Effect of Braking Speeds on the Tribological Properties of Carbon/Carbon Composites

Yu Shu¹, Chen Jie¹, Huang Qizhong¹, Xiong Xiang¹, Chang Tong¹ and Li Yunping²,*

¹State Key Lab for Powder Metallurgy, Central South University, Hunan Changsha, 410083, P. R. China
²Institute for Materials Research, Tohoku University, Sendai 980-0813, Japan

This paper describes the tribological properties of carbon/Carbon composites used as braking materials under various braking speeds, in which the materials with three kinds of microstructures were used: rough lamina (sample A), smooth lamina (sample B), and the mixture of rough lamina and smooth lamina (sample C), respectively. Friction tests were carried out through a laboratory dynamometer. Polared optical microscopy (OM) and scanning electron microscopy (SEM) were used to characterize the microstructure and worn surface. Results indicated that the friction coefficient of sample A reached a peak value at braking speed lower than that in both sample B and sample C. However, when the friction coefficients reached to the peak values the temperature inside the worn surface was observed to be approximately 250°C for all of the samples. The weight losses in all of the samples were observed to increase with increasing braking speed; however, the oxidation losses at speed of 28 m·s⁻¹ are higher than that of 30 m·s⁻¹ for all of the samples. [doi:10.2320/matertrans.M2009390]

(Received November 24, 2009; Accepted March 1, 2010; Published April 15, 2010)

Keywords: carbon/Carbon composites, pyrolytic carbon, tribological property, braking speed, worn surface

1. Introduction

Carbon/Carbon (C/C) composites have unique properties such as high specific strength, specific heat capacity, melting point, and thermal conductivity etc. In addition, the heat shock properties of C/C composites are superior to most of the other friction materials. The aforementioned properties enable the C/C composites to be used as brakes in aircrafts and to carry out three functions simultaneously: producing friction, transferring mechanical load, and absorbing kinetic energy.¹–³ Furthermore, as the aerospace brakes, C/C composites have more merits such as the light weight, long service life, low noise, and smooth operation, etc. compared to other materials used as aerospace brakes.⁴–⁵

In general, C/C composites are manufactured through chemical vapor deposition (CVD) process, impregnating resin or pitch, or the process combining these two methods. The CVD processes are divided into isothermal CVD and thermal gradient CVD processes. It has been proved that the microstructure of C/C composites obtained from CVD process are superior to both resin carbon and pitch carbon from the impregnating process if used for aircraft brakes. In general, the first choice for synthesizing C/C composites used for aircraft braking materials is isothermal CVD because the pyrolytic carbon obtained is a single phase, while a mixed phase pyrolytic carbon exist in final products from the thermal gradient CVD because of the influence of thermal gradients during CVD processing. For C/C composites used in braking process, friction and wear properties are essential, and in reality, different types of aircrafts bring various braking conditions, such as load weight, landing speeds and the serve circumstances, which have a strong influence on the tribological properties of the used materials. In addition, the materials, containing different microstructures, will also give rise to different tribological properties, so it is worthwhile to study the friction and wear performance of C/C composites brakes with different microstructure under different braking speeds.

In this paper, we chose three kinds of samples, in which two kinds of C/C composites with rough lamina and smooth lamina structures were manufactured in isothermal CVD, and the third one with mixed phase structure was manufactured in thermal gradient CVD.

2. Experimental

C/C composites were manufactured from a quasi-three dimensionally needled carbon fiber preform, which was fabricated by needling the stack of alternating layers of non-woven carbon cloth and carbon felt made from chopped fibers. Carbon fiber preform was densified by CVD process under the deposition temperature from 1123 to 1223 K in the atmosphere containing both propylene and nitrogen used as precursor and carrier gas, respectively. The densities of the final C/C composites are 1.60–1.65 g/cm³. Heat treatment temperature at 2573 K for 2 h in the argon protection was carried out to all of the samples. The cylindrical specimen was machined into a size of Φ 438–237 mm × H 22 mm before testing.

The microstructure of pyrolytic carbon was observed under MEF3A polarized optical microscope and the worn surface was analyzed under JSM-5600LV scanning electron microscopy. The flatness of friction film was observed and quantitatively analyzed by the KH-7700 three-dimensional digital microscopy. Thermal diffusivity α was measured in JR-3 laser thermal conductivity instrument, the thermal conductivity λ(W·m⁻¹·K⁻¹) can be obtained from the following equation:

\[ \lambda = 418.65 \times \alpha \times C_p \times \rho, \]

where \( C_p \) and \( \rho \) denote the heat capacity, density of C/C composites, respectively.

