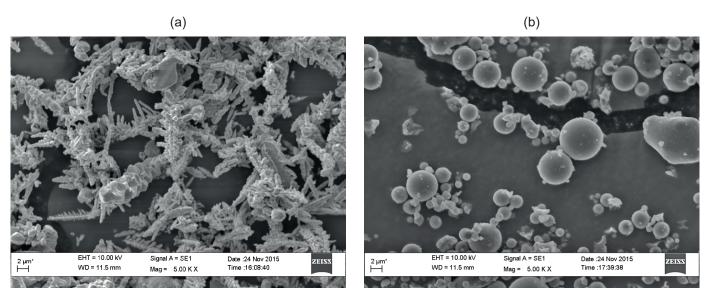


Fig. 2 SEM micrographs of received (a) Cu powders and (b) fly ash particulates

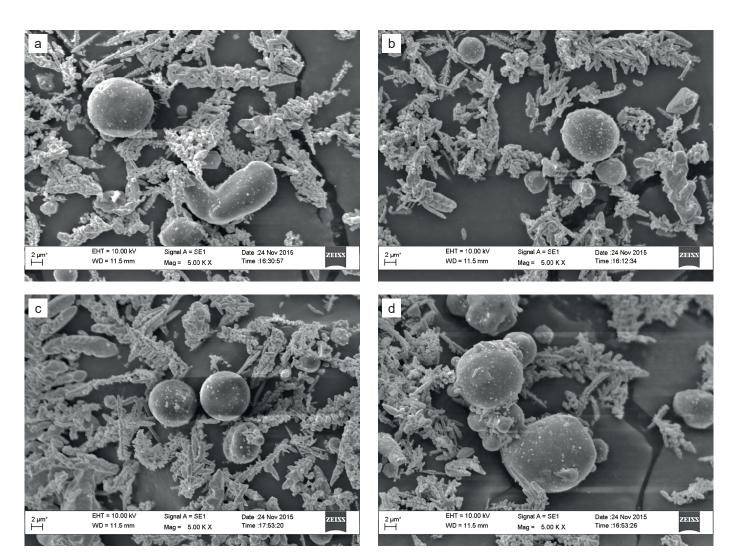


Fig. 3. Typical SEM micrographs of the produced powder mixtures: (a) Cu-3FA (b) Cu-6FA (c) Cu-9FA and (d) Cu-12FA

spheres. By using the rule of mixture, the elemental powders were mixed and the respective SEM was shown in Fig. 3a–d. The SEM micrograph shows that there is no agglomeration of the Cu and FA particulates in the mixture.

The milled powders were consolidated in a powder compaction die under the pressure of 450 MPa at room temperature to form green compacts [23]. Before each run, die wall lubrication was performed manually using graphite. The green compacts were sintered in a muffle furnace at the temperature of 950°C for 60 min, as suggested by Mahani Yusoff et al. [24].

Samples were prepared as per the standard metallographic preparation procedure for an optical microscopy study. The measured density was determined by Archimedes' principle according to ASTM: B962-13. The hardness of fabricated composites was measured using the Rockwell hardness tester, B scale. Electrical conductivity was measured using the four-point probe method [25, 26]. The thermal diffusivity analyzer was used to measure thermal conductivity at room temperature [27–29].

Wear tests were performed using a pin-on-disc tribo tester (Ducom, model No.: ED-201, Bangalore, India) under room conditions [29]. Tests were conducted at a constant normal

load of 15 N, sliding velocity of 1.5 m/s and sliding distance of 1500 m, with the track diameter of 60 mm. The counter disc was made of EN 31 steel with hardness of 64 HRC. Before and after wear testing, the surfaces of the contour steel disc and test samples were grounded and polished by SiC abrasive grit paper and cleaned with acetone in order to remove the traces.

