Tribological Behavior of Copper–Graphite Composites Reinforced with Cu-Coated or Uncoated SiO₂ Particles

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Abstract: Copper–graphite composites reinforced with SiO₂ particles were fabricated by powder metallurgy technique. Electroless copper plating was introduced to improve the interfacial bonding between SiO₂ particles and copper matrix. The microstructure, density, and hardness of the composites were characterized. The tribological properties, such as friction coefficient and wear rate of the composites, were studied using a pin-on-ring tribometer. The results show that the hard SiO₂ can restrict the severe plastic deformation and adhesion contact in the process of wear. At the same time, parts of SiO₂ particles can be broken into fine particles during wear process, which is helpful for decreasing adhesion wear and abrasive wear. Therefore, the addition of SiO₂ leads to increasing friction stability and friction coefficient, and decreasing wear rate. In addition, the electroless copper plating improves the interfacial bonding between SiO₂ and copper matrix, which prevents separation of SiO₂ from copper matrix and further increase tribological properties of the composites.

Keywords: tribological behavior; copper matrix composites; coating; silica; brake

1. Introduction

Copper matrix composites are wildly used as brake materials of high speed vehicles, such as aircraft and high speed train, owing to its good machinability and excellent thermal conductivity [1–4]. Graphite as solid lubricant often is added into the copper matrix friction materials to stabilize the friction coefficient and reduce wear rate during sliding by forming a graphite-rich transfer layer on the counterpart surface [5–7]. However, the addition of soft graphite leads to the decrease of mechanical properties and friction coefficient of the composites [8–14].

The brake materials require not only good lubricating property but also excellent mechanical properties and high friction coefficient. Ceramic particles—such as Al₂O₃, SiC, SiO₂, and TiC—with high hardness, wear resistance, melting point, and thermal stability can be used as reinforced phase to improve the mechanical and tribological properties of copper matrix composites [15–18]. Among those ceramic particles, SiO₂ exhibits many advantages, such as relatively low prices, near zero thermal expansion, and relatively less abrasive effect on the composites. Previous studies have reported that SiO₂ particle could improve the mechanical properties and wear properties of metal matrix composites [19–24]. However, the weak interface bonding between ceramic particle and metal matrix probably results in pulling out of ceramic particle from the matrix during the brake process and deteriorating the tribological properties. In particular, the detached SiO₂ particle may cause severe abrasive wear. Rohatgi et al. [23] reported that the higher amount of SiO₂ particle deteriorates the tribological properties of Al matrix composites due to the poor interface bonding between SiO₂ particle and Al matrix.
Metallization of ceramic particle is a common method to improve the interfacial bonding between ceramic particle and the metal matrix [25,26]. For example, Lee et al. [27] fabricated Cu/SiC_p composites with Cu-coated or uncoated SiC particles. They found that SiC particles coated with a copper layer can improve the interfacial bonding strength between SiC particles and copper matrix and enhance the mechanical properties of the composites. However, to the best of our knowledge, no tribological properties of copper matrix composites reinforced with coated ceramic particles have been reported and the tribological mechanism is not clear.

In this study, SiO_2 particles were coated with a copper layer by electroless method. Copper–graphite composites reinforced with Cu-coated or uncoated SiO_2 particles were fabricated by the powder metallurgy technique. The tribological properties, the friction and wear mechanism of the composites were studied using a pin-on-ring tribometer at different sliding speed and applied load.

2. Experimental

Electrolytic Cu powder (purity >99.9 wt %, 45 µm in diameter, density of 8.92 g/cm^3), flaky graphite (purity >99.5 wt %, 200–300 µm in diameter), and SiO_2 particles (purity >99.5 wt %, size of 10–20 µm, density of 2.2 g/cm^3) were used for fabricating the composites. The SiO_2 particles were coated with copper up to about 60 mass % using electroless method. The composition of the electroless bath is given in Table 1. The macro and micro morphologies of SiO_2 particles before and after electroless plating are shown in Figure 1. It is observed that after electroless process, a uniform and continuous Cu layer is coated on the surface of SiO_2 particles. At the same time, the color of SiO_2 particles is similar to that of copper.

