Original Article

Effects of friction film mechanical properties on the tribological performance of ceramic enhanced resin matrix friction materials

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ABSTRACT

In the tribological properties of ceramic enhanced resin matrix friction materials, friction film created at tribological interface plays significant roles. Herein, the present study primarily focused on the correlation between friction film mechanical properties and friction performance, which had been rarely reported. Bigger quartz particles were designedly selected to reinforce one certain resin matrix. The friction performance was tested on a pad-on-disc friction machine. The mechanical features of friction film formed on particles surface were characterized through the Nano/Micro indentation method, and the microtopography was observed by SEM. Results showed that friction coefficient decreases with the increasing binders in the range of 21.9–29.4 wt.%. It was ascribed to the gradual decrease of friction film strength which reduced the resistance during adhesive friction. The fade resistance was inversely proportional to the friction film thickness. It was attributed to the degradation of organic binders at high temperatures which adversely affected the friction film integrality. The materials wear was not detectable due to the ceramic particles having great wear resistance.

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1. Introduction

Resin matrix friction materials have been historically used in the automotive braking field. However, the reduced friction level at high temperatures (i.e. fade), caused by the degrada-
known as contact patches, third body, friction layer, or contact plateaus [4].

Vlastimil et al. [5] investigated the formation of friction film under different SiC particle size conditions. Results showed that the stability of friction film increases with the particles size, and is responsible for the stable friction coefficient during sliding. Publications [6,7] explored the effects of contact plateaus morphology on the noise propensity. It was concluded that friction systems with many small contact plateaus generate more squeal than those with few large plateaus. Sun et al. [8] studied the roles of friction film in the disc wear rate and concluded that the formation of friction film on the surface of particles can reduce the hurt from sharp corners or edges to the disc. Cho et al. [9] studied the friction film features versus particles size and found that transient friction film can cause excessive materials wear and poor friction stability, while stable friction film can provide excellent friction stability with less materials wear. In addition, some efforts [10] on the composition and microstructure of friction film were made to study the wear mechanisms of friction materials.

Rare studies on the mechanical properties of friction film had been reported. Dadkar et al. [11] investigated the correlation between friction surface morphology and disc temperature, finding that the smooth friction film inducing lower shear stress was responsible for the lower temperature. Huang et al. [12] provided a numerical method to calculate the mechanical properties of friction film.

However, few studies were reported to characterize the friction film mechanical properties for friction composites through experimental methods, particularly so for ceramic enhanced resin matrix friction materials. Hence, the present paper aims to use the Nano-indentation method to characterize the friction film mechanical properties of ceramic enhanced resin matrix friction materials and explore the relation with tribological performance. It is critical to develop new material concepts for applications in the automotive brakes.

2. Experimental materials and methods

2.1. Experimental materials

Three composite samples (S-1, S-2, and S-3) were prepared through hot-press technology according to the conventional procedures given in publications [13]. The hot-press process was controlled at 25 MPa and 170 °C for 30 min. Then the semifinished product was subjected to a heat treatment at 175 °C for 6 h. In each sample, 16 vol.% industrial grade quartz particles with the dimension of 3.0 ± 0.5 mm were selected to reinforce the resin matrix. These ceramic particles of good wear resistance could provide contact plateaus during sliding, which is beneficial to the aggregation of debris surrounding/on them [8]. As a result, uniform friction film of large area is easily to be formed. The shape of particles was triangular prism, as shown in Fig. 1. The contents of binders in the resin matrix corresponding to S-1, S-2, and S-3, were 21.9 wt.%, 24.4 wt.% and 29.4 wt.%, respectively (Table 1). The mass discrepancy was balanced by barite which always works as space filler [14].

2.2. Friction tests

The friction tests were carried out on a pad-on-disk type friction tester. Fig. 2 exhibits the schematic diagram. Gray iron with a nominal radius of 150 mm was employed as the counter disc which was rotated by a motor. Two same samples with the dimension of 25 × 25 × 7 mm³ were pressed on the disc. The tests were performed with a constant rotating velocity of 480 rpm and a constant normal load of 1225 N. The friction temperature was controlled through an air blower and a heat device, and measured by a thermocouple that slightly touched on the disc. In this study, two tests were included as follows.

I The friction coefficient of S-1, S-2, and S-3.

Fig. 1 – The digital photograph of quartz particles.

![Digital photograph of quartz particles.](image)

Table 1 – The composition of resin matrix in different samples.