3. Results and discussion

3.1. Microstructure analysis. Figures 4a—e shows the optical micrographs of sintered composites. The micrographs reveal the FA particulates are uniformly distributed in the copper matrix phase. The absence of pores and cracks was observed in the optical micrographs. There was no aggregation or segregation of FA particulates. Likewise, no interfacial reaction products were observed. FA particulates were distributed homogenously in the copper matrix irrespective of the weight fraction of up to 9%. The sintered composite shows minimum porosity and a void between the Cu matrix and FA reinforced particulates in Fig. 4b—e.

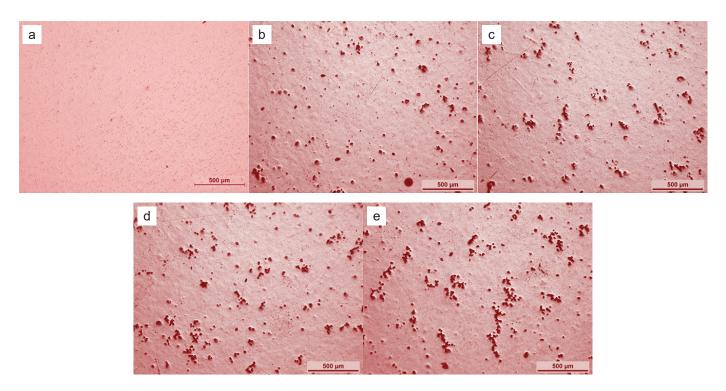
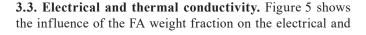


Fig. 4. Optical micrographs of the produced (a) Cu (b) Cu-3FA (c) Cu-6FA (d) Cu-9FA and (e) Cu-12FA composites

3.2. Physical and mechanical properties. Density and hardness values of all the samples are shown in Table 1. It was observed that there is reduction in density with increases in reinforcement particulates of the sintered samples. This can be ascribed to the addition of less significant density of the reinforcements. Moreover, the relative density of sintered composites was dropped with the addition of secondary particles because of material hardening and more dislocations density of the samples being prepared.

Composite hardness increases with an increase of FA content, which is due mainly to the respective hardness of the reinforcement. The increase in hardness with addition of FA to the copper matrix is caused by the decomposition of oxides in FA with the copper matrix of up to 9%. It was found that the Cu-9FA composite is of even higher hardness (94R_B), which may attribute to the formation of a hard phase. Beyond this limit, drop in hardness, caused by particle agglomerations, occurs.



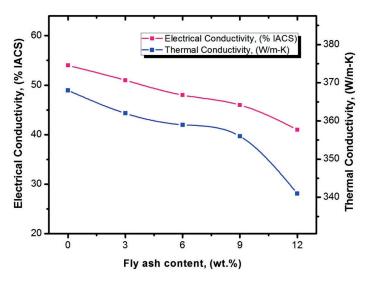


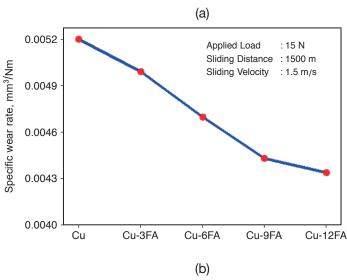
Fig. 5. Electrical conductivity and thermal conductivity of Cu-FA composites

Table 1 Experimental values of densities and micro hardness values

| No. | Material compositions | Theoretical density kg/m ³ | Actual density kg/m ³ | Relative density % | Porosity % | Rockwell hardness RB |
|-----|-----------------------|---------------------------------------|----------------------------------|--------------------|------------|-------------------------|
| 1 | Pure Cu | 8.9640 | 8.2556 | 92.14 | 7.86 | 89 |
| 2 | Cu-3FA | 8.7248 | 8.0302 | 92.04 | 7.96 | 91 |
| 3 | Cu-6FA | 8.4896 | 7.7986 | 91.86 | 8.14 | 92 |
| 4 | Cu-9FA | 8.2544 | 7.5792 | 91.82 | 8.18 | 94 |
| 5 | Cu-12FA | 8.0192 | 7.3488 | 91.64 | 8.36 | 93 |

