# Tribological and Interfacial Phenomena in Al<sub>2</sub>O<sub>3</sub>/SiC and SiC/SiC Couples at High Temperature

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## Abstract

The dry-friction behaviour of two ceramic-to-ceramic tribological couples is shown to be very sensitive to temperature. This is associated with modifications in the rheological properties of the wear debris which form the 'third body'. One 'velocity accommodation mechanism' is mainly involved here. It is the formation of small rolls, composed of agglomerated debris. These rolls act as minute roller-bearings, which leads to a decrease in the coefficient of friction, and therefore a reduction of the cracking of the wear track. Rolls develop at medium temperature only, and their morphology depends on various parameters, in particular the loading force and the sliding distance.

Trockenreibungsverhalten von zwei ver-Das schiedenen tribologischen Keramik-auf-Keramik-Paaren ist, wie gezeigt wird, sehr empfindlich in Bezug auf die Temperatur. Das hängt mit Änderungen der rheologischen Eigenschaften der Abriebspartikel zusammen, die einen 'dritten Körper' bilden. Ein 'Geschwindigkeitsanpassungsmechanismus' ist hierfür hauptsächlich verantwortlich. Es werden kleine Rollen gebildet, die aus agglomeriertem Abrieb zusammengesetzt sind. Diese Rollen wirken für kurze Zeit als Rollenlager und vermindern so den Reibungskoeffizienten und damit auch die Rißbildung in der Abriebspur. Diese Rollenkörper bilden sich nur bei mittleren Temperaturen und ihre Morphologie ist von verschiedenen Parametern abhängig, insbesondere von der aufgebrachten Kraft und der Gleitweglänge.

On montre que la température influence notablement le comportement en frottement à sec de deux couples tribologiques céramique-céramique. Ceci est dû à des modifications des propriétés rhéologiques des débris provenant de l'usure et qui forment le 'troisième corps'. On fait surtout intervenir un 'mécanisme d'adaptation des vitesses' mettant en jeu la formation de petits rouleaux formés par agglomération des débris. Ces rouleaux se comportent comme de minuscules roulements à aiguilles, conduisant à une diminution du coefficient de frottement et par conséquent à une réduction du craquèlement du sillon. Les rouleaux ne se développent qu'à des températures intermédiaires et leur morphologie dépend de divers paramètres, en particulier de la charge et de la distance de glissement.

#### Introduction

Ceramic materials offer great potential in tribology, and ceramic parts are more and more used for wear and friction applications. Ceramics offer the advantages of high hardness, excellent high-temperature resistance and low chemical reactivity. However, brittleness is the main concern. In some tribological configurations, brittleness can even result in cracking of the wear track.<sup>1</sup>

The present work was devoted to the study of the 'velocity accommodation mechanisms' (VAMs). VAMs were introduced by Berthier *et al.*<sup>2</sup> to explain how the interfacial properties in a tribological couple are controlled by the characteristics of the

'first' bodies as well as the rheology of the 'third body' made up of wear debris. Twenty different VAMs are identified, one of them being the formation of small rolls, developed inside the third body due to the loose agglomeration of debris extracted from the worn surfaces. The rheological properties of such rolls are demonstrated to play a prominent role in the tribological behaviour of two ceramic-to-ceramic couples.

#### **Materials and Methods**

The tribological tests were carried out using a laboratory-made, high-temperature tribometer,<sup>3</sup> with the configuration of a fixed ball sliding on a rotating disc. Two materials couples were studied: a heterogeneous one (Al<sub>2</sub>O<sub>3</sub> ball/SiC disc) and a homogeneous one (SiC ball/SiC disc). Table 1 gives the main characteristics of materials. Al<sub>2</sub>O<sub>3</sub> was a 95% pure, 95% dense grade with 2.5% SiO<sub>2</sub> and 1.7% MgO as main additives. SiC was a sintered, 98.5% pure, 96% dense  $\alpha$ -grade. Temperature could be varied from 20 to 1000°C. Atmosphere was controlled. The usual tribological conditions were a

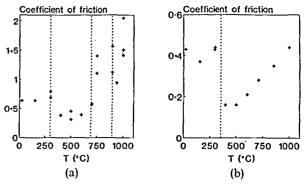


Fig. 1. Coefficient of friction after a sliding distance of 100 m versus temperature: (a) Al<sub>2</sub>O<sub>3</sub> ball/SiC disc; (b) SiC ball/SiC disc.

loading force of 4 N, a sliding distance of 100 m and a sliding velocity of 0.1 m/s, but some additional tests were carried out at 500°C using a higher loading force of 10 N and various sliding distances (12, 25, 500 and 1000 m). A faster sliding velocity (1 m/s) was chosen in special cases.