<table>
<thead>
<tr>
<th>Ingredients</th>
<th>Content (wt. %)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>S-1</td>
</tr>
<tr>
<td>Binders</td>
<td>21.9</td>
</tr>
<tr>
<td>Barite</td>
<td>61.5</td>
</tr>
<tr>
<td>Vermiculite</td>
<td>7.4</td>
</tr>
<tr>
<td>Carbon fibers</td>
<td>6.4</td>
</tr>
<tr>
<td>Graphite</td>
<td>2.8</td>
</tr>
</tbody>
</table>

![Schematic diagram of the friction tester.](image)

Fig. 2 – The schematic diagram of the friction tester.
Prior to the test, S-1, S-2, and S-3 were all ground by 600# SiC paper. Subsequently, a running-in procedure was followed to ensure a conformable contact between friction couples. The test lasted for 5000 revolutions with a constant temperature of 80 °C. The average friction coefficient was collected. For the data accuracy, each sample was tested three times. The materials thickness wear was measured through a height indicator with the accuracy of 0.001 mm.

The fade performance of S-1 versus the roughness of particles surface.

The difference in roughness of particles surfaces was obtained through different size of SiC paper, as reported in the literature [15]. Sample S-1-200# has the roughest particles surface, followed by S-1-600# and then S-1-1000#, which were ground by 200#, 600#, and 1000# SiC paper, respectively. Then a running-in procedure was carried out. The test consisted of five stages: 100 °C, 150 °C, 200 °C, 250 °C, 300 °C, and 350 °C. Each temperature lasted for 5000 revolutions. The average friction coefficient during every stage was collected.

2.3. Friction film characterization

Fig. 3 exhibits the micro-morphology of S-1 surfaces before and after test (I). Before test, the particles surface was very rough, characterized by many convex bodies (Fig. 3 (a)). After test, these particles developed into primary contact plateaus due to the great wear resistance. Meanwhile, a uniform friction film of large area was only observed on the surface of particles as expected but not in the resin region (Fig. 3 (b)). This was because the friction film was mainly formed by compacted debris [8], while the low-lying resin region could not be subjected to the normal load. After test (I), the mechanical properties of friction films corresponding to S-1, S-2, and S-3 were evaluated by using MTS Nano-Indentor XP with a Vickers indenter. The applied load was 0.49 N. The dwell time was 15 s. The micro-topography of indentations on friction film was observed by SEM.

After test (II), the corresponding cross sections of friction films on S-1-200#, S-1-600#, and S-1-1000# were observed by SEM.

3. Results

3.1. Friction performance

Fig. 4 (a) exhibits the friction coefficient of S-1, S-2, and S-3 in test (I). It can be inferred that the friction coefficient decreases with the increasing binders in resin matrix. Some studies focusing on carbon fiber reinforced paper-based friction materials and carbonized copper-phenolic based friction materials got the identical result and held that the easy formation of lubricant film with the increasing resin was the key factor [16,17]. Cho et al. [18] obtained a completely opposite result...
when they performed this research on a non-asbestos organic type formulation. They considered that the high hardness of resin was responsible for the increasing friction coefficient. The roles of resin in the friction mechanisms had been widely studied, but without reaching any agreement.

Fig. 4 (b) presents the fade performance of S-1 versus the roughness of particles surface in test (II). At lower temperatures, the sample with rougher particles surface has a higher friction level. This is in agreement with most publications [19,20] corresponding to ceramic-metal friction couples. In the range of 100–250 °C, the friction coefficient of each sample increases gradually as the temperature rises. The rougher the surface is, the more obvious is. But when the temperature is over 250 °C, a decrease of friction coefficient for every sample is exhibited, indicating a fade phenomenon. Moreover, S-1-200# shows the severest fade, followed by S-1-600# and then S-1-1000#. So far, little literature has been reported to study this subject.

With respect to the materials wear, the thickness reduction for every sample was not detectable. Namely, the wear resistance is excellent. It was attributed to the high hardness of ceramic which determined the materials wear behavior. This issue had been widely reported [5,8,9], so little attention in the present study was payed.

3.2. Mechanical features of friction film

Fig. 5 provides the micro-topography of indentations on different friction films. An extremely irregular indentation on S-1 is exhibited in the form of chunk peeling (Fig. 5 (a)). Several cracks propagate along the diagonal. It suggests an obvious brittleness for this friction film. By contrast, the indentation on S-2 has a slighter damage (Fig. 5 (b)), which means an improvement for the ductility or toughness of friction film. With respect to S-3, the indentation is relatively in a good condition, and cracks are parallel to the sides (Fig. 5 (c)). Thus, a conclusion can be drawn that the friction film on S-3 has the highest plasticity/toughness, followed by the friction film on S-2 and then S-1. Similar results had been obtained by the publications [21,22] studying the effect of coating technology on the toughness of titanium alloy surface.