thermal conductivity of the Cu-FA composites being prepared. It was observed that electrical resistivity increases gradually with increasing FA content. It is clear that with increasing the FA amount, electrical conductivity of composites strongly dropped and had a negative effect. The reduction in conductivity caused by the addition of FA to the Cu matrix is supposed to be the result of the hardness and oxidative nature of FA. Additionally, the decrease in relative density or increase in porosity is yet another phenomenon characterizing high electrical resistance in high FA containing composites [25, 26]. Figure 5 depicts the variation of thermal conductivity of Cu-FA composites with different FA contents. It becomes evident that both EC and TC of composites slightly decreases with an increase in FA up to 9 wt.%. The highest drops in thermal conductivity are obtained when the FA content goes beyond 9 wt.%, which is 341 w/m-K [28, 29].

3.4. Tribological properties. The specific wear rates of Cu-FA composites are shown in Fig. 6a. The high wear rate was noticed



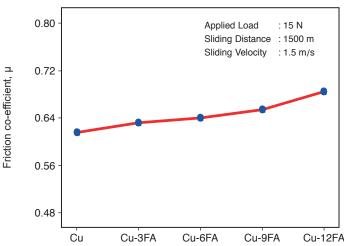


Fig. 6.a) Specific wear rate and (b) friction coefficient of Cu-FA composites

in pure copper, which may be attributable to its high ductile nature. It can be seen from Fig. 6a that the composites SWR instantly decreases, and then increases with the increase of the volume fraction. FA particulates cause hardness improvement in the copper matrix, therefore decreasing its specific wear rate. The composite with 12wt.% FA exhibits the lowest wear rate. For the first rapid decrease stage, instant reduction of the wear rate was attributed to effective enhancement in the hard phase of the MML layer on the worn surface, obtained by adding FA. However, for the second slow decrease stage, damage of the MML layer occurs and it almost no longer increases with further increase of FA content, which may be attributable to its high brittleness.

The friction coefficient values of Cu-FA composites are displayed in Fig. 6b. The incorporation of FA increases the friction coefficient of Cu-FA composites slightly. The higher the content of FA, the higher the friction coefficient of the Cu-FA composite. Nevertheless, the friction coefficients of 3, 6 and 9 wt.% of FA are approximately equal as compared to that of pure copper. The Cu-12FA composite displays a friction higher than for other composites. Furthermore, it can be clearly observed that wear resistance of the copper matrix is improved by adding FA. The decrease in the wear rate of the Cu-FA composite results from the reinforcement effect of secondary particles [30].

4. Conclusion

Cu/X wt.% FA composites (X = 0, 3, 6, 9 and 12) were successfully fabricated using the novel PM method and the influence of FA particulates and their weight fraction on microstructural, physical, electrical, mechanical, thermal and tribological properties were investigated. The results can be summarized as follows:

- The optical microstructure study confirmed that FA particulates are homogeneous and isotropic within the composite.
 No pores or interfacial reaction products were observed.
 In addition, FA particles bonded properly with the copper matrix.
- The hardness of Cu/FA composites increased with an increase in FA content. Among the various composites, Cu-9FA actually has a hardness value higher than that of pure copper.
- However, excessive FA addition will lead to more pores and uneven distribution of the matrix and reinforcement phases, while negative growth will result in a decrease in hardness and the specific wear rate.
- The addition of FA to the copper matrix results in much better mechanical properties and wear resistance while its conductivity dropped strongly. Beyond 9 wt.% of FA content, electrical and thermal conductivities decreased drastically.
- The specific wear rate of the composites decreased instantly with increasing FA content. However, the addition of FA to Cu matrix composites would hardly increase the friction coefficient.

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