## Results

The evolution of the coefficient of friction after a sliding of 100 m is shown in Fig. 1(a) for the  $Al_2O_3/$ 

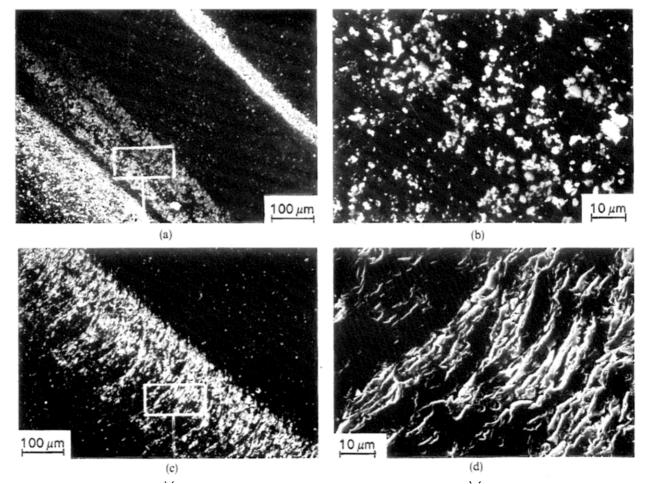


Fig. 2. Al<sub>2</sub>O<sub>3</sub> ball/SiC disc at 300°C: (a) and (b) aggregated debris; (c) and (d) cracking of the wear track on the SiC disc.

Material	Young's modulus (GPa)	Poisson's ratio	Apparent density (g/cm³)
SiC	420	0.16	3.15
$Al_2O_3$	313	0.22	3.71

Table 1. Main characteristics of Al<sub>2</sub>O<sub>3</sub> (from CICE, Montreuil, France) and SiC (from Céramiques et Composites, Tarbes, France)

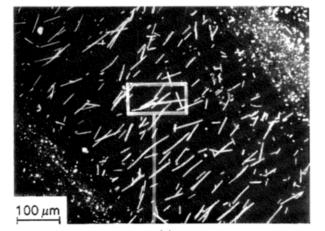
SiC couple and in Fig. 1(b) for the SiC/SiC couple. In both cases there is a rapid fall in the coefficient of friction when temperature increases over 400°C. From 20 to 400°C, f remains at a sensibly constant value (0.65 for Al<sub>2</sub>O<sub>3</sub>/SiC and 0.45 for SiC/SiC). For T > 400°C, f drops to 0.4 for the former couple and 0.16 for the latter one.

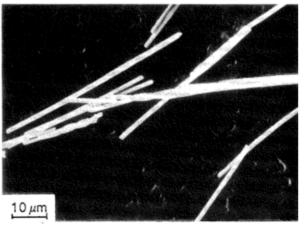
The drop of the coefficient of friction is associated with changes in the morphology of the third body at the sliding interface:

- (1) From room temperature to about 700°C, the two couples show similar behaviour, with two different domains:
  - (a) From room temperature to 400°C, the layer of debris is constituted of aggregates of

various sizes, with no preferential orientation (Fig. 2(a) and (b)). These aggregated debris do not protect the sliding surfaces against further damage. The coefficient of friction is high, which leads to high stresses inside the first bodies, and therefore helps to develop cracks in the wear track. Figure 2(c) and (d) shows such cracks, observed after a chemical attack.

