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Reciprocating sliding tribology of brake oil treated carbon fiber reinforced ceramic matrix composites

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Abstract: The effect of brake oil on sliding behavior of carbon/carbon (C/C) and carbon/carbon−silicon carbide (C/C−SiC) composites was investigated with the variation of laminate orientation and surface conformity. The partial and low conformity contacts with the normal and parallel orientations of laminates were considered. The normal load was varied from 50 to 90 N in a step of 10 N. The friction and wear behavior was investigated under reciprocating sliding conditions. The results showed that friction coefficient and wear loss of composites with normal orientation of laminates were larger as compared to those of composites with parallel orientation of laminates. C/C composites with normal orientation of laminates yielded the highest value of friction coefficient. Wear loss decreased by a maximum of 78%, and friction coefficient decreased by a maximum of 49% in low conformity contacts as compared to partial conformity contacts. The presence of brake oil reduced the adhesion tendency of compacted wear debris because the formation of friction film was difficult, and thus, friction behavior was affected. The wear debris retention between the contact surfaces due to confined area motion in reciprocating sliding depicted the tribological behavior.

Key words: reciprocating sliding tribology; C/C composites; C/C−SiC composites; friction coefficient; wear resistance; laminate orientation

1 Introduction

Carbon/carbon (C/C) composites exhibit unique properties such as high specific heat, low thermal expansion coefficient, high thermal shock resistance, low density, high specific strength, low ablation resistance, excellent biomedical compatibility, and high toughness [1−5]. Due to these properties, C/C composites are the potential candidates for their use in the field of aeronautics and biomedical applications [2,6]. In contrast to metals and ceramics which show a decrease in strength, C/C composites show an increase in strength with increase in temperature [7]. C/C composites are very much sensitive to the humidity [8,9]. Their friction coefficient degrades greatly in humid environment [8]. Adsorbed species on the surface of C/C composites greatly affect their tribological behavior under low energy braking conditions. However, under high energy conditions, adsorbed species evaporate.

Poor oxidation and wear resistance, and instability towards friction coefficient limit the applications of C/C composites. C/C−SiC composites prepared by the incorporation of silicon carbide (SiC) in C/C preform exhibit the properties of C/C composites with further enhancement in the resistance to oxidation, wear, and humidity [10]. C/C−SiC composites also exhibit stable friction coefficient and long service life [9] which make C/C−SiC dual matrix composites better for their use in advanced friction systems, as compared to C/C composites [11−15]. The density of C/C−SiC composites is more as compared to that of C/C composites [16]. Density is a very crucial parameter in designing thermostructural composites in the weight sensitive applications [17] because C/C composites are favorable in some parts of weight sensitive structures.

Although the wear and friction behavior of C/C and C/C−SiC composites has been investigated extensively [15,18,19], most of the studies were carried out in the dry environment. Sometimes damage in the hydraulic line (e.g., brake line) may lead to spilling of oil on the brake discs. Thus, the friction discs of C/C and

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C/C−SiC composites employed in advanced braking system are prone to oil and humidity under service conditions. Some researchers have tried to monitor the friction response of C/C and C/C−SiC composites in different environments such as seawater and acidic environments [8,16,17,20]. The studies revealed that the external environment may change the friction and wear mechanisms during testing and can severely affect the tribological performance.

The sliding behavior of fibrous composites varies with the orientation of fibers with respect to the sliding direction of the counter surface [21−23]. Relations proposed by SMERDOVA et al [24] show that the area fractions of different constituents on the contact surface depict the wear and friction responses. But several other factors are also involved while investigating a material experimentally. Thus, it is difficult to generalize the orientation effect of fibers on tribological behavior of composites. C/C and C/C−SiC composites have been investigated in self-mated pair with fully conformal contacts and parallel orientation of laminates [16,25−27]. Sliding behavior under partial and low conformity contacts has been investigated very recently but in a dry environment [28].

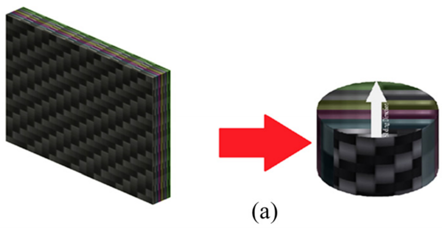
The tribological behavior under reciprocating sliding is different from that under unidirectional sliding [29−32]. Sudden transition in friction regime is not experienced in unidirectional sliding, whereas transition occurs at the beginning of each stroke in reciprocating sliding [33] which changes the material behavior in reciprocating conditions. C/C and C/C−SiC composites have been investigated in dry, wet and acidic environments [16,20,26,27,34,35] but the effect of brake oil on their tribological behavior has not been investigated yet. Therefore, in the present study, the research was focused on investigating the wear and friction behavior of C/C and C/C–SiC composites in brake oil environment with the help of reciprocating friction monitor by varying the laminate orientation and surface conformity.