The friction and wear test of C/C composites, simulating the real condition of aircraft brake discs, were conducted on
a home-made laboratory scale MM-1000 inertia type of dynamometer (Fig. 1), in which the top speed is 9000 rm s\(^{-1}\) and the largest torque is 15 kg m. Ring specimens (Φ75–53 mm × H14 mm) for friction testing are coupled with one dynamic and one static disk. When the flywheel, which rotated with the dynamic ring sample, is accelerated to a certain rotational velocity, braking is achieved through the friction between the dynamic and static rings under a certain braking pressure. The relationship between braking moment and braking time was recorded by computer. The friction coefficient is evaluated by the following expression

\[ M = \mu (r_1 + r_2) F_n / 2, \]  

where \( M \) denotes moment; \( \mu \) the friction coefficient; \( F_n \) load; \( r_1 \) inner radius; \( r_2 \) outer radius. The braking parameters used in current research are listed as followed:

- inner radius: 30 kg m\(^{-2}\); braking pressure: 0.6 MPa, braking speed: 5 m s\(^{-1}\), 10 m s\(^{-1}\), 15 m s\(^{-1}\), 20 m s\(^{-1}\), 25 m s\(^{-1}\), 28 m s\(^{-1}\), and 30 m s\(^{-1}\).

The weight loss \( L_w \) and the linear wear \( L_d \) of sample can be obtained directly by measuring the sample after testing, so the oxidation loss \( L_o \) could be calculated by

\[ L_o = L_w - S \times \rho \times L_d \]  

where \( S \) and \( \rho \) denote the frictional surface area and density of sample, respectively. The temperature of sample during braking process is detected in the area 1 mm beneath the frictional surface of stator sample.

3. Results and Discussions

3.1 Microstructure

Several factors that affect the microstructure of pyrolytic carbon during CVD process are temperature, furnace pressure, gas residence time and the initial porosity of felt body etc. In the current study, same felts (fiber volume including Z-fiber content, preparation process and heat treatment temperature etc.) were used in various CVD processes, and by adjusting the process parameters aforementioned, C/C composites with different microstructure were obtained in CVD process as shown in Fig. 2. In the Fig. 2, Samples A, B were deposited in two isothermal CVD processes with different experimental conditions, while sample C was from thermal gradient CVD process. It can be observed from Fig. 2(a) and (b) that: due to the uniform temperature distribution inside the isothermal CVD chamber, single phase structure of pyrolytic carbon can be obtained under the defined process parameters. Due to the wide temperature gradient in the thermal gradient CVD process, the structure of pyrolytic carbon shows a large diversity, namely, the coexistence of various structures and boundaries that differ in microstructures could be observed (Fig. 2(c-1) and (c-2)).

Under polarized optical microscope, the Maltese Crosses have been observed in the cross-section of all samples indicating that the deposition were oriented concentrically to the fiber. The microstructure of sample A was optically anisotropic with a high phase shift in which the extinction angles was high as 21° and the thin and narrow columnar growthcones, that running through the whole cross-section of sample, was the most important characteristics of rough laminar\(^{6,7}\) for its primary only one cones,\(^8\) not including the second or more regeneration cones. Since the Maltese Cross was strongly disrupted by the cones, so that its appearance is rough (justifying the generic name of rough laminar, RL in short). At the same time, for the high degree of preferred orientation and anisotropy of RL, cross-section surface appear rough and layering because the reflected light is stronger than that of diffuse scattering under the polarized light. The cross-section of sample B manifests very smooth and regular Maltese Crosses. The ringed cracks can be seen clearly after heat treatment, indicating this material, named smooth laminar (SL), is hard and brittle. In the same polarized light intensity and magnification, the reflectance of sample B is lower than that of A with its’ extinction angles was 10, so the image appears to be more smooth.\(^9\) AS to sample C, there are two kinds of structures coexisted, which are similar to both sample A and B, respectively. The structure of C-1 belongs to RL for the thin and narrow growthcones that grows throughout the cross-section of sample. While in structure of C-2 the circular crack after heat treatment was observed, which is characterized as SL structural. Therefore, in sample C mixed structure of both RL and SL existed, which is the characteristics of pyrolytic carbon manufactured in thermal gradient CVD process.