- (b) From 400 to 700°C, the debris agglomerate and organize to form tribological rolls, which arrange themselves perpendicularly to the sliding movement of the ball (Fig. 3(a) and (b)). These rolls act as minute roller-bearings, which decreases the coefficient of friction, hence preventing the formation of cracks (Fig. 3(c) and (d)).
- (2) At temperatures over 700°C, the two couples under study exhibit different behaviour:
  - (a) For the heterogeneous Al<sub>2</sub>O<sub>3</sub>/SiC couple at temperatures from 700 to 900°C, the formation of rolls is accompanied by some material transfers from the Al<sub>2</sub>O<sub>3</sub> ball onto the SiC disc. The transferred debris are strongly





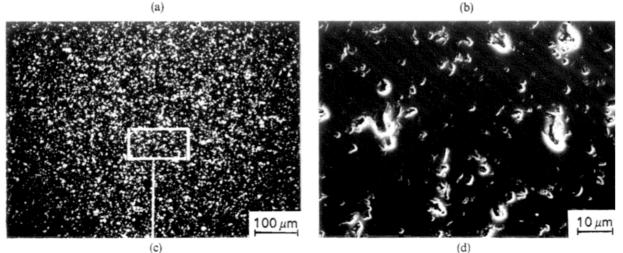
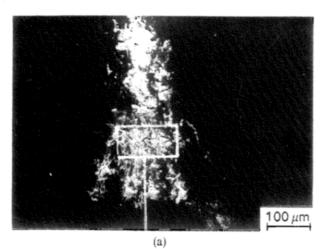


Fig. 3. Al<sub>2</sub>O<sub>3</sub> ball/SiC disc at 500°C: (a) and (b) tribological rolls; (c) and (d) uncracked wear track on the SiC disc.



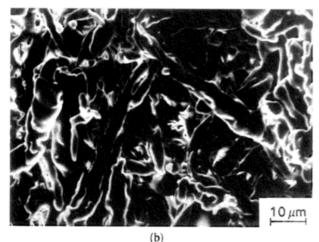
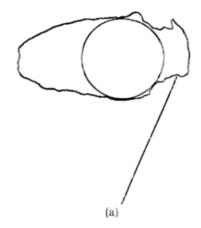


Fig. 4. Al<sub>2</sub>O<sub>3</sub> ball/SiC disc at 1000°C: (a) and (b) agglomerated rolls.

stuck onto the wear track, which leads to a noticeable increase in the coefficient of friction (f > 1), and therefore to the development of cracks in the disc. At the highest temperatures which were studied (from 900 to 1000°C), the rolls become aggregated by a viscous phase, and they are pushed in the front of the ball by a prow effect (Fig. 4(a) and (b)). Such aggregated rolls do not continue to act as roller-bearings, and the coefficient of friction exhibits a dramatic increase (up to f=2).

(b) For the homogeneous SiC/SiC couple, there is no material transfer. Around 700°C, f is at a low value of about 0·3. At higher temperatures, however, the surface of SiC oxidizes. A viscous, silica-rich phase develops, which prevents the formation of rolls. The wear debris are sintered together, and can pile up onto the front and the rear of the worn zone on the ball (Fig. 5(a) and (b)).

The prominent role played by the tribological rolls was studied in the frame of complementary



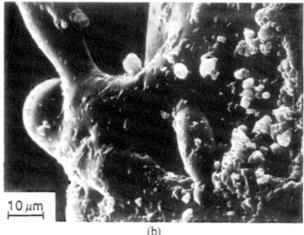


Fig. 5. SiC ball/SiC disc at 1000°C: (a) schematic of the location of sintered debris on the front and the rear of the worn zone of the ball; (b) micrograph of sintered debris.

tests, conducted at 500°C on the heterogeneous  $Al_2O_3/SiC$  couple. The tests were stopped after different sliding distances (x = 12, 25 and 100 m), and the samples were observed by SEM in order to study the formation of tribological rolls. Figure 6(a) shows the evolution of the coefficient of friction vs the sliding distance.

After a distance of 12 m, the elementary debris pack together to form small, rather elongated agglomerates, with a size ranging from 3 to  $10 \,\mu m$ . The agglomerates are randomly orientated (Fig. 6(b)), and the coefficient of friction begins to vary in an erratic manner. After a distance of 25 m, the agglomerates pack to form medium-size rolls (from 10 to 30  $\mu$ m) but there is still no preferred orientation (Fig. 6(c)). The coefficient of friction fluctuates in a wide range  $(\pm 0.15)$ , but its general trend is to drop. After 100 m, the sizes of rolls have increased to some hundreds of micrometres, with diameters of  $1-2 \mu m$ . A preferred orientation is now well marked, with very elongated rolls arranged perpendicularly to the sliding direction (Fig. 6(d)). This state corresponds to a minimum in the coefficient of friction (f < 0.45), with the lowest scattering.