2 Experimental

2.1 Materials

The details of material, fabrication process, sample preparation, and wear testing have already been explained in our previous article [28]. The description of the materials used for the present study is given in brief here. The precursor for carbon matrix in C/C composites was the phenolic resin which was reinforced by two-dimensional (2D) high tenacity carbon fiber woven fabrics. The resulted carbon fiber reinforced polymer (CFRP) composite was carbonized at 1000 °C. Repeated impregnation of phenolic resin in vacuum was performed to densify the carbonized CFRP which was further accompanied by carbonization. Final heat treatment was done at 1500 °C to obtain C/C composites. The resulted C/C composites contained 50% carbon fiber and 50% carbon matrix by volume. The density of the obtained C/C composites was 1.8 g/cm^3. These C/C composites were used as the preform for C/C−SiC composites which were fabricated through liquid silicon infiltration (LSI) route. Siliconization of C/C preform was performed by heating it with silicon at a temperature of 1650 °C. The resulted C/C−SiC composite contained 38% silicon carbide and 2% free silicon by volume. The density of C/C−SiC composites was 1.85 g/cm^3.

Partial (pin on plate) and low conformity contacts (ball on plate) were considered for wear testing. For partial conformity contacts (pin on plate), samples were prepared in the form of cylindrical pins having 9 mm in diameter for normal (designated as C/C normal and C/C−SiC normal) and parallel (designated as C/C parallel and C/C−SiC parallel) orientations of laminates. Figure 1 shows a schematic diagram of composites with the normal and parallel orientations of laminates. SEM images depicting the surfaces of as-prepared composites with the normal and parallel orientations of laminates are shown in Fig. 2.


(a)

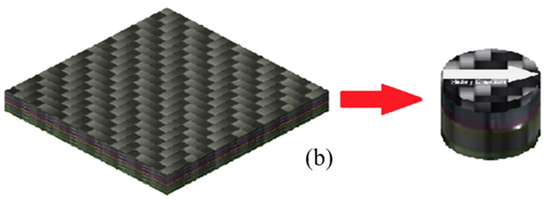

(b)

Fig. 1 Composite pins showing orientations of laminates: (a) Normal; (b) Parallel

Samples were prepared in the form of square plates (designated as C/C plate and C/C−SiC plate) having 25 mm in side and 4 mm in thickness for low conformity contacts (ball on plate).

2.2 Brake oil absorption

Prior to wear testing, the prepared samples were immersed in brake oil for almost 5 h. The brake oil used was “Servo brake fluid HD” which met the DOT 3, SAE J1703, FMVSS No. 166, and IS 8654−2001 specifications. Brake oil was absorbed by C/C and C/C−SiC composites. Samples were weighed before and

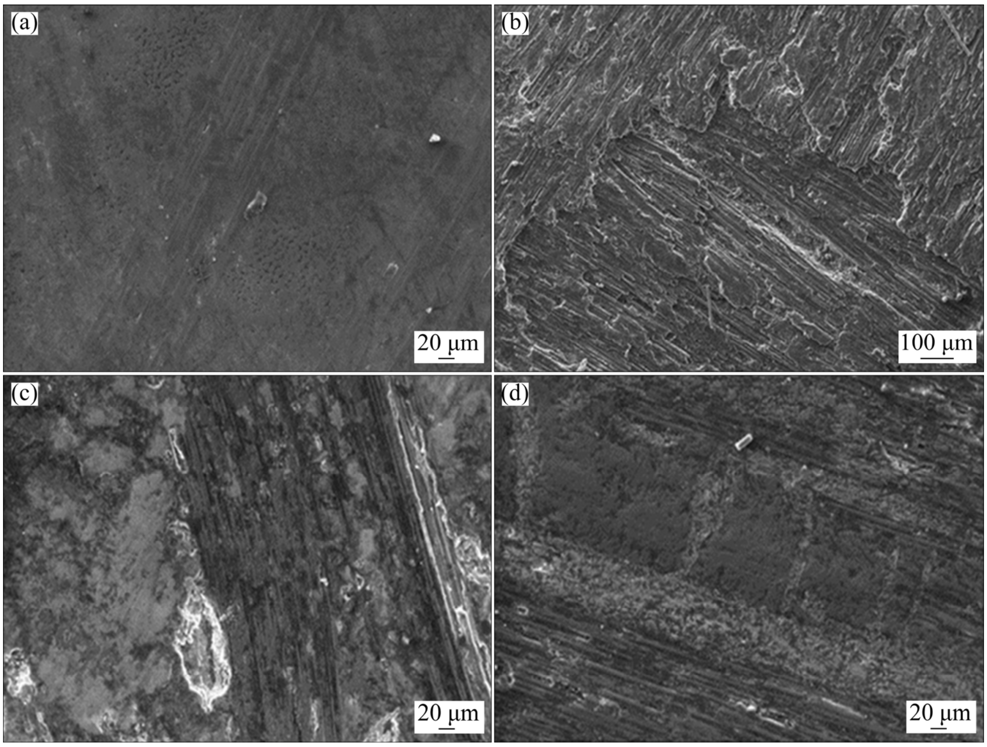


Fig. 2 SEM images showing composites with normal and parallel orientations of laminates: (a) C/C, normal; (b) C/C, parallel; (c) C/C−SiC, normal; (d) C/C−SiC, parallel

after immersing in brake oil. After taking out samples from brake oil, samples were cleaned with jute cloth and weighed using a weighing machine (DENVER INSTRUMENT, SI−234). The average mass gain per unit surface area was calculated by an increase in mass fraction as follows:

$$\Delta m = \frac{m_f - m_i}{m_i} \times 100\% \quad (1)$$

where Δm is the increase in mass fraction, m_f is the mass of the sample after taking it out from the brake oil, and m_i is the mass of the sample before putting it in brake oil.

