FRICTION OF C/C COMPOSITES: WEAR MECHANISMS AS A FUNCTION OF THE TEMPERATURE IN THE CONTACT

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Abstract

C/C composite disc brakes demonstrate their advantages in aeronautical braking due to their performances maintained at high temperature. Here is reported the tribological behaviour of two composites (graphitized and non-graphitized) tested on a laboratory pin-on-disc tribometer. In order to better understand the friction and wear mechanisms, experiments corresponding to cold taxiing (150°C), heat taxiing (450°C) and intermediate ones were performed. It results that both friction coefficient and wear rate decrease as the temperature in the contact increases, they are always higher in graphitized materials. Detailed characterizations (OM, SEM, profilometry) of the worn surfaces (3rd body trapped in the porosities, scratches, pyrocarbon and fiber damages, …) and of the ejected 3rd body particles (XRD, OM, TEM) allow to identify two wear mechanisms. They resultof the particle detachment in relation with the flow source of 3rd body formed during friction. At low temperature, a "catastrophic" wear is dominant due to source and wear flows relatively high, surfaces after friction in majority damaged and formation of large size 3rd body particles. At high temperature, a "soft" wear is dominant, characterized by a lower wear speed, a "polished" aspect of the surfaces and the detachment of small size particles. Explanations of the phenomenon at the origin of these mechanisms are given.

Keywords: *carbon/carbon composites, mass spectroscopy, oxidation, tribological properties, wear*

Introduction

The carbon/carbon (C/C) composites have a low density and preserve their mechanical and thermal properties at very high temperature. Therefore, they have become the best candidate as aeronautical brake disk material, being efficient in the different types of aircraft braking operations. To assure braking effectiveness, comfort and transportation safety, a stable friction coefficient is required, combined with a low wear rate to reduce costs.

In order to progress in the understanding of the friction and wear mechanisms, our approach is to correlate the tribological data (friction coefficient, wear) to the analysis of the gas exchanges taking place in the contact during friction [Kasem et al., 2006] [Gouider et al., 2004], to the characterizing of the worn C/C composite surface [Rousseau et al., 2005] and also to the formation of 3rd body (particles detachment) [Berthier, 2005]. Our objective is to progress in the understanding of the wear mechanisms (mechanic and oxidation) of C/C composites during friction [Kasem et al., 2007].

For that, the friction experiments are performed at temperature ranging from 150 to 450°C, on a laboratory pin-on-disc tribometer equipped with a mass spectrometer.

Experimental

Materials

In this study the materials carbon/carbon PAN-CVI composite, SEPCARB™. They are made of 3D ex-PAN fiber mats densified with pyrocarbon (rough lamellar texture). One sample is treated at high temperature, i.e. over 2000°C after densification ("G" material), he other one is untreated (only densified)

"NG" material. For each friction experiment the pin and the disc are machined from the same carbon composite (G or NG).

Set-up: pin-on-disc tribometer equipped with a mass spectrometer

The friction experiments are performed on a laboratory pin-on-disc tribometer with vertical axis equipped with a mass spectrometer. This set-up allows analysing the gas exchanges taking place in the contact during friction. Two external heating (one for the pin the other for the disc) permit to impose and to maintain the chosen temperatures during friction (Figure 1).

Experiments are performed on two materials (G and NG) in air, under a relative humidity of about 50%. The experimental parameters are:

- Imposed temperatures corresponding to cold taxiing (150°C), heat taxiing (450°C) and intermediate ones (250 and 350°C),
- Linear sliding speed of 2 m/s at the center of the track friction
- Pressure equal to 0.5 MPa,
- Each friction experiment lasts about 30 min.

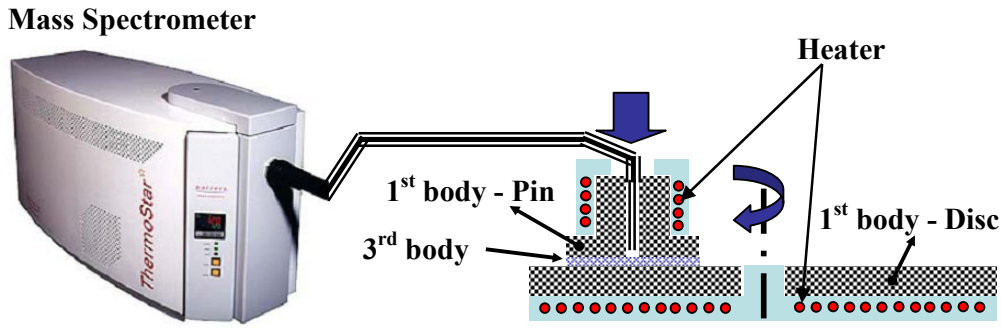

Figure 1. Experimental set-up: laboratory tribometer in pin/disc configuration coupled with a mass spectrometer.