Additionally, it can be observed that the diagonal length of indentions in Fig. 5 is about 12 μm. Consequently, the depth of the indentation is about 1.8 μm, which is calculated according to the geometrical feature of the Vickers indenter. Compared with the thickness of friction film shown in Fig. 6, the indentation was so shallow that would not be influenced by the substrate.

Fig. 6 provides the micro-topography and cross sections of friction films formed on S-1-200#, S-1-600#, and S-1-1000#. As shown, the thickness of friction films in Fig. 6 (a), (b) and (c) are about 20 μm, 10 μm, and 6 μm, respectively. A gradual increase as a function of particles surface roughness is revealed.

4. Discussions

Fig. 3 (a) and (b) exhibit the micro-morphology of particles surface before and after friction test (l), respectively. It can be observed that the particles surface before friction is very rough and characterized by many convex bodies. When the relative sliding between friction pairs began, these hard convex bodies interacted with the counter disc violently, causing a mechanical plowing to the disc, as well as the micro-fracture of the convex bodies [23]. At the same time, the disc also rubbed against the resin matrix. Lots of wear debris generated during above process could assemble on the particles surface and develop into friction film under normal load and shear stress [24]. To verify this deduction, the micro-morphology of particles surface during test was observed by SEM. Fig. 7 (a) shows the micro-morphology of particles surface corresponding to 2400 r in test (l), where some region on the particles surface has been covered with friction film. Fig. 7 (b) presents the magnified morphology of local region marked by a black box in (a), where the obvious compacted wear debris is exhibited. The friction film formation mechanism is absolutely confirmed. As the friction process continued, wear debris was continuously generated and compacted, resulting in the growth of
friction. Finally, the friction film tended to be uniform and continuous, as shown in Fig. 3 (b).

Fig. 4(a) shows the effect of resin content on friction coefficient. For this issue, some attention had been given, as listed in Table 2. Because of the different formulation and testing conditions, there were some disputes on the roles of resin binders. For instance, Fei and Ho et al. [16,17] held that the higher content of resin is useful for the formation of lubricant film at tribological interface, while Cho et al. [18] argued that the resin possessing higher hardness enhances the abrasive friction.

In this paper, the particles addition actually dominated the friction mechanisms. It is well known that the friction mechanisms during sliding are closely related with the worn surfaces of materials [3,4]. Fig. 3 (b) gives the worn surface of S-1, which is characterized by particles rising above the surrounding lowland. Thus, two different kinds of friction force could be identified to be responsible for the friction coefficient, namely mechanical plowing and adhesive friction [25]. The former was generally induced by sharp corners or edges of particles inserting into the softer disc, also known as abrasive friction. When relative sliding between the friction couples happened, the plastic deformation resistance of the disc resisted against the mechanical plowing. The latter was established between the friction film and disc through molecular force or other forms of bonding force developing some adhesive junctions, also known as adhesive friction [25,26]. The resistance against the rupture of adhesive junctions contributed a lot to the friction force. The former was determined by the mechanical properties of softer disc, while the latter was deeply influenced by the real contact area and the shearing strength of adhesive junctions. In our previous work [27], the friction coefficient of ceramic enhanced resin matrix friction materials was quantified by a simple physical model, as given in Eq. (3). Where \( \mu \) stands for friction coefficient, \( \lambda \) means the geometrical factor of asperities on particles surface, \( S_0 \) the total contact area of samples, \( \omega \) the content of ceramic particles, \( r \) the shearing strength of friction film, \( N \) the applied normal load, and \( \sigma_0 \), the yield strength of disc.

\[
\mu = \frac{\lambda}{2} + \frac{S_0 \omega r}{N} - \frac{r}{\sigma_0} \tag{1}
\]

\( r \) for polymer-metal friction couples usually ranges from 0.2 to 0.5 MPa [28], which is far less than the yield strength of disc \( \sigma_0 \), so \( \frac{r}{\sigma_0} \) can be ignored here. In addition, \( \lambda, S_0, \omega \) and \( N \) are constants in this case. Consequently, the different friction coefficient for S-1, S-2 and S-3 was mainly caused by the shearing strength of friction film. This can be proved by Fig. 8 to some extent, where an obvious shearing deformation of the film is presented.