It can be observed from Fig. 3 that C/C composites absorbed more oil as compared to C/C−SiC composites due to more open porosity of C/C composites. Composites with the parallel orientation of laminates contained more surface pores. Thus, composites with the normal orientation of laminates absorbed less oil as compared to composites with the parallel orientation of laminates.

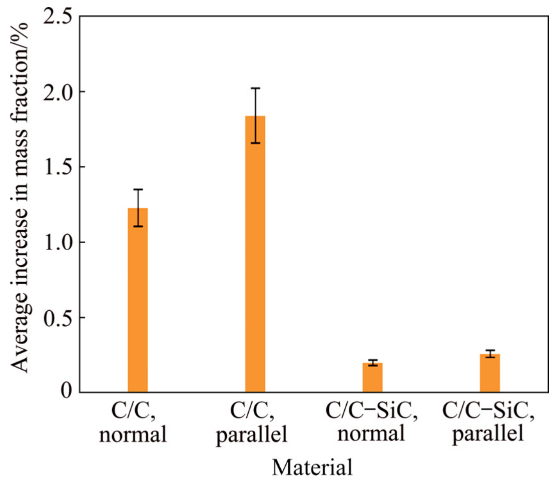


Fig. 3 Average increase in mass fraction for different orientations of laminates after immersing in brake oil

2.3 Reciprocating wear tests

Samples were weighed prior to wear tests. The details of wear testing were presented in our previous article [28]. To carry out reciprocating wear tests, chrome steel was used as counter surface material (plate in the case of pin/plate arrangement and ball in the case of ball/plate arrangement). The hardness of the counter surface material was HRC 62. The friction coefficient and wear loss were investigated. To investigate wear loss, samples were weighed before and after the test using a weighing machine (Model RA310, Roy Electronics, Varanasi, India). The effect of load on reciprocating wear behavior was also investigated. The load was varied ranging 50, 60, 70, 80 and 90 N. Each test was performed for 13500 cycles with a frequency of 5 Hz. The stroke length of the reciprocating arm was kept constant at 3 mm. The schematic diagram showing reciprocating wear test rig is shown in Fig. 4. For partial

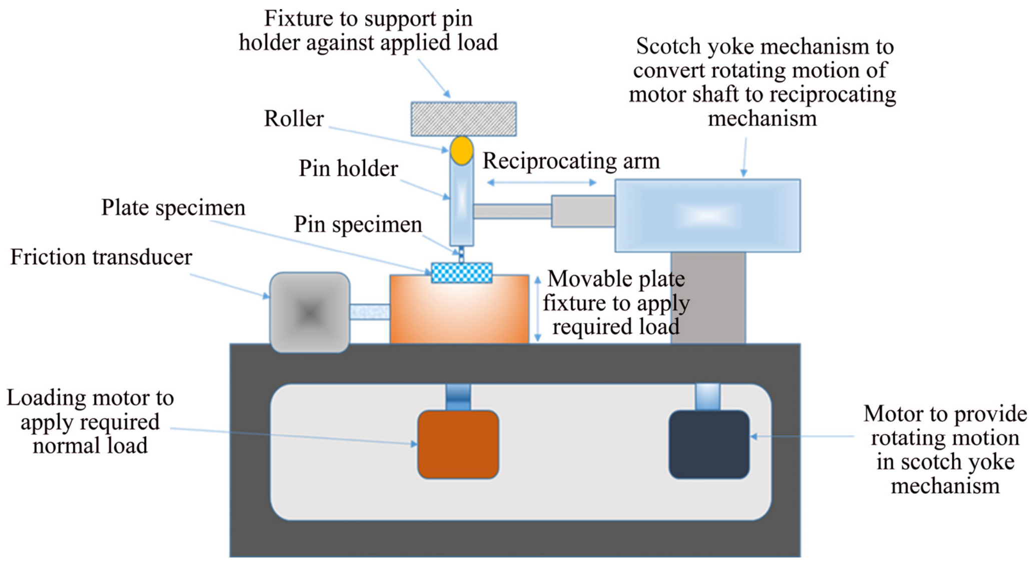


Fig. 4 Schematic diagram showing reciprocating wear test rig

conformity contacts (pin on plate), reciprocating wear tests were performed for normal and parallel orientations of laminates. The results of the wear tests of partial and low conformity contacts were plotted for the average of four repetitions per sample.

After wear testing, worn surfaces were analyzed under a scanning electron microscope to know the dominant wear mechanisms. A ZEISS EVO 18 RESEARCH (20 kV) scanning electron microscope was employed.

3 Results

3.1 Variation of friction coefficient with load, laminate orientation and surface conformity

Figure 5(a) shows the variation of friction coefficient with time in the case of C/C composites. It was observed that the friction coefficient of C/C normal first increased with time and after some time, it decreased whereas C/C parallel showed almost stable behavior. The fluctuation in friction coefficient in the case of C/C normal was more obvious as compared to C/C parallel.

Figure 5(b) shows the variation of friction coefficient with time in the case of C/C−SiC composites. It was observed that the rise in friction coefficient was sharp for the first few cycles in the case of C/C−SiC normal. However, the friction coefficient for both orientations of laminates acquired almost the same values when the number of cycles increased.

