MASS SPECTROMETRY ANALYSIS DURING C/C COMPOSITE FRICTION

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Introduction

The frictional properties and the wear mechanism of carbon-carbon (C/C) composites used as disc brakes need to be better understood to improve aircraft brake performances. In the literature, significant parameters governing the different friction and wear regimes have been identified such as the nature and structure of the composite, the type of the device (pin/disc, disc,/disc) and its scale (laboratory, industrial), the friction conditions (temperature, pressure, speed,…) and the gaseous environment (air, water vapor,…) [1-6].

The C/ C composite friction may adopt different regimes: in the case of a low regime the coefficient friction μ is about 0.1 –0.15, for a high regime μ values are in the range 0.3-0.5. In this study we try to precise the nature of these two regimes and to determine the parameters governing the transition from low to high friction regime, transition we are able to induce only in playing on the pin/disc device temperature. To better understand the mechanism governing friction and wear of contact surfaces, we equipped a pin–on-disc tribometer with a mass spectrometer in order to analyze the gas exchange in the contact. After each experiment, the disc and pin surfaces were also characterized by microscopies, to follow the formation and the evolution of the third body in the contact, as well as the degradation of the fibers and the pyrocarbon matrix after friction [7-10].

Experimental

The experiments were performed on a vertical pin-on-disc tribometer complemented with a mass spectrometer [7-9]. This allows analysis of the gas composition in the contact during friction. The disc diameter is 250 mm and the cylindrical pin one is 30 mm. The pin is axially pierced in order the spectrometer capillary (ϕ_i = 0.125 µm, length = 2 m) reaches the contact, gases being at atmospheric pressure. During a trial we measured:
- the tangential force undergone by the pin. Knowing the imposed force, the friction coefficient μ is deduced,
- the pin and disc temperatures,

- the wear, estimated through the distance between the upper pin surface and a fixed reference
- the gas exchanges taking place in the contact, detected by mass spectrometer. The main information will be drawn from the signals given by carbon dioxide, oxygen and water vapor (respectively mass 44, 32 and 18).

The rubbing trials are performed in air under a relative humidity ranging from 40-60 % and the friction parameters (speed, pressure, heating ramp, pin and disc temperatures) were chosen close to cold taxiing ones.

The trials start by putting on the disc rotation and the pin/disc heating. The desired temperature is maintained constant during 10min, heating is then stopped but rotation goes on during 1h or more.

After each experiment, the disc and the pin surfaces are characterized by optical microscopy (OM between crossed polarizers with a λ plate added), scanning electron microscopy (SEM) and atomic force microscopy (AFM).

Results and Discussion

Figure 1 exhibits typical curves of pin and disc temperatures, friction coefficient (μ), and wear versus time. The gas analyses in the contact are shown respectively as carbon dioxide, oxygen and water vapor partial pressures [7-9].

The comparison of all these data allowed us to better define the two friction regimes. We have seen that they are not only defined by the friction coefficient but by the existence or not of carbon oxidation reactions. These reactions express as:
- CO_2 and CO release, which perfectly coincides with the friction coefficient abrupt variations,
- O_2 consumption, nearly equal to CO_2 concentration increase, which also perfectly coincides with the friction coefficient abrupt variations
- H_2O consumption.

So during friction we can identified two regimes:
- a "non reactive regime" characterized by a low friction coefficient value, a non measurable wear, no CO_2 production
- a "reactive" regime characterized by a high friction coefficient value, a high wear rate, CO_2 release concomitant to O_2 consumption

The change from the non reactive regime to the reactive one is always abrupt and is a real transition function of the temperature. Figure 1, which shows such transition, is representative of reproducible experiments performed on graphitized C/C composites. In the case of non-graphitized C/C composites, reproducible conditions of the transition are not still well established. In such material some transitions are only transient and express as peaks, more or less short but with concomitant events on HTT, μ, CO_2, O_2, H_2O. For such materials we also observed "non reactive" regime: without CO_2 release, without O_2 consumption, without wear, but with slow friction coefficient increase up to μ= 0.4 without any transition.
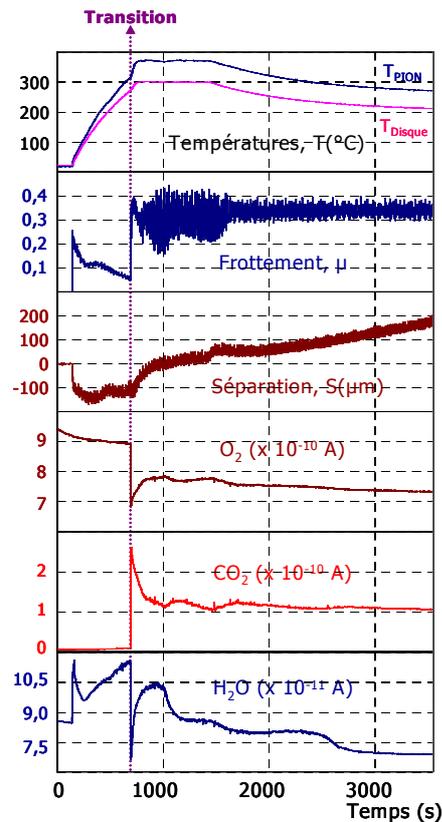
Figure 1. Correlation between pin and disc temperatures, friction coefficient, wear, CO_2, O_2, H_2O partial pressures versus time. These data are representative of a "reactive" friction regime.