Table 1. Technology conditions of electroless copper coating.

| CuSO_4·5H_2O | EDTA·2Na | KNaC_4H_8O_6 | HCHO (Solution) | NaOH | Temperature |
|--------------|----------|--------------|-----------------|------|-------------|
| 16 g/L       | 25 g/L   | 20 g/L       | 14 mL/L         | 14 g/L | 40 °C       |

Figure 1. Macro and micro morphologies of SiO_2: (a,c) uncoated; (b,d) Cu-coated.

Copper–graphite–silica composites were fabricated by powder metallurgy technique. A high-frequency mixer (GQM-2-5, Wuxi Jialong Equipment Technology Co., Ltd, Wuxi, China)
with a rotation speed about 400 rpm was used to mix copper powder, flaky graphite and silica particles. The composition of the composites is shown in Table 2. Then these mixtures were cold compacted by plate vulcanizer (XLB, Qingdao Yadong Machinery Group Co., Ltd, Qingdao, China) at a load of 500 MPa to achieve a green specimen. Sintering was performed at 900 °C for 1h under a uniaxial pressure of 2 MPa by using a vacuum-hot-press-sintering furnace (HVRY-I, Shenyang Yite Electrical Apparatus Co., Ltd, Shenyang, China).

The actual density of the composites was tested based on Archimedes’ law. The relative density was calculated by the ratio of actual density to theoretical density. The hardness was measured using Brinell hardness (HBW) by applying a load of 500 N for 15 s. The hardness tests were carried out at least five different locations for each sample and the average value was reported. The microstructures of composites were characterized by scanning electron microscope (SEM, JSM-5500LV, JEOL Ltd., Tokyo, Japan).

| Sample | Copper (mass %) | Flaky Graphite (mass %) | Silica (mass %) |
|--------|----------------|--------------------------|-----------------|
| 0S     | 97             | 3                        | 0               |
| 1S     | 96             | 3                        | 1               |
| 3S     | 94             | 3                        | 3               |
| 1S-Cu  | 95.4           | 3                        | 1 + 0.6 (weight of coating) |
| 3S-Cu  | 92.2           | 3                        | 3 + 1.8 (weight of coating) |

The friction and wear tests were carried out using a pin-on-ring tribometer (FHC-05, Jinan Hengxu Testing Machine Technology Co., Ltd, Jinan, China), as shown in Figure 2. A ring with an outer diameter of 240 mm, inner diameter 160 mm, and thickness 8 mm was made by 30CrMoV with a hardness of 60 ± 2 HRC. The diameter and height of the tested pins is 10 mm and 13 mm, respectively. Before each test, the specimens were finished by emery papers to obtain an average surface roughness of 0.4 μm (Ra), and then cleaned with acetone to remove any surface contaminants. The wear and friction behavior were investigated at load of 0.4, 0.6, 0.8, and 1 MPa, sliding speed of 3, 6, 9, and 12 m/s, and the sliding distance of 12,000 m. The distance between the two pins is 200 mm. The tests were performed under unlubricated conditions at room temperature.

The wear lost was measured according to the change in weight of the pins before and after the test. The wear rate was measured as a function of the wear lost divided by the sliding distance. The coefficient of friction as a function of the sliding distance was calculated and recorded in the computer by sensors. The friction coefficient values were calculated by using the formula $\mu = F/N$, where $\mu$ is the friction coefficient, $F$ is the frictional force in Newtons, and $N$ is the normal force in Newtons.

![Figure 2. Pin-on-ring tribometer illustration: (a) test machine; (b) schematic of pin-disc tribometer.](image-url)
The morphologies of worn surfaces and wear debris were observed by scanning electron microscope (SEM). The chemical composition of worn surfaces was examined by energy dispersive spectroscopy (EDS, EDAX-Falcon, EDAX Inc., Mahwah, NJ, USA).

