Effect of Graphite Content on the Tribological Behavior of a Cu–Fe–C Based Friction Material Sliding against FC30 Cast Iron

Sun-Zen Chen*1, Jiin-Huey Chern Lin and Chien-Ping Ju*2

Department of Materials Science and Engineering, National Cheng-Kung University, Tainan 701, Taiwan, R.O.China

Focus of the study has been placed on the effect of graphite content on tribological behavior of a Cu–Fe–C based friction material sliding against FC30 cast iron. Experimental results indicated that, after sintering in air, the densities of Cu–Fe–C specimens decreased, oxygen contents increased, and essentially all original metallic constituents turned into oxides. As graphite content increased, the amounts of Fe2O3, Cu2O and Cu–Sn decreased, while CuO and FeSn3 contents increased. The friction coefficient profiles of Cu–10%Steel wool–10%Al–10%Al2O3–10%Sn (vol%) material (C0) and Cu–10%Steel wool–10%C–10%Al–10%Al2O3–10%Sn (vol%) material (C10) are almost identical. After sliding for a few minutes, their friction coefficients start to decay. The friction coefficient of Cu–10%Steel wool–20%C–10%Al–10%Al2O3–10%Sn (vol%) material (C20) maintained to be high and stable throughout tests. The mass loss of C20 was larger than that of C0 or C10. The mass loss of FC30 sliding against C20 was smaller than that of FC30 sliding against C0 or C10. While both particulate and film types of debris were observed on each Cu–Fe–C specimen, more particulate debris was observed on C0 and C10 than on C20 surface. Chemical analysis of worn surfaces indicated that Fe was dominant on C0 and C10 surfaces, while large amounts of Fe and Cu were detected on the worn surfaces of both C20 and FC30.

(Received March 25, 2003; Accepted May 14, 2003)

Keywords: Friction material, copper-iron-graphite, tribology, cast iron, wear

1. Introduction

Copper and iron have been used extensively for various tribological applications.1–6 The copper or iron-based friction materials are most often fabricated using powder metallurgy technique due to its many advantages such as the elimination of solidification-induced chemical segregation and structural defects often accompanied with melting-solidification processes. The ease in mixing of different powders leads to the possibility for developing new composite materials with special physical and/or mechanical properties that are difficult to be produced by melting-solidification processes.

Graphite is often added to copper or iron-based friction materials due to its beneficial effects in sintering process as well as in the modification of friction behavior of the materials. The morphology and distribution of graphite can affect the friction coefficient and wear mode of iron-based materials. For an example, Dudrová et al. found that the density of vacuum-sintered M2 powder steel was sensitive to the amount of copper addition (2–10 mass%) in the steel.7 The authors also observed that a small amount (0.35 mass%) of graphite addition could significantly increase the sintered density by reducing oxide formation and lowering the solidus temperature of the material. Zhang et al. concluded that the cast iron with compacted (vermicular type) graphite had the best combination of high friction coefficient and low wear, in their study of the effect of graphite morphology on tribological behavior of phosphorous cast irons.8 The cast iron with flake type graphite had the largest wear in the cast irons with vermicular, flake and spherical type graphite. The cast iron with spherical graphite exhibited higher wear resistance but lower friction coefficient.

Pabinowicz suggested that only an oxide film with a thickness greater than a critical value (10−8 m) had lubricating capability (decreasing friction coefficient), in his study of the wear behavior of copper and other metals sliding against themselves and stainless steel.9 According to this a relatively high temperature may be needed for an oxide film of sufficient thickness to form.

While transfer film is involved in the wear process of copper and/or iron based materials, the friction and wear behavior of the materials can be largely dependent upon the nature of the film. Ju and Chern Lin observed a film transferred from the softer Cu–Fe–C to the harder 17–22 AV counterpart during the wear process, in their study of the tribological behavior of a Cu–Fe–C alloy sliding against 17–22 AV steel.9) The film thickness could reach several micrometers. Their Auger electron spectroscopic (AES) analysis indicated that the transfer film consisted of large amounts of contaminant elements, mainly carbon and oxygen, and to a lesser extent S, Ca, Na, N and Cl. The same authors also observed that neither transferred element distribution nor wear-induced oxidation process was uniform. The carbon concentration was found lower in the high oxygen region than in the low oxygen region.