It can be observed from Fig. 5(c) that fluctuation in friction coefficient in the case of low conformity contacts was more obvious as compared to partial conformal contacts. The friction coefficient of C/C composites increased sharply with time, whereas the friction coefficient of C/C−SiC composites did not vary much with time in low conformity contacts.

It can be observed from Fig. 6 that at low loads, the friction coefficients of C/C and C/C−SiC composites are almost same for parallel as well as the normal orientations of laminates. As the load increased, the friction coefficient of C/C normal, C/C parallel and C/C−SiC normal first increased and then decreased. However, in the case of C/C−SiC parallel, friction coefficient first increased with an increase in load, then decreased and again increased after that.

It can be observed from Fig. 7 that the friction coefficient of C/C−SiC composites was larger as compared to C/C composites in low conformity contacts. C/C–SiC composites showed an increase in friction coefficient with the increase in load. However, the friction coefficient of C/C composites first increased with the increase in load and then decreased.

3.2 Variation of wear loss with load, laminate orientation and surface conformity

It can be observed from Fig. 8 that C/C normal composites showed the highest wear loss. Wear loss of C/C parallel and C/C−SiC normal composites increased with an increase in load, while wear loss of C/C normal first increased with an increase in load and decreased after that.

It can be observed from Fig. 9 that in low conformity contacts, wear loss of C/C composites increased with increase in load, whereas wear loss of C/C−SiC composites first increased with increase in load and decreased after that.

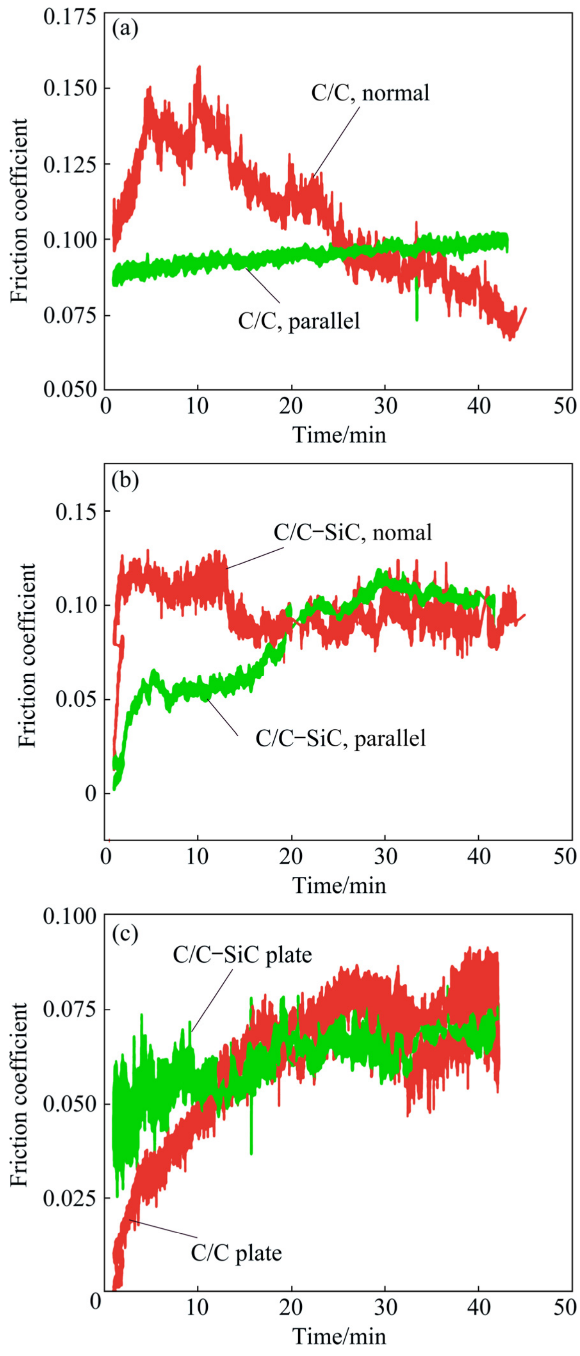


Fig. 5 Representative plots of variation of friction coefficient with time at load of 70 N: (a, b) C/C and C/C−SiC composites tested in partial conformity contacts, respectively; (c) C/C and C/C–SiC composites tested in low conformity contacts

3.3 Comparison of friction coefficient and wear loss in dry and brake oil environments

Bar charts (Figs. 10 and 11) were plotted to compare the friction coefficient and wear loss of C/C and C/C–SiC composites in dry and brake oil environments with variation of laminate orientation and surface conformity at a load of 70 N, respectively. The friction coefficient and wear loss values for the dry environment were taken from our previous article [28]. Figures 10 and 11 indicated that the friction coefficient was reduced by 70%−80% and wear loss was reduced by 75%−90% in brake oil as compared to the dry environment.