During an experiment the following parameters are measured:

- The friction coefficient ***μ,***
- The wear rate (expressed in μm/min) estimated through the distance between the upper pin surface and a fix reference. It is calculated from the slope of the separation curve versus time. This "wear rate" takes only into account the thickness decrease, not the weight loss due to oxidation.
- The pin and the disc temperatures measured through thermocouples located at about 1.5mm from the contact.
- The gas exchanges taking place in the contact, detected by the mass spectrometer. The main information is brought from the signals coming from carbon dioxide, oxygen and water vapor (respectively masses 44, 32 and 18). Only the results concerning CO_2 concentration are reported here; its average concentration is directly measured through its partial pressure in the contact (expressed as an electric current in A unit).

During the friction tests and after the transition, the particles ejected from the contact are captured by means of an adhesive tape set parallel to the friction direction. The ejected particle sizes are determined from optical microscopy images (magnification ×200) using image analysis processing.

Worn surfaces are characterized by optical microscopy (OM, Leica DM IRM) and by scanning electron microscopy (SEM, Hitachi S4200). OM images are performed between crossed polarizers with addition of a retarder plate (λ plate).

Results and discussion

Friction transition and CO_2 production in the contact

At first, all friction experiments show at low temperature a weak wear regime called "non reactive". It is characterized by a weak friction coefficient (from 0.1 to 0.2), with no wear detected by our device. Then, as temperature increases, and after a brutal transition, a high wear regime called "reactive" occurs. It is characterized by a high friction coefficient (from 0.3 to 0.4) and high wear rate [Gouider et al., 2004]. The gas analyses in the contact bring to light that only the "reactive" regime is systematically accompanied by a CO_2 production and a O_2 consumption, with always a strong complementarity between CO_2 emission and O_2 consumption [Kasem et al., 2007].

Wear and CO_2 production in the contact during friction

The wear rate and the average CO_2 concentration in the contact are measured at temperatures ranging from 150 to 450°C. For both G and NG materials, as temperature increases, the friction coefficient decreases until 350°C, and then slightly increases at 450°C (Figure 2a). Concerning the wear rate, it continuously decreases as temperature increases (Figure 2b). Similar decrease is observed when considering CO_2 production in the contact. For a better understanding of the CO_2 dependence on wear, the ratio of CO_2 production over wear rate is reported versus temperature (Figure 3). In fact, this ratio represents the CO_2 production per worn thickness unit, i.e. per amount of 3rd body production. Paradoxically, this ratio increases as temperature increases. To understand this behavior, the origin of CO_2 production has to be determined. It comes from the carbon components able to oxidize in the contact, and during friction, both the first bodies and their debris may undergo oxidation. However, the debris, composed of particles, are more sensitive to oxygen than the first bodies.

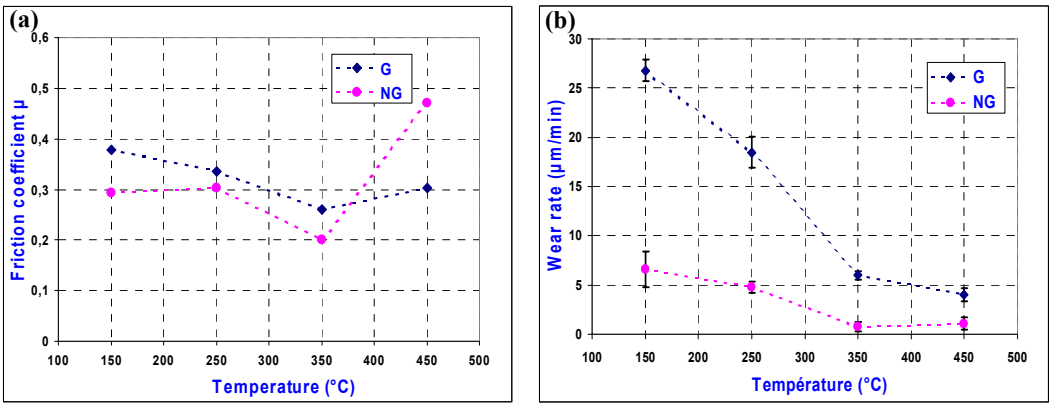

Figure2. Variation of (a) friction coefficient and (b) wear rate, as a function of the temperature in the contact

