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Tribological behavior of a silicon carbide/carbone dry contact

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1. Introduction

The development of new high-performance mechanical seals working in severe conditions requires higher material performances. Sintered silicon carbide (SSC), widely used as a hard mating material, is a potential candidate but its friction and wear properties need to be investigated in the scope of these new applications.

Silicon carbide offers good mechanical properties (high hardness, high Young modulus), good corrosion resistance and good thermal conductivity, that make it suitable for tribological applications in different atmosphere (in air, argon or vacuum) [1] and in dry or lubricated sliding [2].

Combined with a counter-face ring made of a softer carbon-graphite, the dry sliding of SSC can be sustained even under severe conditions of pressure and speed [3]. Graphite has been intensively studied in tribology since Bragg first described its lamellar structure. It has been thought during many years that graphite could act as a solid lubricant thanks to this structure. In fact, the environmental conditions strongly influence its tribological behavior [4]. The hardness of the ceramic facing the carbon seal has also an impact on its friction properties. A transfer layer of carbon is generally found on the ceramic surface [5].

In this study, a first experiment assesses the tribological behavior of SSC sliding against itself and three different carbon-graphite materials. Dry friction and ring-on-ring configuration are considered. A second test uses an infrared camera to estimate the temperature variations of a SiC/C couple during sliding, which determines relationship between displacement resistance and the heat generation.

2. Experimental procedure

2.1. Sample materials

As sample material for the primary ring, sintered silicon carbide, SiC BOOSTEC®, was used. The surface was polished to show an arithmetic roughness, R_a, of less than 0.1 µm.

Three different carbon-graphite materials from MERSEN were selected as counter-face rings: a carbon-graphite without impregnation (referred as C_1 material), a phenolic resin impregnated carbon-graphite (C_2), and an antimony impregnated carbon-graphite (C_3). In the same way as for the SiC, the surfaces were polished to obtain $R_a < 0.1$ µm.

The carbon ring dimensions were 38 mm outer diameter, 32 mm inner diameter and 13 mm thickness. For the SSC, it was 40 mm outer diameter and 30 mm inner diameter (Figure 1).

2.2. Tribometer

Experiments were performed under dry friction using a ring-on-ring tribometer. This tribometer rotates the mating ring against the counter-face ring loaded by means of dead weights. A torque and force sensor records continuously the applied normal force, F_N, and the resistant torque, C_r. These parameters give the friction coefficient, µ, through the equation:

$$\mu = \frac{3}{2} \times \frac{C_r}{F_N} \times \left(\frac{r_1{}^2 - r_2{}^2}{r_1{}^3 - r_2{}^3}\right) \qquad (1)$$

where r_1 and r_2 are respectively the outer and inner radii of the samples.

A sound level sensor (Pulsar 94) was used to correlate the sound and the friction behavior. Tribological tests were run at room temperature, at a constant sliding speed of 0.5 m/s under 0.1 MPa contact pressure on a contact area of 333 mm^2.

Experiments were conducted for two hours (total sliding distance of 3600 m) inside a tighten enclosure. Four pairs of materials were tested: a SiC/SiC couple and three SiC/C couples using the three supplied carbons C_1, C_2 and C_3.

A complementary experiment was reduced to 20 minutes (total sliding distance of 600 m) and tests were performed on the SiC/C_3 pair only. For thermal acquisitions, we used a FLIR Titanium SC7000 retrofitted camera with InSb sensors.

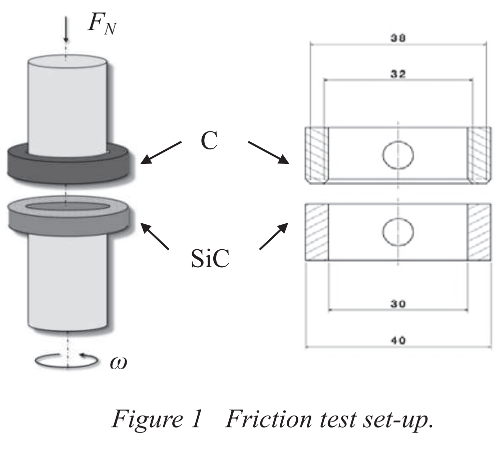

Figure 1 Friction test set-up.