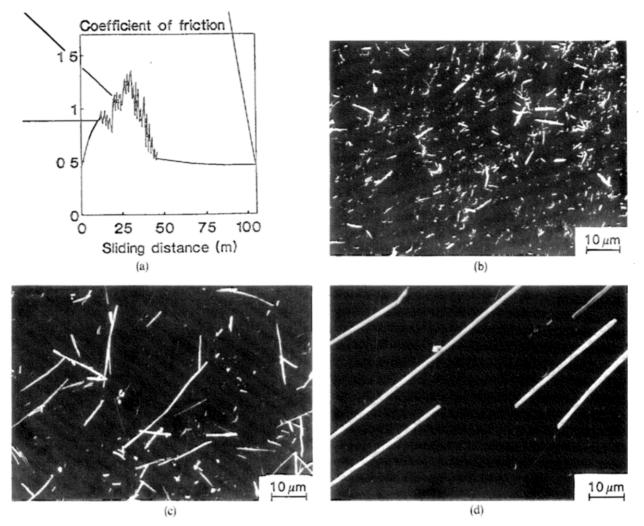


Fig. 6. Al<sub>2</sub>O<sub>3</sub> ball/SiC disc at 500°C: (a) coefficient of friction versus sliding distance; (b)-(d) micrographs showing the formation of rolls after (b) 12 m, (c) 25 m and (d) 100 m.

Additional tests were conducted at 500°C on the Al<sub>2</sub>O<sub>3</sub>/SiC couple. They demonstrated that the formation of rolls is not very sensitive to velocity (at least in the domain of 0.1 to 1 m/s) but that it is sensitive to load. An increase in the loading force from 4 to 10N results in a modification in the morphology of the layer of debris. Under a load of 10 N, the third body is constituted of a mixture of equiaxed, aggregated debris and of elongated rolls. Such a duplex morphology can also be observed at lower temperatures.<sup>4</sup> Finally, long-distance (500 and 1000 m) tests showed that the coefficient of friction remains at rather a small value ( $\approx 0.45$ ) but with some fluctuations. These fluctuations are associated with the presence of heterogeneities in the arrangement of rolls, which are now heaped up to form piles, organized along the wear track with a periodicity whose length is close to the diameter of the worn zone on the ball.

The rolls formed at 500°C are very flexible (Fig. 3(b)) and their 'dynamic' properties in the tribological interface seem very stable. However, their

'static' cohesion is very poor. For instance, they become disaggregated when wetted by liquids such as acetone. This suggests that the cohesion forces inside the rolls may be of Van der Waals or electrostatic origin but that there is no sintering of debris.

Rolls are not stable outside the temperature range where they were formed.<sup>5</sup> In the case of tests started at 500°C to develop rolls and then continued at room temperature, the rolls were quickly disaggregated, and the coefficient of friction increased again (from 0.45 to 0.60).

In the domain of medium temperatures, rolls develop in a similar manner for the  $Al_2O_3/SiC$  couple and the SiC/SiC one. As far as the phase composition is concerned, energy dispersive X-ray micro-analysis (Kevex) was carried out using a special window of  $0.25 \,\mu$ m (Quantum), which allows the detection of light elements, in particular carbon and oxygen. It indicated that silicon is fully oxidized into silica inside agglomerates having the shape of rolls, whereas it is partly oxidized into SiO<sub>2</sub> and

partly combined into the initial SiC compound inside the non-roll agglomerates.

#### Conclusions

The third-body approach was mainly developed to explain the tribological behaviour of metallic couples, which involve rather ductile materials. However, the present work demonstrates that this approach is also fully applicable to brittle ceramic materials, namely  $Al_2O_3$ /SiC and SiC/SiC couples.

The velocity accommodation mechanism which is dominant here is the formation of rolls. Rolls act as roller-bearings, which reduces the coefficient of friction, and therefore prevents the development of cracks in the first bodies. However, rolls are only observed at medium temperature, and for special conditions of loading force and sliding distance. A study is currently being conducted to elucidate the mechanisms which explain the formation and the stability of rolls. It can be thought that tribochemistry effects play a prominent role. Moreover, the grain-boundary segregations which exist in the 95% pure alumina could lead to low viscosity phases in the case of the Al<sub>2</sub>O<sub>3</sub>/SiC couple at high temperature. The point is to determine whether the domain of temperature where rolls are stable could be widened, in order to use ceramic parts showing a self-lubricating behaviour.

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