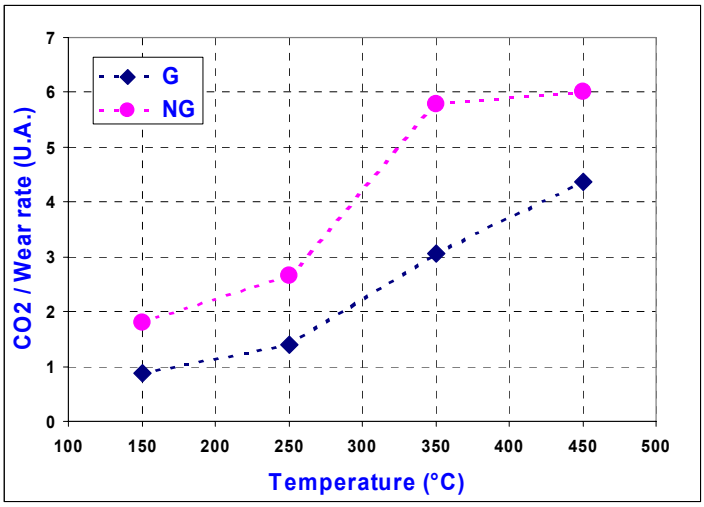

Figure 3. Ratio CO_2 concentration over wear rate as a function of the temperature in the contact

For a same material, the reactivity to oxidation of the particles closely depends on their size. In next section, we will see that, at low temperature the friction induces the formation of large size particles. This explains the low ratio of CO_2 production per wear unit. On the contrary, at high temperature small size particles are formed during friction, resulting in a higher ratio. Furthermore, as the NG material is not heat treated (only densified), its texture is less organized with more aromatic layer edges. Thus, particles resulting from its degradation during friction will also be less organized than those coming from G material.

Characterization of worn surfaces after friction

For both materials, after friction at 150°C (high wear rate), OM images show that the first body surfaces are in majority degraded at a large extend (Figure 4a). As seen in Figure 5a, the degradation consists of a lot of 3rd body {1}, torn off fibers {2} and degraded pyrocarbon {3}. Obviously, a high flow source (particles detachment) is dominating, and large size particles are observed in the contact.

Considering the two materials after friction at 450°C (weak wear rate), the surfaces are bright, as if they were "polished" (Figure 4b). They consist of mainly polished pyrocarbon {4}, smooth fibers {5}, small quantity of 3rd body {6} and a few torn off fibers (Figure 5b). The "polished" aspect of the surface results from small size detached particles. In this case, a low flow source is obviously dominating.

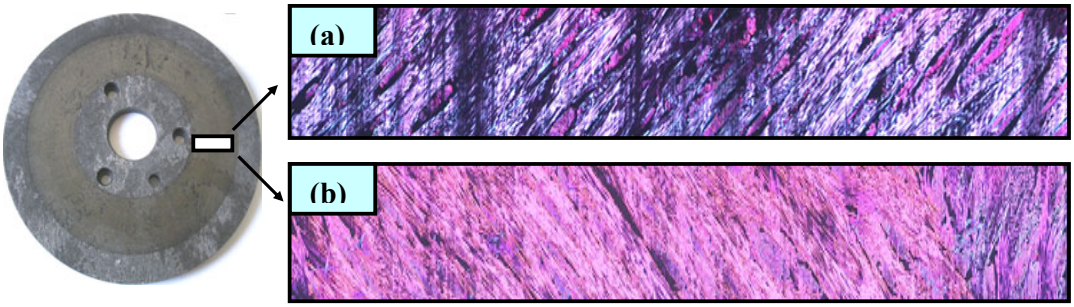

Figure 4. Characterization of the worn surfaces after friction at (a) 150°C and (b) 450°C for G material, OM images x50 between crossed polarizers with addition of a retarder plate

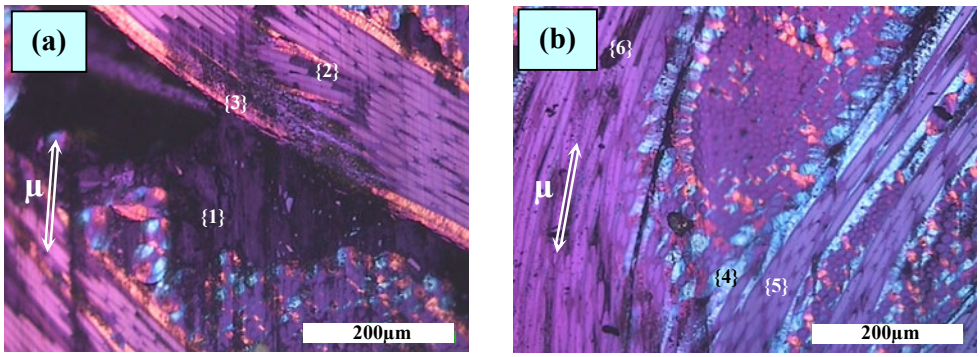

Figure 5. Worn surfaces of G material: OM images x200 between crossed polarizers with addition of a retarder plate. (a) After friction at 150°C (damaged surfaces); {1} a lot of 3rd body, {2} torn off fibers and {3} degraded pyrocarbon. (b) After friction at 450°C (smooth surface); {4} "polished" pyrocarbon, {5} "smooth" fibers and {6} small amount of 3rd body