3. Results

3.1. Microstructure, Density, and Hardness of Composites

Figure 3 shows the SEM morphologies of specimens reinforced with Cu-coated and uncoated SiO$_2$, respectively. Some pores can be observed in the interface between SiO$_2$ particles and copper matrix for specimen reinforced with uncoated SiO$_2$, as shown in Figure 3a,c. However, it can be seen from Figure 3b that there is a good bonding between SiO$_2$ and copper in specimen reinforced with Cu-coated SiO$_2$. The encapsulation of SiO$_2$ with Cu results in good contacts between SiO$_2$ particles and copper matrix during sintering. Therefore, it is effective to improve the interfacial bonding between SiO$_2$ and Cu by electroless copper plating.

![Figure 3. SEM morphologies of the composites: (a,c) uncoated SiO$_2$; (b,d) Cu-coated SiO$_2$.](image)

The density and hardness of the composites are presented in Table 3. It is observed that the relative density decreases with the increase of SiO$_2$ particles for uncoated SiO$_2$ particles reinforced composites, due to the formation of pores. However, the coating of SiO$_2$ particles with Cu before sintering is helpful to increase the relative density of composites. The hardness of the composites increases with the increase of SiO$_2$ content. It means that SiO$_2$ particles play a good dispersion strengthened effect. In addition, the hardness of specimens 1S-Cu and 3S-Cu is higher than that of specimens 1S and 3S, respectively. This should be attributed to the well bonding of SiO$_2$ particles with copper matrix and high relative density in specimens 1S-Cu and 3S-Cu.

| Sample | Relative Density | Brinell Hardness (HBW) |
|--------|------------------|------------------------|
| 0S     | 93.50%           | 39.8 ± 0.5             |
| 1S     | 92.55%           | 40.6 ± 0.9             |
| 3S     | 91.76%           | 41.4 ± 1.1             |
| 1S-Cu  | 93.10%           | 41.2 ± 0.6             |
| 3S-Cu  | 93.03%           | 43.7 ± 1.0             |
3.2. Friction and Wear Characteristics

Table 3. Density and hardness of the composites.

| Sample   | Relative Density | Brinell Hardness (HBW) |
|----------|------------------|-------------------------|
| 0S       | 93.50%           | 39.8 ± 0.5              |
| 1S       | 92.55%           | 40.6 ± 0.9              |
| 3S       | 91.76%           | 41.4 ± 1.1              |
| 1S-Cu    | 93.10%           | 41.2 ± 0.6              |
| 3S-Cu    | 93.03%           | 43.7 ± 1.0              |

Figure 4 shows the typical variation of friction coefficient with sliding distance at applied load of 0.4 MPa and sliding speed of 9 m/s. The wear process can be divided into two stages [28,29]. At the first stage of wear test, the friction coefficient of all specimens increases with the increasing of sliding distance. At this stage, the removing of surface oxide layer leads to the metal-to-metal contact, which causes the increasing of friction coefficient [30]. At the same time, the formation of wear debris also enhances the friction resistance of worn surface [31]. At the second wear stage, a rapid increase of friction coefficient can be observed in specimen 0S. This is because the temperature of worn surface rises with the increase of sliding distance, which results in softening and plastic deformation on the worn surface of specimen 0S. The plastic deformation leads to asperity junctions between the counterparts [32]. However, specimens 1S, 3S, 1S-Cu, and 3S-Cu exhibit a slow increase of friction coefficient value. Further, the friction coefficient of specimens 1S-Cu and 3S-Cu is more stable than that of specimens 1S and 3S, respectively. This should be attributed to the hard particles restricting the plastic deformation of composites by imbedding in the matrix and bearing most of the wearing force [33].
3.3. Worn Surface and Wear Debris

Figure 6 shows the SEM morphologies of the worn surface of specimens at sliding speed of 3 m/s and applied load of 0.4 MPa and 0.8 MPa. The typical chemical composition of the worn surface was shown in Table 4. A large number of plowing grooves, delamination, adhesive pits, and severe plastic deformation can be observed on the worn surface of specimen 0S, as shown in Figure 6a–c. It indicates the wear mechanism of specimen 0S is abrasive wear, delamination, and adhesion wear.