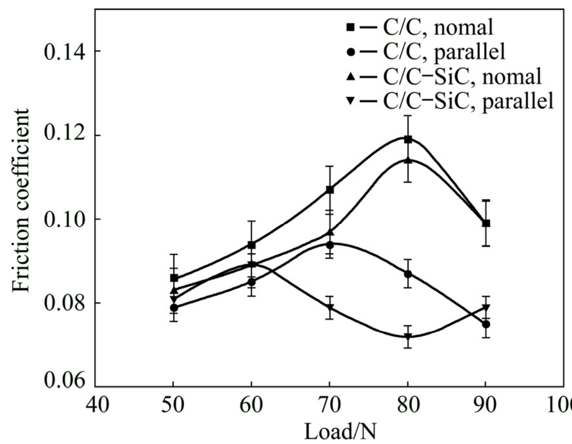


Fig. 6 Variation of friction coefficient with load in partial conformity contacts

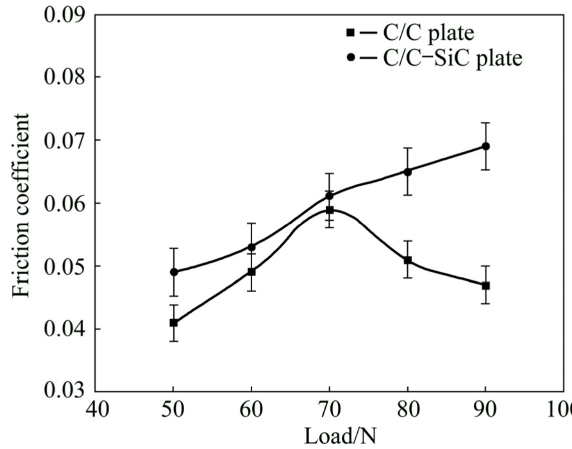


Fig. 7 Variation of friction coefficient with load in low conformity contacts

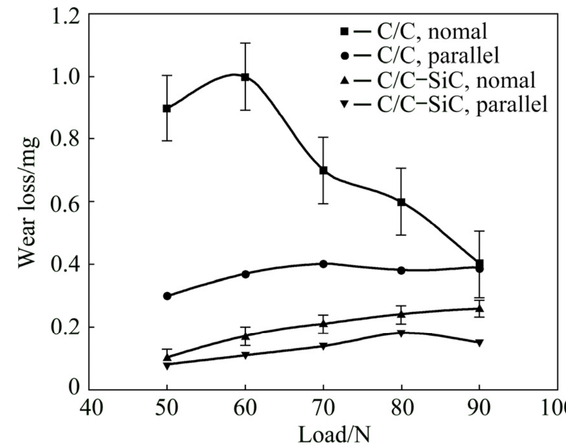


Fig. 8 Variation of wear loss with load in partial conformity contacts

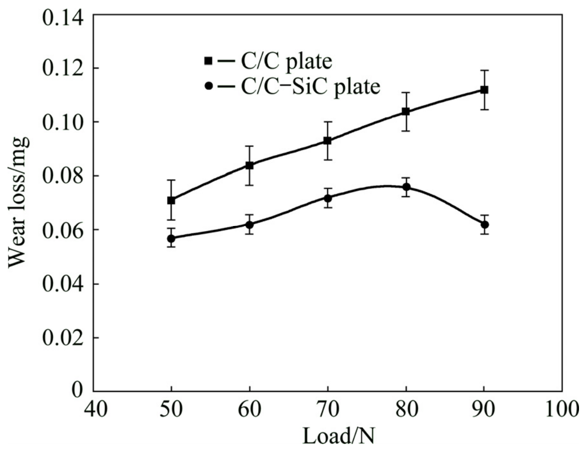


Fig. 9 Variation of wear loss with load in low conformity contacts

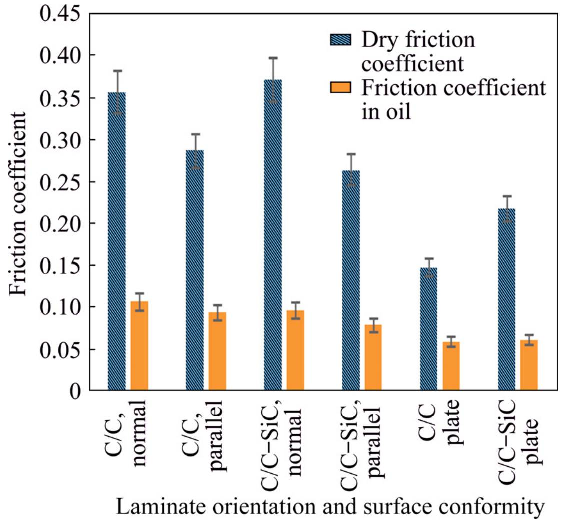


Fig. 10 Comparison of friction coefficient in dry and brake oil environments at load of 70 N

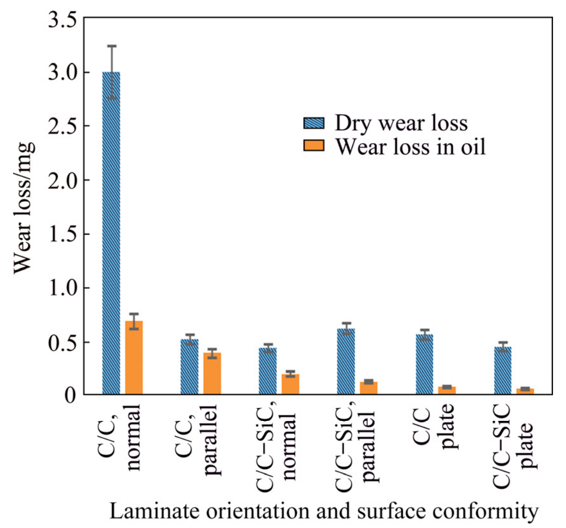


Fig. 11 Comparison of wear loss in dry and brake oil environments at load of 70 N

4 Discussion

Friction film formation, disruption, adhesion, and abrasion of contact conjunctions depict the tribological behavior of C/C and C/C−SiC composites in the dry environment [28], but the formed friction film was less in the presence of brake oil because brake oil got mixed with wear debris and prevented adhesion of wear debris on the surface. Thus, the tribological response of C/C and C/C−SiC composites was different in the brake oil environment.