In a study of the wear behavior of radioactive brass pins sliding against hard steel rings, Kerridge and Lancaster concluded that the equilibrium rate of film transfer was equal to the rate of pin wear and that the wear debris was derived from the transfer film, not directly from the pin.10) They also noticed that patches of debris on the wear surface were larger than individual transfer particles and suggested that agglomeration must have occurred. It was also reported that transfer can go in either direction, although transfer in one direction may dominate.1,11) The transfer material tends to be patchy (seldom uniform),2,9,12,13) Transfer can lead to low friction/low wear or high friction/high wear, and can be responsible for sudden tribological transitions.2,8,14–16)

The sintering of iron or copper-iron based materials was...
conducted under vacuum or inert atmosphere at temperatures ranging from about 1023 to 1623 K conventionally. Instead of using vacuum or inert atmosphere, the present Cu–Fe–C based friction materials were sintered at lower temperatures in an ordinary air furnace as a cost-effective way. The focus of this study has been placed on the effect of graphite content on the tribological behavior of a Cu–Fe–C based friction material sliding against FC30 cast iron.

2. Experimental and Methods

Table 1 lists compositions of the materials prepared for the study. The powders were mixed in a double-cone type mixer and compacted at room temperature into green bodies under a unidirectional pressure of 590 MPa. The compacted green bodies were heat-treated at 523 K for 7200 s (2 h) at a heating rate of 0.167 K/s (10 K/min), then heated to 905 K for 172800 s (48 h) at the same heating rate.

The sintering process was conducted in an ordinary air furnace. Cu–Fe–C specimens (the stator) of 26 mm in diameter were cut from the sintered body and ground to remove surface oxide and control surface roughness for wear tests. The square-shaped (32 mm × 30 mm) counterface material, as-cast FC30 cast iron (the rotor), had a chemical composition listed in Table 2.

The porosities of as-sintered Cu–Fe–C specimens were determined using ASTM C830 method. The hardness values of Cu–Fe–C specimens as well as FC30 counterface material were determined at HRS and HRB levels, respectively, due to their large differences in hardness. X-ray diffraction (XRD) was carried out to analyze phase changes after sintering. Phases were identified by matching each characteristic peak with that compiled in the JCPDS files. A D-Max/VIII diffractometer (Rigaku, Tokyo, Japan), operated at 30 kV and 20 mA with a nickel filter was used for the study.

Friction and wear tests of the Cu–Fe–C specimens against FC30 were conducted using a home-made disc-on-disc sliding wear tester, as schematically shown in Fig. 1. For this wear tester, a unit rpm is equivalent to an average linear speed of 0.68 m/s. A pressure of 1 MPa and a constant speed of 17 s⁻¹ (1000 rpm) were used for all tests. A strain gage-equipped LRK-100K load cell (NTS Technology, Nara, Japan) was used to determine the friction force/friction coefficient. A five-digit balance was used to measure mass losses of the specimens. Mass loss data were averages of at least five fresh test runs. A K-type thermocouple was placed at a distance of about 3 mm from contact surface to measure the sliding-induced temperature rises. It should be noted that the flash temperatures at very local spots could be far higher than the measured ones.

The roughness of worn surfaces was measured using a profilometer (SE-40D, Kosaka Laboratory, Tokyo, Japan). The surface roughness data were the average values of at least ten measurements. The worn surface and debris morphology were examined using a Nikon FM2 camera and a Hitachi S-4200 scanning electron microscope (SEM). A Vantage (Noran, Wisconsin, US) energy dispersive spectrometer (EDS) attached to the SEM was used for chemical analysis of worn surfaces.

3. Results and Discussion

As shown in Fig. 2, the densities of all Cu–Fe–C specimens decreased after sintering due to the formation of large amounts of various oxides (copper oxide, iron oxide, etc.)
during sintering in air, as will be shown later in XRD patterns. These oxides had much lower densities than their respective non-oxidized metals (copper, steel, etc). The sintering-induced porosity, especially in high graphite specimen (about 34% for C20; 25% for C10 and 26% for C0, as determined using ASTM C830 method), also contributed to the sintering-induced decrease in density. As expected, when graphite content increased, the density decreased due to the lower density of graphite.