2.3. Characterization methods

The samples were characterized before and after wear tests using different techniques to determine the wear mechanism of each material couple:

- Wear loss was measured by weighting the samples using an accurate scale (R1809, Sartorius Research),
- Topography and surface profiles of the worn ring specimens were determined using a 3D optical profilometer (Wyko NT1100, Veeco),
- The surfaces of tested samples were all examined by optical microscopy as well as scanning electron microscopy (SEM-FEG 7000F, Jeol),
- Elemental chemistry of the wear surfaces was finally analyzed by Energy Dispersive X-Ray Spectroscopy (EDX).

3. Results

3.1. Main series of experiments

3.1.1 Friction coefficient and sound level

The coefficient of friction (COF) was calculated from the normal force and the torque values with Equation 1, at 0.1 seconds intervals.

During the SiC/SiC contact, a high damage of the upper sample occurred and a COF of about 0.6 was recorded. The test was stopped after the first 100 s of sliding.

For the three SiC/C tribo-tests, COF and sound level as a function of time are shown in Figure 2. After a stationary period of 400 s, the COF of carbon sample without impregnation keeps oscillating between 0 and 0.4 with a mean value of 0.25 (Figure 2a). The sound level follows the same behavior and shows values between 73 and 100 dB with a mean value of 81 dB.

With a phenolic resin impregnation (Figure 2b), the oscillation frequency of the COF is reduced and the sound level seems to stabilize at the end of the experiment at a value of 74 dB. The mean value of the COF is about 0.3.

The antimony impregnation leads to the best results in terms of stability and noise level (Figure 2c). The value of COF, after a short running-in period of 500 s, remains stable around 0.3. The sound level exhibits the same evolution and stays quasi constant all along the test, at 75 dB. In the middle of the test, between 2300 and 4600 s, the sound level was perturbed and a slight increase in the COF was observed.

Sudden changes in the COF are related to a stick-slip phenomenon that occurs during friction. This is also highlighted by the noise level study.

3.1.2 Wear mechanism

The SiC/SiC test was found to be destructive for the upper ring. Mechanical overstresses on the edge of this sample induced a first cracking. Additionally, a high amount of debris was created by a chipping process. On the other hand, with SiC/C contacts, the softer carbon samples have allowed pursuing the dry sliding without major deterioration.

Again, the change in carbon impregnations leads to notable differences. The antimony impregnated carbon had the lowest weight loss while non-impregnated carbon got the highest one (Figure 3). The same weight loss was measured for each SiC sample.

Wear particles were collected after the tests. While the SiC/SiC pair created chips particles, the SiC/C couples produced spherical wear particles, rounded mechanically, with diameters ranging from 50 µm up to 500 µm (Figure 4).

In the three SiC/C experiments, a tribological film appeared on both surfaces. The coefficient of friction and the wear are very sensitive to the formation of this third body and its circulation inside the contact. Some part or the totality of this film can be pulled out, increasing both wear and COF.