Fluctuation in friction coefficient for composites having normal orientation of laminates was more obvious as compared to composites having parallel orientation of laminates due to higher surface porosity and more absorption of brake oil in the case of parallel orientation of laminates. Sliding occurred in a very confined region under reciprocating conditions. Thus, some of the wear debris filled the pores on the surface, and the rest of them made a film of low shear resistance on the surface of composite with the parallel orientation of laminates. The escapement of wear debris from in-between the contacting surfaces by any kind of force (e.g. centrifugal force in the case of unidirectional pin on disc sliding), was very less. In the case of composites having normal orientation of laminates, surface porosity and absorption of brake oil were less. Although the generated wear debris made a film of low shear resistance on the surface of composite, due to presence of oil, the film did not show strong adhesion to the surface of the composite. Thus, the formed film was easily drawn away by counter surface in the subsequent cycle. Pulling out of oil film from the surface was also observed by YANG et al [36]. Therefore, composites with normal orientation of laminates showed more obvious fluctuation in friction coefficient.

Composites with normal orientation of laminates showed higher friction coefficient as compared to composites with parallel orientation of laminates in the first few cycles. As the number of sliding cycles increased, the proportion of wear debris increased in wear debris and oil mixture. Some of the wear debris and oil mixture squeezed out with time, from in-between the contact surfaces. Squeezing out of excess oil from in-between the contact surfaces was also observed by PASKVALE et al [37] in oil-lubricated tribo-contacts. Thus, the chances of formation of friction film increased after some cycles in the case of composites having normal orientation of laminates as they absorbed less oil. However, in the case of composites having parallel orientation of laminates, squeezing out of oil led to the interaction of fibers of composite and asperities of counter surface. Fiber provided more resistance to sliding as breakage of fibers occurred which required more braking energy. This increased the friction coefficient after few cycles in composites having parallel orientation of laminates. Direct solid−solid contact, interaction of fibers with the counter surface, and the increase in friction coefficient after squeezing out of oil were also observed by ZHAO et al [38].

In the case of low conformity contacts, friction behavior of C/C−SiC composites was more stable as compared to C/C composites. The friction coefficient of C/C composites increased with time but the friction coefficient of C/C−SiC composites did not vary much with time. In the case of low conformity contacts (i.e. ball on plate arrangement), the stresses induced were high and localized as compared to that of partial conformity contacts (pin on plate arrangement). Thus, the oil between the contacting surfaces was very less due to the generation of high contact stresses and the mixture of wear debris and oil stuck to the ball near the contact area. High stresses pulverized the wear debris and formed friction film. However, the generation of cyclic compressive stresses of opposite sign led to the disruption of friction film [16] which increased friction coefficient of C/C composites as the number of cycles increased in low conformity contacts.

The friction coefficient of composites with normal orientation of laminates was higher as compared to that of composites with the parallel orientation of laminates in brake oil environment. This was attributed to the absorption of more brake oil in the case of composites having parallel orientation of laminates. The friction coefficient of C/C composites first increased with an increase in load and decreased after that, whether it was loaded with the normal orientation of laminates or parallel orientation of laminates. C/C−SiC normal composites also showed the same trend. The increase was attributed to the more asperity interaction and squeezing out of excessive oil from in-between the contacting surfaces [39]. Figure 12 shows micrograph of C/C normal composite tested at 80 N. Wear debris in the form of fiber fragments and particles from carbon matrix can be observed. The asperities from the counter surface led to grain abrasion, and fiber breakage occurred due to grain abrasion at high loads. The size of fiber fragments in wear debris was not the same. The grain abrasion increased with the increase in load, which in turn increased the friction coefficient. However, as the load was increased further, pulverization of debris took place. Due to squeezing out of excess oil, the pulverized wear debris was able to form a friction film on the surface which decreased the friction coefficient at high loads.

Figure 13 shows the micrograph of C/C−SiC normal composite tested at 80 N. Wear debris in the form of SiC particles and fiber fragments can be observed. SiC particles are hard to pulverize. Thus, as the load was increased, these SiC particles penetrated the counter surface and increased the friction coefficient through grain abrasion. However, as the load was increased further, the SiC particles erupted from the matrix in the form of wear debris as a result of repeated flexion in opposite directions. The sliding area was confined in reciprocating sliding. Thus, SiC acted as the third body and prevented the direct contact of the interacting surfaces, which in turn decreased friction coefficient.

Figure 14 shows the micrograph of C/C parallel composite tested at a load of 70 N. Some wear craters were observed. Softening of matrix occurred when composites were treated with oil [40]. However, softening was predominant in the case of parallel orientation of laminates due to more absorption of oil.