With the content of SiO2 particles increase, the worn surfaces of specimens become smoother. Only plowing grooves, slightly plastic deformation and delamination can be found on the worn surface of specimen 1S, 1S-Cu, 3S, and 3S-Cu, as shown in Figure 6d–o. It indicates that the hard SiO2 particles can restrict the plastic deformation and adhesion wear of composites. Therefore, the wear mechanism of specimen 1S, 1S-Cu, 3S, and 3S-Cu is mainly abrasive wear and delamination wear.

In addition, for composite with SiO2 as reinforcement, much fine SiO2 particles can be found on the worn surface. Especially, with the increase of applied load, the amount of fine SiO2 particles becomes more and more. This indicates that the big SiO2 particles can be broken into fine particles during wear process. Meanwhile, the broken SiO2 can be embedded into the soft copper matrix (see Figure 6d,e,g,h,j,k,m,n). However, the exfoliation of SiO2 particles can be found for the composite with uncoated SiO2 as reinforcement. For example, the separated SiO2 particles can be observed on the worn surface of specimen 1S (see Figure 6e), the holes are noted on the worn surface of specimen 3S (see Figure 6k). Finally, it can be seen from Figure 6(k1,n1) that there is a big gap between SiO2 and copper matrix in specimen 3S. However, no obvious pores can be found between SiO2 and copper matrix in specimen 3S-Cu.
Figure 6. SEM morphologies of the worn surfaces of 0S (a–c), 1S (d–f), 1S-Cu (g–i), 3S (j,k,l), and 3S-Cu (m,n,o) at sliding speed of 3 m/s and applied load of 0.4 MPa (a,d,g,j,m) and 0.8 MPa (b,c,e,f,h,i,k,l,n,o).

Table 4. Chemical composition (at %) of the worn surface.

| Point | Si  | O   | Cu  | C   | Possible Phase |
|-------|-----|-----|-----|-----|----------------|
| A     | -   | 0.8 | 10.3| 88.9| Graphite       |
| B     | -   | 8.1 | 90.7| 1.2 | Cu matrix      |
| C     | 30.3| 63.2| 6.2 | 0.3 | SiO₂           |
| D     | 29.6| 61.5| 8.5 | 0.4 | SiO₂           |
Figure 7 shows the SEM morphologies of wear debris of the composited at applied load of 0.8 MPa and sliding speed of 9 m/s. With the content of SiO$_2$ particles increases, the size of wear debris becomes more and more small. In addition, there are some SiO$_2$ particles in the wear debris of specimens 1S and 3S. This is further evidence that the SiO$_2$ particles without copper plating easily pull out from matrix and removed from worn surface during wear test.

![SEM morphologies of wear debris](image)

**Figure 7.** SEM morphologies of wear debris of the composited at 9 m/s and 0.4 MPa: (a) 0S; (b) 1S; (c) 1S-Cu; (d) 3S; (e) 3S-Cu.

### 4. Discussions

#### 4.1. Effect of SiO$_2$

It is well known that the SiO$_2$ particles possess higher hardness than the Cu matrix. During the wear process, the harder SiO$_2$ particles are exposed to the worn surface, which impedes the relative sliding of counterpart and leads to the increasement of friction coefficient. At the same time, the addition of SiO$_2$ also increases the hardness of composite (Table 3). According to the empirical Archards’ model [34,35], the wear resistance is proportional to the hardness of the wearing body. Therefore, the wear rate decreases with the addition of SiO$_2$ particles.

Furthermore, from the point of view of wear mechanism, the hard SiO$_2$ particles can restrict the severe plastic deformation and adhesion wear based on the observation of wear surface. However, the addition of hard ceramic particles may cause serious abrasive wear. For example, Alpas and
Embury [36] reported that the addition of SiC particles to the 2014 Al alloys caused a marginal increase in the wear rate. However, in this work, it can be found that parts of big SiO$_2$ particles can be broken into fine particles during wear process. Therefore, no serious abrasive wear is observed for the SiO$_2$ reinforced composite. At the same time, these fine particles increase the contact area of SiO$_2$-ring, which further hinders the deformation and adhesion contact of the material during the sliding process [37,38]. Therefore, the addition of SiO$_2$ can increase the friction coefficient, decrease the wear rate, and improve the friction stability of composites.