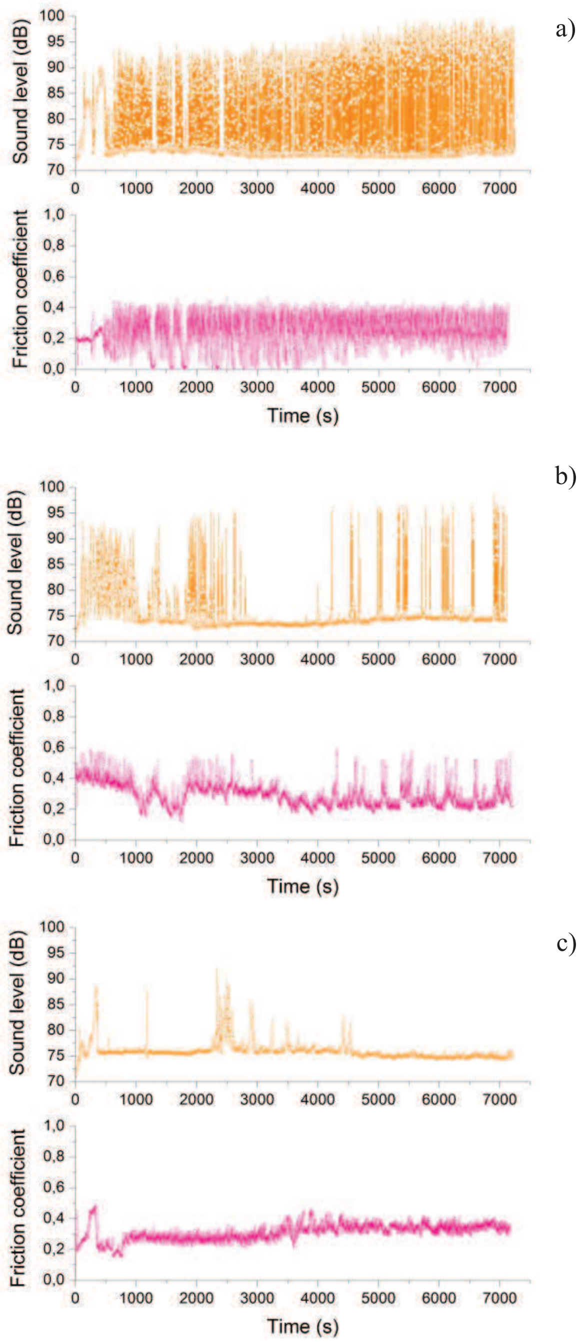

Figure 2 Friction coefficient and sound level for SiC/C_1 (a), SiC/C_2 (b) and SiC/C_3 (c) couples.

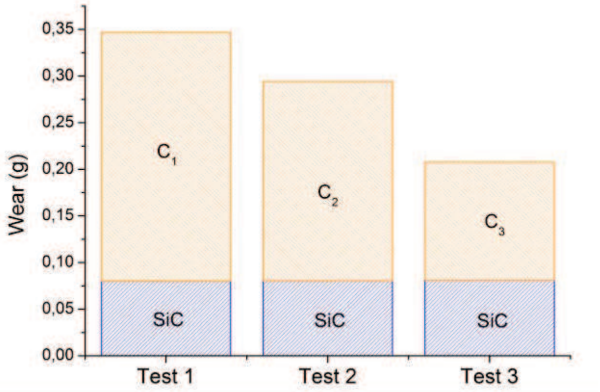

Figure 3 Wear loss.

EDX analyses of tribofilms show a carbon content higher than 85 wt.% and 10 wt.% for the oxygen, on both SiC and C surfaces. The carbon materials participate mainly to the formation of the third body.

Even if the tendency is the same for the three tests, differences in the wear mechanism behavior were also noticed.

In the case of the non-impregnated carbon, this film does not adhere to the SiC surface. Thus, it keeps on destructing, increasing the wear loss of the carbon sample.

With impregnations (C_2 and C_3), the film presents a better adhesion, leading to a decrease of wear loss.

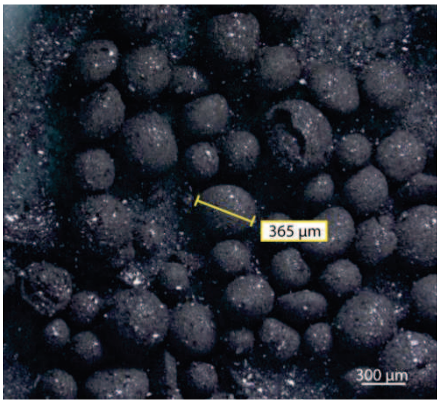

Figure 4 Wear particles of SiC/C tribo-tests.