3.2 Tribological properties under different braking speeds

The changes in coefficients of friction (COF) as a function of braking speed are showed in Fig. 3: firstly, COF of sample A is only 0.1 at 5 m s\(^{-1}\) that is lower than that of B and C. With the increase braking speeds, the COF increased to a peak value of about 0.47 at a braking speed of approximately 15 m s\(^{-1}\), and then declined toward a stable value of approximately 0.35 when the braking speed is above 20 m s\(^{-1}\). Longley et al.\(^{10}\) suggested these phenomena are consistent with the theory that the first low COF values of highly graphitic carbon is due to low surface forces associated with the presence of absorbed gases, not to low shear strength, nor to the separation of surfaces by rolled-up wear products. The latter increase of COF was attributed the
increase in the real contact area with the increase of the friction force.

The curves of sample B and C are similar for they commonly demonstrated hard, largely non-graphitic structure-SL: in the early stages of sliding (from 5 m/s to 15 m/s) between freshly-prepared surfaces, the COF decrease to a very low value (0.25), but then subsequently increase to a high value (0.42) at the 20 m/s and keep the constant value (0.40) at higher speed. This appearance is in accord with the study of Longley and J. K. Lancaster,11) the low value is associated with the formation of a uniform film of consolidated wear debris which appears to act as a classical thin-film solid lubricant—a film of low shear strength on a harder substrate.12) After the formation of the debris film, the wear rate also becomes very low. Insufficient debris is now produced to maintain the film and as it is gradually removed, the friction increases to a higher constant value which can be preserve with the increase of the real area of contact.

According to the study of Fitzer,5) the friction coefficient peak of C/C brakes appeared at the low run-in speed which is called “low-energy peak”. Results show that: “low-energy peak” appeared at 15 m/s in case of sample A (RL), while for the materials containing SL structure (Sample B and C), the “low-energy peak” was observed at higher speed (at about 20 m/s in both materials). The above-mentioned differences in the three kinds of materials are considered to be related to the desorbing ability of water molecules in the friction surface since the microstructures of these materials are different.

Worn surfaces of C/C composites after braking test in various speeds were observed as shown in Fig. 4. The friction film of material A is continuous with fewer broken fibers and pores from low speed (5 m/s) to high speed (30 m/s), while the film of material B has obvious pores even under the high speed (30 m/s), indicating the friction film has not been formed sufficiently. The formation of film has a great impact on the fictional properties of C/C brakes including the friction coefficient and the wear rate. The smooth and continuous friction films lead to the stable frictional coefficient and low wear. However, no obvious difference could be observed from SEM imagine although the materials and friction behaviors are much different as shown in Fig. 3.
According to the reports of C. Pevida and J.-C. Rietsch, the wear debris of different C/C composites or under different braking energy has similar physicochemical properties. Though there was also no obvious difference as shown in the SEM micrographs in the current study, by using three-dimensional digital microscopy, the flatness degree of friction films for these three kinds of materials at braking speed of 20 m/s were observed to be different as shown in Fig. 5, the results are tabulated in Table 1. It was observed that the film flatness of material A is superior to that of material B and C for the lowest drop height, indicating the friction film is relatively uniform, which is considered to be reason of no frequent wave in friction coefficients from 10 m/s to 30 m/s as shown in Fig. 3.

The temperature of samples in various braking speeds, measured inside of the sample 1 mm beneath the worn surface, are shown in Fig. 6; it can be observed that, temperatures of friction surface were observed to increase with the increasing braking speed (Fig. 6). In both samples A and B, at higher speeds the temperatures inside the sample could not be measured because the temperature have been beyond the range of the thermal couple, and only the temperature of sample A have been detected throughout seven speeds for its highest thermal conductivity, specially its highest thermal conductivity ($\lambda_\perp$) perpendicular to the friction surface, which is thought to be also beneficial to maintain smooth torque, to reduce material oxidation loss, and to prolong the service life of brake disc (see Table 2). The higher the thermal conductivity of material is, the faster the heat, which generated in the braking process, is transferred to outside. In addition, it can also observed that no matter what kind of materials, once the “low-energy peak” appears, the temperature detected are observed to be approximately 250°C which is close to the report of B. K. Yen, in which the “low-energy peak” was reported to

<table>
<thead>
<tr>
<th></th>
<th>A</th>
<th>B</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Avera</td>
<td>56.76</td>
<td>68.38</td>
<td>64.73</td>
</tr>
</tbody>
</table>

Table 1 Average drop height of friction film in three kinds of samples after friction test at 20 m·s⁻¹.