Ejected particle size

The particles ejected from the contact are captured by the mean of an adhesive tape set parallel to the friction direction, and then they where characterized by optical microscopy. Using an image processing software, the particles are classified according to their individual surface. The results show that, during friction at 150°C (high wear rate), ejected particles and agglomerates have large size. It ranges from 80 to 200µm for G material and from 10 to 30µm for NG material (Figures 6a and c). On the contrary, during friction at 450°C (weak wear rate), their size is smaller, ranging from 10 to 50µm for the G material and from 5 to 15µm for NG material (Figures 6b and d). These results support the data presented in the previous paragraphs and give evidence that the detached particle size decreases with the temperature, contributing to the increase of oxidation reactivity.

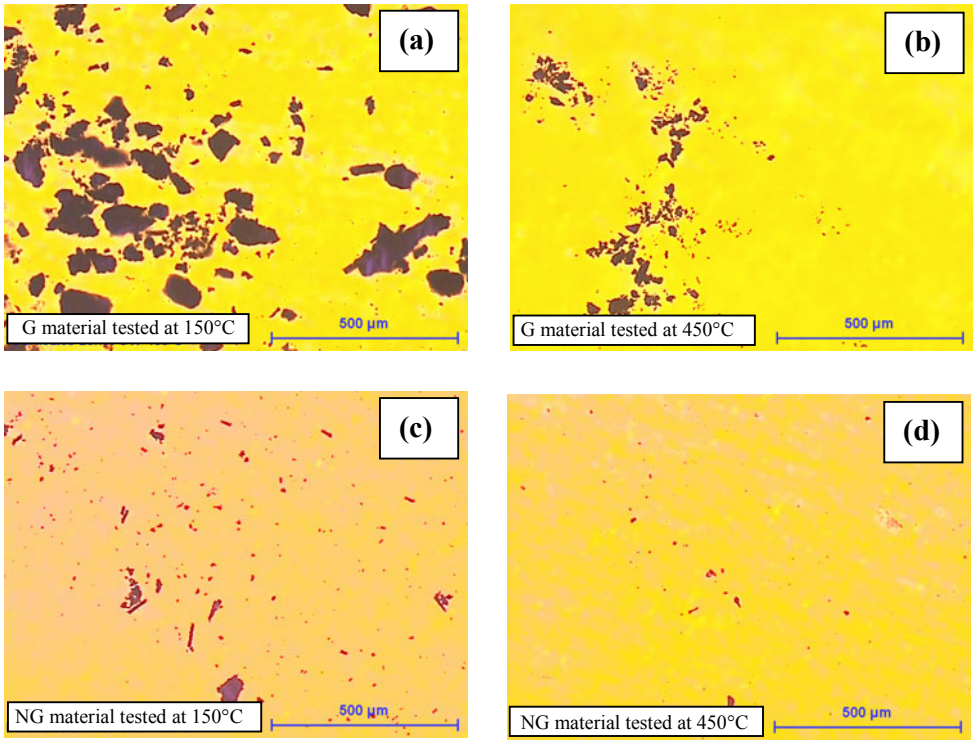

Figure 7. OM images of the ejected particles captured on an adhesive tape for the particle size measurement by image processing : G material during friction at (a) 150°C and (b) 450°C. NG material during friction at (c) 150°C and (d) 450°C

Conclusion

This study clearly shows a relationship between the CO_2 production analyzed in-situ in the contact during friction, the detached particle size, the surface damages and the source flow of 3rd body. The correlations obtained evidence that the quantity of CO_2 produced in the contact closely depends on the experiment temperature, the amount of third body formed during friction and also the size of the detached particles.

All our investigations allow to propose two wear mechanisms, depending on the temperature in the contact during friction:

- During friction at low temperature (150°C), the wear rate is particularly high due to detachment of large size particles, which is responsible for important surface damages. The large size of the particles limits their oxidation, thus, the ratio CO_2 concentration over wear unit is the weakest, especially for the G material.

- During friction at high temperature (450°C), the wear rate is rather low due to detachment of small size particles leading, in majority, to a polished aspect of the surfaces. The particles formed being smaller are more sensitive to oxidation. This explains the high ratio CO_2 concentration over wear unit, especially for NG material.

This study clearly highlights the necessity to couple mechanical and physicochemical approaches to progress in the understanding of the wear mechanisms during C/C composite friction.

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