3.2. Complementary experiment

For this test, the focus was put on the SiC/C_3 pair whose tribological performances seemed promising after the first series of experiments.

A tribo-test was run for 20 minutes on this pair of materials and the temperature was assessed by the infrared camera with a thermal resolution of 20 mK. The maximum acquisition frequency (150 Hz for full frame) and a uniform and constant value of emissivity corresponding to carbon (0.98) have been considered.

Moreover, a data processing after the acquisition phase provides the temperature variations fields $\Delta T = T - T_0$, with T the temperature field at time t and T_0 the temperature field in the initial state $t = 0$.

3.2.1 Regions of interest

Seven regions of interest were analysed (Figure 5):

- Zone 1 includes the two samples at the contact area;
- Zone 2 records the temperature of the carbon contact area;
- Line 3 studies the temperature profile along the two samples close to the contact area;
- Zone 4 and line 7 follow the temperature of the alumina part of the lower shaft;
- Zone 5 and line 6 follow the temperature of the alumina part of the upper shaft.

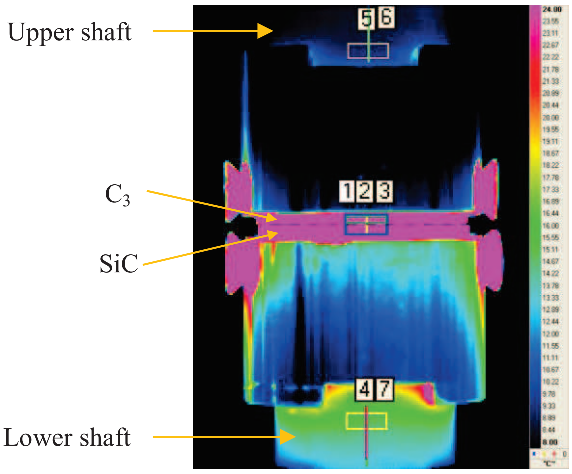

Figure 5 Regions of thermal interest for the complementary experiment.

3.2.2 General heat field

Figure 6 shows the average temperature rise (relative scale) for the different zones.

Temperature in zones 1 and 2 stabilizes after the initial 400 s with a rise of around 35 K.

For zones 4 and 5, heat propagation starts after 80 s of test and evolves more slowly than on the contact area. The temperature rise in zone 4 becomes stable at 35 K above room temperature, just before the end of the test (1160 s). The temperature in zone 5 continues to rise all along the test.

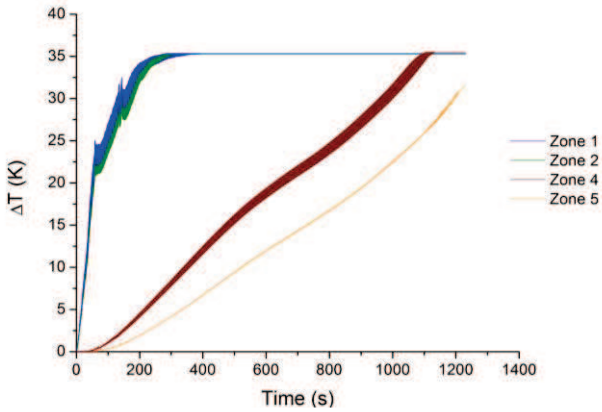

Figure 6 Temperature rise of the zones 1, 2, 4 and 5.

3.2.3 Contact area

The line 3 follows the temperature evolution through the C_3 sample, the contact point and the SiC sample. The profile of the temperature along that line is shown on Figure 7.

The maximum value is reached at the contact point (0 mm) and the temperature evolves until the saturation. Furthermore, the temperature increases with different speed for the samples. The SiC reaches the saturation at 200 s and C_3 at 300 s.