Fig. 4 The morphology of the worn surface under the different braking speeds.
According to the report of C. Blanco, there are many structural defects such as pores and dislocations in C/C composites, because the matrix carbon (including pyrolytic carbon and resin carbon) formed in CVD process is not the ideal graphite crystal. In friction surfaces with numerous defects, oxygen atoms are easily to be chemically absorbed and form the bands of C-O and C=O, and the water molecules are physically adsorbed outside of the oxygen atoms. At lower speed, the water molecules is able to act as friction lubricant, which leads to the low friction coefficient; with the increasing braking speed, surface temperature increased to a certain value and water molecules begin to be released, and the friction coefficient will reach to a peak value for the lubrication disappearing, accordingly. In addition, after the heat treatment at temperature of 2300°C, structure of sample A is the closest to the ideal graphite crystal if referred the graphitization degree of C/C composites from XRD measurements (see Table 3), so it reach the peak in a earliest stage for its least adsorption of water molecules in the friction surface in sample A.

The weight losses of three kinds of materials include linear wear and high temperature oxidation abrasion. The weight loss can be calculated through different weight of sample before and after experiment, and the linear wear can be detected by the measurement of the thickness of sample before and after experiment. The value of former is observed to be always higher than that of latter, and the gap is considered as the oxidation abrasion (eq. (3)). The weight loss and oxidation abrasion of three samples were also observed to increase with the speed (Fig. 7). However, it has to be noted that, oxidation abrasion was observed not to increase with the speed all the time: the oxidation abrasion at 30 m/s is lower than that of 28 m/s in all the samples (see Fig. 8). That is to say the linear wear in these kinds of three samples increased greatly if referred eq. (3). Authors believe that under the condition of high-speed and high-

<table>
<thead>
<tr>
<th>Thermal conductivity</th>
<th>a</th>
<th>b</th>
<th>c</th>
</tr>
</thead>
<tbody>
<tr>
<td>$\lambda_{\perp}$ (W m$^{-1}$ K$^{-1}$)</td>
<td>47.1</td>
<td>19.38</td>
<td>23.15</td>
</tr>
<tr>
<td>$\lambda_{\parallel}$ (W m$^{-1}$ K$^{-1}$)</td>
<td>99.8</td>
<td>47.15</td>
<td>78.09</td>
</tr>
</tbody>
</table>

Table 2 Thermal conductivity of three kinds of samples after heat treatment at 2300°C for 2h, where $\lambda_{\perp}$ and $\lambda_{\parallel}$ represent the thermal conductivity vertical and parallel to the friction surface.

<table>
<thead>
<tr>
<th>Graphitization degree (%)</th>
<th>A</th>
<th>B</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>63.2</td>
<td>28.9</td>
<td>55.43</td>
</tr>
</tbody>
</table>

Table 3 Graphitization degree of three kinds of sample after heat treatment at 2300°C for 2h.
energy, there are more serious damaged in the friction surface: the old lubricant film was constantly destroyed, and the new lubricant film has been continually formed although continuous friction films were formed during testing (Fig. 4(a-4), (b-4), and (c-4)). In this process, there is always a part of “fresh” surface exposed that makes linear wear and oxidation abrasion relatively large. Furthermore, the oxidation weakened the surface and sub-surface, which in turn facilitated removal by mechanical action at the rubbing surface. However, with the increase of braking speed, it is considered that with the high temperature in the friction surface, the bonding energy between the graphite layers is lowered, and the graphitization degree of the wear debris particles is improved accordingly, which is considered to lower the hardness of wear debris, which made the friction film more susceptible to be deformed. So the lubricant film is formed in the friction surface to reduce the oxidation abrasion effectively (Fig. 4). In addition, according to the study of Li and Sheehan, the phenomenon of that the oxidation abrasion at 30 m·s⁻¹ is lower than that of 28 m·s⁻¹ for all of the samples due to the low bond energy between the graphite layers and the hardness of wear debris at high temperature in the friction surface.

4. Conclusion

With the increasing braking speeds, the friction coefficient of sample A increased to the peak value at the speed of 15 m·s⁻¹, and then declined toward a stable value. The friction curves of sample B and C were similar: coefficient of friction is minimum at the speed of 15 m·s⁻¹ and increased to a peak value at the 20 m·s⁻¹, then decreased to a constant value.

No matter what kinds of material, when “low-energy peak” appears, the temperatures under the friction surface 1 mm are observed to be approximately 250°C.

The weight loss of materials increased with the increasing braking speed, while the oxidation abrasion at 30 m·s⁻¹ are less than that of 28 m·s⁻¹ for all of the samples due to the low bond energy between the graphite layers and the hardness of wear debris at high temperature in the friction surface.

Acknowledgements

This research was supported by National natural science foundation (50702078), National key basic research support foundation (2006CB600901), and National High-tech R&D Program (2009AA03Z536).

REFERENCES