Despite their decreased densities, the hardness values of C0 and C10 specimens increased after sintering (Fig. 3) primarily due to the formation of harder oxide phases. The decrease in hardness of C20 is considered due to its much higher porosity level.

Table 3 lists the chemical compositions of polished Cu–Fe–C specimens before and after sintering. The chemical compositions of green bodies were calculated according to their initial volume fractions of each constituent. The chemical compositions of sintered bodies were determined by SEM-EDS. Due to the composite nature of the specimens, non-uniform distributions in chemical composition were observed in all green and sintered specimens. Also indicated in Table 3, despite somewhat non-uniform chemical distributions, the oxygen contents of all Cu–Fe–C specimens always largely increased after sintering due to oxidation of the specimens.

The sintering-induced oxidation phenomenon may be described in more detail from XRD patterns. As indicated in Fig. 4(a), all Cu–Fe–C green bodies exhibited similar XRD patterns, except for graphite peak intensity that increased with graphite content in the specimen. All the metallic constituents remained in their non-oxidized forms.

As shown in Fig. 4(b) after sintering, essentially all original metallic constituents turned into oxides. In other words, the air-sintered Cu–Fe–C specimens largely became composites of oxides. The only metallic phases shown in XRD patterns were Cu–Sn and FeSn2 compounds, that were supposedly reaction products between Cu and Sn and between Fe and Sn, respectively, at high temperature. It was observed that as graphite content increased, the amounts of Fe2O3, Cu2O and Cu–Sn decreased, while the amounts of CuO and FeSn2 increased. It is not certain how graphite has affected the various phase transitions at this moment.

Typical variations in friction coefficient and temperature with time of the three Cu–Fe–C specimens sliding against FC30 at 17 s⁻¹ are given in Figs. 5 and 6, respectively. As shown in Fig. 5, the friction behavior of C0 and C10 appeared almost identical, indicating that a graphite content of 10 vol% (3.3 mass%) in green body did not affect significantly the friction behavior of the present Cu–Fe–C material. Initially the friction coefficients of all three specimens were similar.
After a small rise to 0.45–0.5, the friction coefficients of C0 and C10 started to decay. After 3000 s, the friction coefficient went down to 0.15–0.2. This decay in friction coefficient might not be explained by the formation of sliding-induced oxides that act as a solid lubricant as observed in other cases. As a matter of fact, C20 had much more oxides on its surface (as will be shown in Table 5), yet exhibited a much higher friction coefficient than C0 and C10. As shown in Fig. 5, the friction coefficient of C20 maintained high (0.5–0.6) and stable throughout tests. As will be discussed later, the dramatic difference in friction behavior between C20 and C0/C10 might be explained by their difference in worn surface/debris morphology.

The variations in temperature of Cu–Fe–C specimens sliding against FC30 were in a similar trend to that in friction coefficient. As shown in Fig. 6, the temperatures rose more rapidly for C0 and C10 than those for C20 during early stage of sliding. After sliding for about 600 s, the temperatures for C0 and C10 went down from their peaks of about 493–503 K (Note: the actual contact surface temperature could be much higher than the temperature measured by the thermocouple that was placed at a distance of about 3 mm from the contact surface, as mentioned earlier). At the end of tests (3000 s), they went down to about 423 K. These decreases in temperature with sliding time (Fig. 6) were directly related to their decreases in friction coefficient (Fig. 5). In general, when the temperature decreased, the friction coefficient decreased. The observation that the temperatures for C0 and C10 declined later than their friction coefficients is probably due to the fact that the change in temperature is normally less sensitive than the change in friction coefficient.

For C20, the temperature rose more slowly than that of C0 or C10 at the early stage, but continued to rise to 523–538 K and maintained at this high temperature throughout tests. Again, this non-declining temperature was a result of its stable, non-declining friction coefficient throughout the tests. For many friction materials, a rise in temperature is often accompanied with changes/degradation of properties of the material, that, in turn, causes changes in friction behavior. In the present study, a graphite content of 20 vol% in the green body had apparently stabilized the friction behavior even at a constantly high temperature of 523–538 K for a long time, i.e. 1800 s. Practically, compared to C0 or C10, C20 has a greater potential to be used under severe friction conditions, such as a racing car brake lining material.