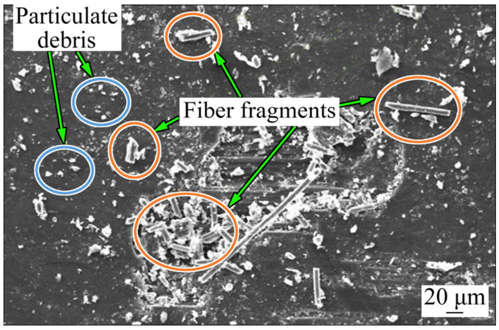


Fig. 12 Micrograph of C/C normal composite tested at load of 80 N

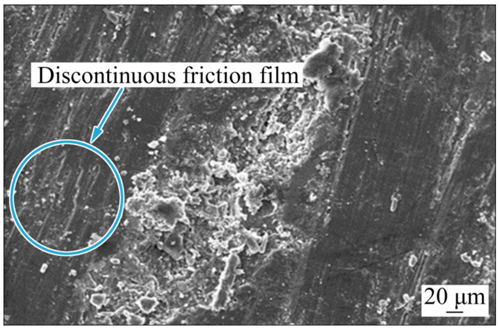


Fig. 13 Micrograph of C/C−SiC normal composite tested at load of 80 N

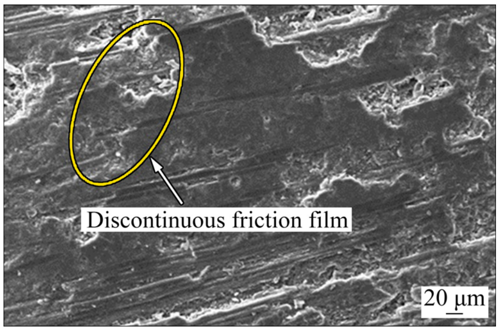


Fig. 14 Micrograph of C/C parallel composite tested at load of 70 N

Thus, matrix material was ejected easily and made the surface rougher, which increased the friction coefficient. The chance of adhesion of wear debris on the surface was less due to the presence of brake oil. As the load was increased further, squeezing out of excess brake oil and formation of the smooth surface by filling up craters with wear debris led to the decrease in friction coefficient.

Figure 15 shows the micrograph of C/C−SiC parallel composite tested at a load of 90 N. SiC particles in the form of wear debris can be observed. Fiber breakage can also be observed which was due to grain abrasion of C/C−SiC parallel composite at high loads. In the case of C/C−SiC parallel composite, the absorption of brake oil was less as compared to C/C parallel composite. Thus, brake oil did not lubricate the surface very much. The penetration of hard SiC particles in the counter surface led to the first rise in friction coefficient value. As the load was increased, SiC particles in wear debris acted as the third body and decreased the friction coefficient. As the load was increased further, the applied pressure was sufficient for the embedment of SiC particles in the counter surface which in turn increased the friction coefficient through grain abrasion of C/C−SiC parallel composite.

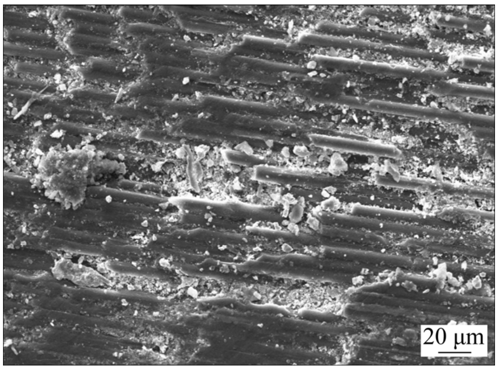


Fig. 15 Micrograph of C/C−SiC parallel composite tested at load of 90 N

It was observed that generally C/C composites showed high friction coefficient value as compared to C/C−SiC composites in reciprocating sliding in brake oil environment, which was opposite to the results obtained in unidirectional sliding under dry conditions [41,42]. This was due to sliding in a confined region because of small stroke length in the case of reciprocating sliding. In the case of unidirectional sliding, mutual overlap coefficient (MOC) was less, and the wear debris was gradually eliminated from the contact area. However, in the case of reciprocating sliding, MOC was high and wear debris got entrapped in the contact area. The entrapment of SiC particles in the contact area led to the decrease in friction coefficient of C/C−SiC composites because SiC particles were not easy to cut even at high loads and acted as the third body. In the case of C/C composites, the entrapment of carbon wear debris led to the formation of friction film which was easily disrupted due to repeated flexion in opposite direction in the case of reciprocating sliding.

For low conformity contacts, the friction coefficient of C/C−SiC composites was higher as compared to that of C/C composites. Stresses were very high and localized in low conformity contacts. The generation of high stresses led to the disruption of friction film formed on the surface of C/C composites, which increased the friction coefficient as the load was increased. However, as the load was increased further, negative pressure was generated on the surface of the composite due to reciprocating sliding. As oil was entrapped in the pores of C/C composites due to its open surface porosity, it rushed to the surface due to negative pressure which lubricated the surface. This, in turn, decreased the friction coefficient at high loads. Figure 16 shows the worn surface of C/C plate tested at a load of 80 N in low conformity conditions. Wear debris observed was very less. The wear debris generated got mixed with oil and stuck to the ball near the contact area.