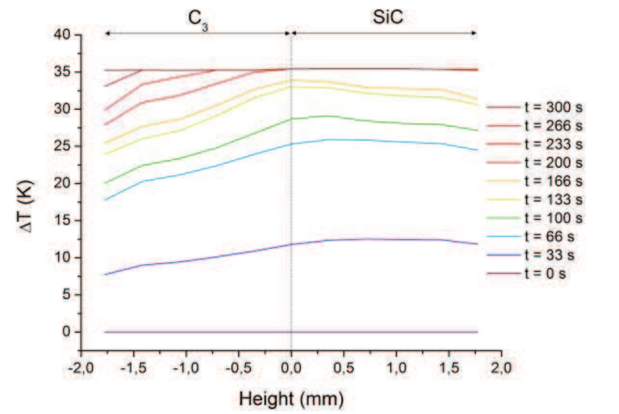

Figure 7 Temperature profile of the line 3.

4. Discussion

4.1. Main series of experiments

From the main series of experiments, a wear mechanism can be proposed for the SiC/C dry sliding. During a running-in period, an accommodation is observed between materials showing a higher wear loss. Then, the third body composed by superficial films and interfacial film is created, mainly by the wear of the carbon sample.

The impregnation of the carbon material plays also a predominant role in the tribological performances of the pair. Phenolic resin or antimony impregnations reduce the stick-slip phenomenon inside the contact. While a non-impregnated carbon shows a higher destruction rate (resulting in high variations of the COF), the antimony impregnated carbon creates a more stable third body, willing to remain in the contact area and to act as a solid lubricant. A better resistance to wear is achieved with these materials.

Accommodation of the contact reduces also mechanical vibrations and thus, noise emission. The antimony impregnation provides then the best behavior in terms of friction coefficient, wear and noise emission.

4.2. Complementary experiment

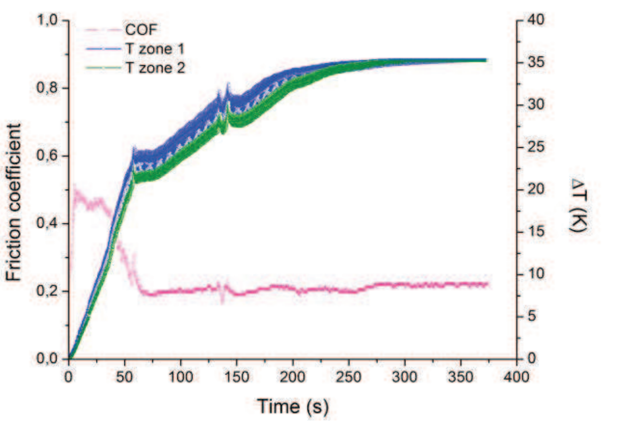

Figure 8 Friction coefficient and temperature elevation of the SiC/C_3 couple.

The temperature rise of the samples can be correlated to the friction coefficient. As shown in Figure 8, peaks in temperature correspond to peaks in the friction coefficient. Moreover, at the beginning of the test, when COF is high, the slope of temperature is high. As soon as the friction coefficient stabilizes, the slope decreases until the stabilization.

Mechanical friction dissipates energy in the form of heat and acoustic emissions. When the increase in temperature of the samples is stopped, other parts of tribometer carry on warming up. A difference is seen between the upper shaft fixed to the C_3 sample and the lower part fixed to the SiC. The temperature rise of the lower part is quicker and stabilizes before the end of the test. This is due to the difference of thermal conductivities of our materials.

Note finally that infrared recorded videos show also ejected wear debris whose diameters were superior to 350 µm (pixel size), in good agreement with the wear debris found outside the contact in the main series of experiments.

5. Conclusions

Tribological tests were run at room temperature on different SiC/C couples. These materials present different tribological behavior in terms of friction coefficient and wear.

This study showed that the ability for the carbon material to create a stable third body inside the contact controls the performances of the couple.

In particular, using an antimony impregnated carbon reduces the stick-slip phenomenon and thus, the noise emission and the wear loss.

The thermal analysis on the antimony impregnated carbon sliding against SiC shows relations between the temperature rise and the friction coefficient change. The temperature of the system was found to stabilize after an elevation of 35 K.

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