It is interesting to note that the mass loss of FC30 was larger than that of its counterpart C0 or C10 despite the higher hardness level of FC30 (90 HRB), as indicated in Fig. 7. As will be shown later, this was due to a debris film transferred from FC30 onto C0/C10 surface that effectively reduced wear of C0/C10. From a practical point of view, this high wear disk/low wear pad combination is usually not a good combination for applications such as brake and clutch.

On the other hand, while the mass loss of C20 was much larger than those of C0 and C10, the mass loss of FC30 sliding against C20 was much smaller than that of FC30 sliding against C0 or C10. The larger mass loss of C20 than that of C0 or C10 is considered to be a result of its higher porosity level (lower density) and lower hardness. The smaller mass loss of FC30 sliding against C20 than against C0 or C10 is considered due to the large amount of debris transferred from C20 to FC30, as will be shown later.

The wear debris on worn Cu–Fe–C surfaces could be categorized into two types by morphological observation: particulate type and film type (Fig. 8). While both types of debris were observed on each Cu–Fe–C surface, the particulate debris was observed on C0 and C10 surfaces more frequently than on C20 surface. In the naked eye the particulate debris-covered surface appeared dull, while the film debris-covered surface appeared shiny. As shown in...
more detail in Fig. 9, the particulate type debris was loosely-bonded, while the film type debris was more tightly-bonded to the surface. Stott has discussed the formation mechanisms and effects of different types of debris.21) Basically, the film type debris is formed by the repeated agglomeration, compaction and sintering processes of fine wear particles during sliding. When loose wear particles are unable to be compacted into layers, particulate type debris are generated.

The observation that more particulate type debris was formed on C0 and C10 surfaces than on C20 surface might explain their different friction coefficients. According to Xiang et al., spherical-shaped particulate debris could reduce friction by the rolling between sliding surfaces.12) On the other hand, the smaller quantity of such lubricative “rollers” on C20 surface led to its higher friction coefficient. Despite more film debris on its surface, the worn surface of C20 was much rougher than that of C0 and C10 (Table 4). It is considered to be a result of its lower hardness, higher porosity level, where more severe ploughing/scratching processes occurred, contributing to its higher friction coefficient and mass loss. The high friction-induced high temperature in C20 might also contribute to its larger mass loss by more severe oxidation at higher temperature.

In the present study the most critical factor that caused formation of the two morphologically different types of debris has to be their different graphite contents. The presence of a large amount of graphite (C20) seems to have assisted in the compaction process of fine wear particles into films. The higher temperature (C20) could also help the compaction of wear particles due to a sintering effect.

Table 5 lists chemical compositions of worn surfaces of both Cu–Fe–C and FC30. As indicated in the table, Fe was found to dominate the worn surfaces of C0 and C10. This clearly indicates that a debris film has transferred from FC30 to C0 and C10 during sliding. Garbar suggested that more factors than hardness (e.g., plastic flow of harder part) could

| Table 4 Roughness values (μm) of worn Cu–Fe–C and FC30 surfaces. |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
|                 | **Cu–Fe–C**     | **FC30**        | **Cu–Fe–C**     | **FC30**        | **Cu–Fe–C**     | **FC30**        |
| **Rₐ**         | 1.82 ± 0.84     | 0.94 ± 0.43     | 3.85 ± 1.24     | 1.24 ± 0.46     | 6.60 ± 3.10     | 4.19 ± 1.09     |
| **Rₘₐₓ**       | 8.40 ± 2.48     | 3.90 ± 1.71     | 13.70 ± 3.36    | 4.10 ± 1.26     | 22.35 ± 12.57   | 13.55 ± 6.03    |

1Arithmetic average of absolute values of all points of profile.
2Maximum peak-to-valley value.

Fig. 8 Photographs of worn surfaces after sliding for 3000 s.