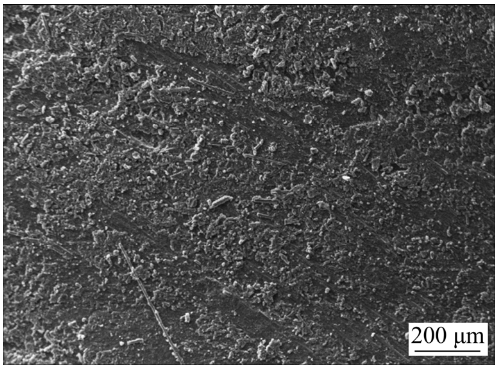


Fig. 16 Micrograph of worn surface of C/C plate tested at load of 80 N in low conformity contacts

In the case of C/C−SiC composites tested in low conformity contacts, the wear debris generated (SiC particles) embedded in the counter surface; however, the contact area was small. The embedded SiC particles in the counter surface abraded the composites. Resistance to sliding was also provided by fibers. Broken fibers can be observed in Fig. 17. The higher the load was, the deeper the penetration of SiC particles was and the more the resistance to sliding was as deeper penetration covered more fiber filaments. This increased the friction coefficient as the load was increased. The friction coefficient in the case of low conformity contacts was lower as compared to partial conformity contacts.

It was observed that wear loss in the case of C/C parallel composite, C/C−SiC normal composites and C/C−SiC parallel generally increased with an increase in

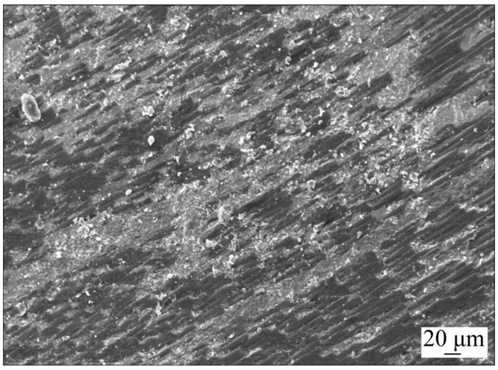


Fig. 17 Micrograph of C/C−SiC plate tested at load of 70 N in low conformity contacts

load. But the increase was not much significant. However, in the case of C/C normal composite, wear loss first increased and then decreased significantly. The increase in wear loss of C/C composites was attributed to the increase in abrasive wear with the increase of load and the decrease in wear loss was attributed to the formation of friction film at high loads. The possibility of friction film in the case of C/C parallel composite was less due to more absorption of oil. Thus, wear loss increased with increase in load. However, the increase was not much significant because brake oil lubricated the surface. The hardness of C/C−SiC composites is higher as compared to that of C/C composites. Thus, wear loss of C/C composites was more as compared to that of C/C−SiC composites. In low conformity conditions also, C/C composites showed high wear loss as compared to C/C−SiC composites.

Friction coefficient and wear loss decreased to a greater extent in brake oil. The dispersion of wear debris in the brake oil prevented adhesion on the surface of the composite as well as counter surface. But the presence of oil on the surface and subsurface led to lubrication of surface, which decreased the friction coefficient. Furthermore, adhesion and formation of friction film were prevented by brake oil, which reduced the wear loss.

5 Conclusions

(1) The reciprocating sliding behavior of C/C and C/C−SiC composites was different in brake oil condition as compared to dry environment. The effect of laminate orientation and surface conformity also changed with the environment.

(2) The stability of the friction coefficient changed with the orientation of laminates. Composites with the parallel orientation of laminates showed less fluctuation in friction coefficient due to more absorption of oil and low adhesion of wear debris to the counter surface in the presence of oil. The formation of friction film was difficult in brake oil condition due to the dispersion of wear debris in the oil.

(3) The presence of brake oil reduced friction coefficient by 70%−80% and wear loss by 75%−90% as compared to dry environment.

(4) The friction coefficient of C/C composites was generally more than that of C/C−SiC composites in partial conformity conditions due to the presence of hard SiC particles which prevented direct contact of interacting surfaces, and confined region sliding which prevented escapement of SiC particles.

(5) The friction coefficient of composites with the parallel orientation of laminates was generally less due to more surface porosity and absorption of oil as compared to composites having normal orientation of laminates.

(6) The friction coefficient and wear loss of composites were less in the case of low conformity contacts. Friction coefficient decreased by a maximum of 49% and wear loss decreased by a maximum of 78% in low conformity contacts as compared to partial conformal contacts, due to less asperity interaction, and high and localized stress regions at the same load level.

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制动油处理的碳纤维增强陶瓷基复合材料的往复滑动摩擦学

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摘　要：研究制动油、层板取向和摩擦接触方式对碳/碳(C/C)和碳/碳−碳化硅 (C/C–SiC) 复合材料摩擦磨损行为的影响。研究往复滑动条件下材料的摩擦磨损行为，分别采用两种不同的接触方式进行摩擦试验，即销−盘式和球−盘式，同时考虑层板方向(垂直和平行方向)的影响。法向载荷范围为 50−90 N，变化间隔为 10 N。结果表明，与层板平行取向复合材料相比，垂直取向复合材料的摩擦因数和磨损量均较大，垂直取向 C/C 复合材料具有最大的摩擦因数。与销−盘式接触相比，球−盘式接触摩擦的样品其磨损量最大可降低 78%，摩擦因数最大可降低 49%。润滑油的存在减小磨屑被压实的倾向，从而使摩擦膜难以形成，因此影响摩擦行为。在往复滑动过程中，由有限区域运动而引起的接触面间的磨屑滞留可描述摩擦磨损行为。

关键词：往复滑动摩擦学；C/C 复合材料；C/C−SiC 复合材料；摩擦因数；耐磨性；层板取向

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