Characterization of the pin and disc rubbing surfaces after "reactive" friction [10]:

In scanning electron microscopy, the penetration depth of the electron beam and the analysis depth strongly depend on the acceleration voltage. In order to limit the volume analysis to the outermost surface, the acceleration voltage must not be higher than 1 keV. The focus depth is apparently as great as the penetration depth and the image contrast is directly given by the surface roughness: rough surfaces appear bright, and smooth surfaces appear dark.

In optical microscopy, the penetration depth of the visible wavelength is no more than 0.6 µm and the focus depth is below 1µm for a ×1000 magnification, so OM images also give data of the outermost surface. When C/C composites are observed between crossed polarizers with addition of a λ plate, it is easy to identify its different components: fibers, pyrocarbon matrix, third body. Furthermore, the colors observed (magenta, yellow, black, blue) for the pyrocarbon matrix allow to know the orientation of the aromatic layers. This will be important data to consider relative to the friction direction. In the case of third body, it always appears magenta because it corresponds a poor organized carbon.

The complementarity of data given by OM and SEM images of the same area of C/C composite surfaces allowed to underline:

- the differentiated wear of the fiber and the matrix. Furthermore, in the case of pyrocarbon, wear is sensitive to aromatic layers orientation relative to friction direction,
- the absence of a continuous film covering the surface after the contact opening. Only the presence of third body in limited areas was detected.

Concerning wear mechanism, our first data obtained by atomic force microscopy (AFM) show that wear could be due to the removal of some very small particles, probably less than 10 nm in size.

An important data in this study, considering the tribometer used and our experimental conditions, is the fact that no continuous and cohesive third body film of wear debris is observed on the surfaces. The third body compacted powder is only located in cavities and porosity, either initially present in the C/C composite or created during friction, resulting of breakage and removal of fibers and pyrocarbon bits. It means that the third body has not a so important role in the friction phenomena and the speed accommodation is realized directly between the two first bodies, cause of their wear.

In the case of "non reactive" friction regime, the speed accommodation probably takes place in a layer of adsorbed water and oxygen complexes which plays a lubricant role and protects from wear. We still have to understand how such layer can survive and keep its lubricant properties up to 450°C.
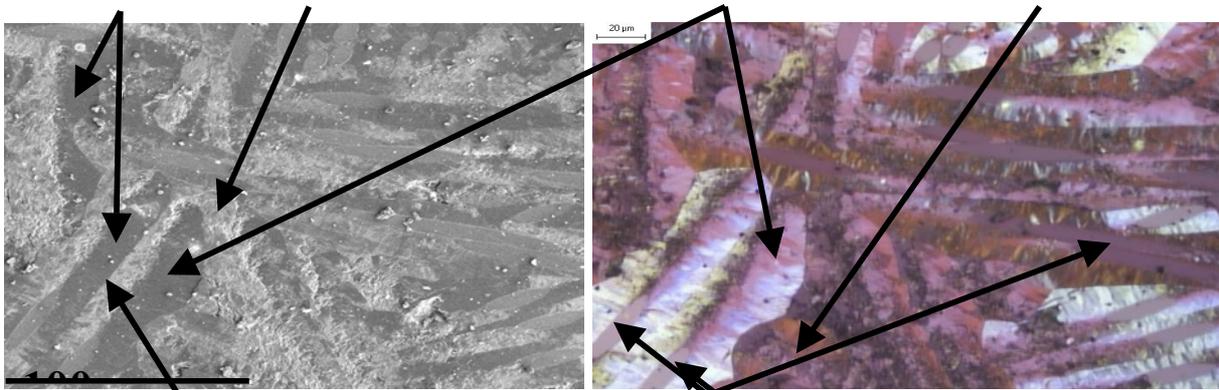

Figure 2. Confrontation of the data given by scanning electron microscopy and optical microscopy images

Conclusions

Thanks to a mass spectrometer coupled to a pin-on-disc tribometer working in air, we clearly evidenced the oxidation process occurring during C/C composite friction. From the gas analyses, the friction coefficient variations and wear, we defined two friction regimes. The "reactive" one where concomitant CO_2 release and O_2 consumption coincide perfectly with the transition of the friction coefficient, it is correlated with a high wear rate. Following the transition the oxidation process lasts as long as the friction

coefficient remains high. A "non reactive" regime characterized by a low friction coefficient value, a non measurable wear and no CO_2 production.

In the case of "reactive" friction regime, the confrontation of OM, SEM and AFM data allowed to better understand the way wear expresses on the fibers and the matrix and to underline the importance of aromatic layer orientation relative to friction direction. The microscopy data prove the absence of a continuous and cohesive film at the pin and disc surfaces after the contact opening. The third body only filled cavities and porosity, where it remains more or less compacted during friction. It means that during friction the speed accommodation is realized directly between the two first bodies.

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