4.2. Effect of Cu Coating

According to the Figure 3, it can be concluded that the Cu coating is helpful for improving the interfacial bonding between the SiO$_2$ particles and copper matrix. For the specimens 1S and 3S, the detached SiO$_2$ particle can be observed at the wear surface (Figure 6e) and in the wear debris (Figure 7b,d). It indicates that the SiO$_2$ particles without copper coating can be pulled out from matrix and removed from worn surface during wear test due to the poor interface bonding. This can decrease the function of SiO$_2$ particles. On the contrary, for specimens 1S-Cu and 3S-Cu, excellent interfacial bonding between Cu-coated SiO$_2$ particles not only improves the density and hardness, but also restrains the extraction of SiO$_2$ particles from the copper matrix.

4.3. Wear Process

The schematic diagram of the wear process of composites is illustrated in Figure 8. With the sliding distance increase, the temperature of worn surface rise lead to soften of the composites. Under the combined action of shearing stress and normal force, plastic deformation appeared, and thermal stress cracks initiation for all specimens.

![Figure 8](image-url)

**Figure 8.** Schematic diagram of the wear process of the composites: (a) 0S; (b) 3S; (c) 3S-Cu.

For specimen 0S, as the thermal stress cracks grow, large block of materials break off from the composites, resulting in big wear pits on the worn surface, as shown in Figure 8a. For specimen 3S, SiO$_2$ particles can carry the part of normal load [18]. When the cracks encounter SiO$_2$ particles, they may end or spread around from the SiO$_2$ particles. Both of that result in a relatively slight plastic deformation of worn surface and low wear rate. Meanwhile, the hard SiO$_2$ particles exposed on the worn surface can cut the spalling blocks into fine wear debris (see Figure 7a,b,d), this may decrease the
adhesion contact of the sliding counterpart. In addition, a part of SiO\textsubscript{2} particles fracture and embed into the soft copper matrix. The dispersion distribution of broken SiO\textsubscript{2} particles further decreases the metal–metal contact and adhesion wear. However, due to the poor interfacial bonding between uncoated SiO\textsubscript{2} particles and copper matrix, some SiO\textsubscript{2} particles is easy to be pulled out from copper matrix. For specimen 3S-Cu, the excellent interfacial bonding between Cu-coated SiO\textsubscript{2} particles and copper matrix, restrains the extraction of SiO\textsubscript{2} particles from the copper matrix. Therefore, composites reinforced with Cu-coated SiO\textsubscript{2} particles have a lower wear loss.

5. Conclusions

Copper–graphite composites reinforced with Cu-coated or uncoated SiO\textsubscript{2} particles were fabricated by the powder metallurgy technique. The microstructure, density, hardness, and tribological properties of the composites were characterized. The conclusions are drawn as follows:

(1) The hardness of the composites increases with the increase of SiO\textsubscript{2} content. However, the poor interfacial bonding between SiO\textsubscript{2} particles and Cu matrix, result in the relative density decreased with the increase of SiO\textsubscript{2} particles for uncoated SiO\textsubscript{2} particles reinforced composites. Electroless copper coating is helpful to increase the relative density of composites by improve the interfacial bonding between SiO\textsubscript{2} particles and copper matrix.

(2) The addition of SiO\textsubscript{2} lead to increasing of friction stability, friction coefficient, and decreasing of wear rate. This should be attributed to the hard SiO\textsubscript{2} can restrict the severe plastic deformation and adhesion contact in the process of wear.

(3) The electroless copper plating improve the interfacial bonding between SiO\textsubscript{2} and copper matrix, which helps to prevent SiO\textsubscript{2} pull out from copper matrix and further increase tribological properties of the composites.

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