Fig. 9 Scanning electron micrographs showing film type (a) and particulate type (b) wear debris.
be involved when transfer between two surfaces is considered.11) The large amounts of Fe and Cu observed on the worn surfaces of both C20 and FC30 indicates that debris has also transferred from C20 to FC30. As mentioned earlier, the debris transferred from Cu–Fe–C to FC30, that is significant for C20 but not for C0 or C10, is believed to be the primary reason for their large differences in tribological behavior. The observation that the oxygen content was much higher on worn C20 than on C0 or C10 surface indicates a more severe oxidation on C20 surface during sliding. This observation is consistent with the measured higher friction coefficient and higher sliding-induced temperature.

4. Conclusions

(1) After sintering in air, the densities of Cu–Fe–C materials decreased, oxygen contents increased, and essentially all original metallic constituents have turned into oxides. As graphite content increased, the amounts of Fe$_2$O$_3$, Cu$_2$O and Cu–Sn decreased, while the amounts of CuO and FeSn$_2$ increased.

(2) The friction behavior of Cu–10%Steel wool–10%Al–10%Al$_2$O$_3$–10%Sn (vol%) material (C0) and Cu–10%Steel wool–10%C–10%Al–10%Al$_2$O$_3$–10%Sn (vol%) material (C10) appeared almost identical. After sliding for about 200 s, their friction coefficients started to decay. The friction coefficient of Cu–10%Steel wool–20%C–10%Al–10%Al$_2$O$_3$–10%Sn (vol%) material (C20) maintained high and stable throughout tests. The sliding-induced temperature changes were in a similar trend to that in friction coefficient. While the mass loss of C20 was larger than C0 and C10, the mass loss of FC30 sliding against C20 was smaller than that sliding against C0 or C10.

(3) The wear debris on worn Cu–Fe–C surface could be categorized into two types by morphological observation: loosely-bonded particulate type and more tightly-bonded film type. While both types of debris were observed on each Cu–Fe–C specimen, more particulate debris was observed on C0 and C10 surfaces than on C20 surface.

(4) Chemical analysis of worn surfaces indicated that Fe was dominant on C0 and C10 surfaces, indicating debris has transferred from FC30 to C0 and C10 during sliding. The large amounts of Fe and Cu observed on the worn surfaces of both C20 and FC30 indicates that debris has also transferred from C20 to FC30. The much higher oxygen content on worn C20 surface as compared to that on C0 or C10 was consistent with its higher friction coefficient and higher sliding-induced temperature.

REFERENCES


Table 5 Chemical compositions (mass%) of worn surfaces determined by EDS.

<table>
<thead>
<tr>
<th></th>
<th>Cu</th>
<th>Fe</th>
<th>C</th>
<th>Al</th>
<th>Sn</th>
<th>O</th>
<th>Si</th>
<th>Mn</th>
</tr>
</thead>
<tbody>
<tr>
<td>C0</td>
<td>7.3</td>
<td>72.5</td>
<td>1.2</td>
<td>0.7</td>
<td>1.4</td>
<td>14.1</td>
<td>2.1</td>
<td>0.8</td>
</tr>
<tr>
<td>FC30 (vs. C0)</td>
<td>3.1</td>
<td>86.8</td>
<td>3.9</td>
<td>0.2</td>
<td>0.2</td>
<td>2.5</td>
<td>2.0</td>
<td>1.2</td>
</tr>
<tr>
<td>C10</td>
<td>2.2</td>
<td>81.4</td>
<td>0.5</td>
<td>0.2</td>
<td>0.1</td>
<td>12.7</td>
<td>2.0</td>
<td>1.0</td>
</tr>
<tr>
<td>FC30 (vs. C10)</td>
<td>1.3</td>
<td>86.3</td>
<td>4.2</td>
<td>0.1</td>
<td>0.3</td>
<td>4.3</td>
<td>2.3</td>
<td>1.3</td>
</tr>
<tr>
<td>C20</td>
<td>25.4</td>
<td>41.7</td>
<td>1.4</td>
<td>2.3</td>
<td>4.6</td>
<td>23.2</td>
<td>0.9</td>
<td>0.5</td>
</tr>
<tr>
<td>FC30 (vs. C20)</td>
<td>26.1</td>
<td>42.5</td>
<td>1.3</td>
<td>2.3</td>
<td>4.3</td>
<td>22.0</td>
<td>0.8</td>
<td>0.6</td>
</tr>
</tbody>
</table>