Fig. 5 reveals that the plasticity or toughness of friction film increases with the increasing binders in resin matrix. This is in agreement with the publications [29,30], where held that high content of binders having many polar and flexible segments is useful for the improvement of friction film integrality and flexibility. In this paper, the friction film chemistry for S-1, S-2, and S-3 was analyzed through XPS (Spectrometer ESCA-750, Mg-Ka radiation). Obvious changes in the C 1s spectra were observed, as shown in Table 3. With the increasing binders, the relative intensity of -C−OH and −C=O groups [29] contained in the polar or flexible segments increase gradually. This is in agreement with above theory to some extent. However, the increasing plasticity had some negative effects on the strength of friction film, reducing the deformation resistance. Thus, the decreasing friction coefficient with the increasing binders occurred (Fig. 4(a)).

For the friction performance in Fig. 4(b), S-1-200# and S-1-600# have higher friction level than S-1-1000# at lower temperatures. This could be explained by the higher abrasive friction. Namely, rougher surfaces with more convex bodies caused violent mechanical interactions at the friction interface [31]. From 100 to 250 °C, the friction coefficient for each sample increases gradually as the temperature rises. Moreover, the rougher the particles surface is, the more obvious
Table 2 – The relation of friction film mechanical properties with friction coefficient.

<table>
<thead>
<tr>
<th>Serial number</th>
<th>Materials</th>
<th>Resin content</th>
<th>Testing conditions</th>
<th>Friction coefficient versus resin content</th>
<th>Reasons</th>
</tr>
</thead>
<tbody>
<tr>
<td>Peers’ work [16]</td>
<td>Paper-based friction materials</td>
<td>35–50 wt.%</td>
<td>0.5 MPa, 70–75 °C, 9.2 m/s</td>
<td>Friction coefficient decreases with resin content</td>
<td>The easy formation of lubricant film</td>
</tr>
<tr>
<td>Peers’ work [17]</td>
<td>Carbonized copper-phenolic based friction materials</td>
<td>5–25 wt.%</td>
<td>1 MPa, Atmospheric temperature, 1.5 × 10^2 m/s</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Peers’ work [18]</td>
<td>Non-asbestos organic type formulation</td>
<td>15–30 wt.%</td>
<td>0.3 MPa, 25 °C, 1 m/s</td>
<td>Friction coefficient increases with resin content</td>
<td>The high hardness of resin binders</td>
</tr>
<tr>
<td>This work</td>
<td>Ceramic enhanced resin matrix friction materials</td>
<td>21.9–29.4 wt.%</td>
<td>0.98 MPa, 80 °C, 7.9 m/s</td>
<td>Friction coefficient decreases with resin content</td>
<td>The decrease of friction film shear strength</td>
</tr>
</tbody>
</table>

is. This was because more debris could be generated and arrested surrounding the convex bodies for S-1-200# and S-1-600#, which would develop into subsequent friction film under normal load and shear stress. As a result, the faster growth of friction film led to the more obvious increase of adhesive friction. But when the temperature is over 250 °C, S-1-200# and S-1-600# show a severe fade. This was closely related with the thickness of friction film. Fig. 6 reveals that the rougher particles surface has a relative thicker friction film. The absolute content of organic binders in the thick film was higher, which was responsible for the accelerated collapse of friction film at higher temperatures due to the thermal degradation. Therefore, a severe fade was performed for S-1-200# and S-1-600#. 
Table 3 – The XPS analyses of C 1s in friction film for different samples.

<table>
<thead>
<tr>
<th>Samples</th>
<th>C 1s</th>
<th>— C—OH</th>
<th>Binding energies</th>
<th>Relative intensity</th>
<th>— C=O</th>
<th>Binding energies</th>
<th>Relative intensity</th>
</tr>
</thead>
<tbody>
<tr>
<td>S-1</td>
<td>285.6 ev</td>
<td>5.7%</td>
<td>287.2 ev</td>
<td>2.4%</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S-2</td>
<td>285.6 ev</td>
<td>6.1%</td>
<td>287.2 ev</td>
<td>2.7%</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>S-3</td>
<td>285.6 ev</td>
<td>6.8%</td>
<td>287.2 ev</td>
<td>3.2%</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

5. Conclusions

The relationship between the friction film mechanical properties and friction performance of ceramic enhanced resin matrix friction materials was primarily investigated. Some conclusions were drawn as follows:

1) Friction coefficient decreased with the increasing binders in resin matrix. Materials with rougher particles surface had a higher friction level at lower temperatures, but a severer fade at higher temperatures.
2) The shearing deformation resistance of friction film decreased with the increasing binders in resin matrix, which was responsible for the decreasing friction coefficient.
3) Rougher particles surface was beneficial to the formation of thicker friction film, but not to the fade resistance of friction materials.

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