

# Self and Complementary Mating Of Carbon-Carbon and Carbon-Carbon-Silicon Carbide Composites in Reciprocating Tribo-Interactions

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## ABSTRACT

The counter-surface (CS) material during tribological interactions significantly affects the prevalent wear and friction mechanisms, and correspondingly tribological behaviour. Although disparate uni-phase (i.e., single constituent) counter-surface materials exhibit contrast tribological phenomena, the presence of multi-phase (i.e., multiple constituents) CS may further complex the tribological analysis. The complexity of tribo-system further increases in the presence of two multi-phase materials rubbing against each other. Thus, the present article aims to investigate the reciprocating sliding of carbon-carbon (C/C) and carbon-carbon-silicon carbide (C/C-SiC) composites in self and complementary mated pairs with variation of laminate orientation (i.e., normal and parallel orientation of laminates) and normal load. The results indicate that C/C and C/C-SiC complementary mated pair exhibited coefficient of friction (COF) in between the COF of C/C and C/C-SiC self-mated pairs, whether used in parallel/parallel or normal/parallel combinations. Wear loss exhibited by C/C composites in complementary mated pair was higher than wear loss of C/C composites in self-mated pair. The synergism between debris retention in between the interacting surfaces, friction film formation, extent of ploughing effect and repeated flexion in opposite direction dictated the tribological behavior of C/C and C/C-SiC composites in reciprocating sliding.

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## 1. INTRODUCTION

The demand of light weight structures makes ceramics and its composites the promising candidates for weight sensitive applications. In the plethora of ceramics based high performance composites, carbon-carbon (C/C) composites

have special place due to their high specific strength, high modulus, high specific heat and thermal conductivity, low coefficient of thermal expansion, and low density [1-3]. The strength of C/C composites increases with increase in temperature whereas strength of metals decreases with increase in temperature [4]. C/C

composites are being in use as brake materials of military and commercial aircrafts, motorcycles, and racing cars [2]. Despite its various outstanding properties, C/C composites suffer from poor oxidation resistance, low wear resistance and high sensitivity to humidity which limit their applications such as in marine sectors [5,6]. Although the effect of humidity is low at high energy braking conditions, the tribological behavior is severely affected at low energy braking conditions because the adsorbed species on the surface of C/C composites lubricate the surface and greatly reduces the coefficient of friction (COF). To counter these limitations, some researchers [7-10] have applied wear and oxidation resistance coatings on the surface of C/C composite to improve its wear and oxidation resistance in weight sensitive applications. Moreover, some researchers [11,12] have also tried to modify matrix to improve its wear resistance. But these methods require a plenty of experimentation, and takes a lot of time.

Recently developed carbon-carbon-silicon carbide (C/C-SiC) composite have shown its potential as high performance brake material by overcoming the disadvantages of C/C composites. [6]. The C/C-SiC composites contain dual matrix of carbon and SiC, and exhibit excellent properties such as low wear rate, stable coefficient of friction, high strength and good thermal properties [2,13]. These composites are not considerably affected by ambient conditions such as humidity and temperature [14] which make them better as compared to C/C composites. However C/C-SiC composite has higher density as compared to C/C composite [15] which limits its usage in weight sensitive applications.

A strategy may be developed to encounter the limitations of both C/C and C/C-SiC composites in weight sensitive wear related applications, by using them in complementary mating friction pairs. However, the tribological behavior of C/C and C/C-SiC composites varies with the counter-surface material [3,13]. Most of the researchers have investigated the tribological behavior of both C/C and C/C-SiC composites with self-mated pairs and parallel orientation of laminates [1,5,15,16]. But the investigations regarding tribological behavior of C/C and C/C-SiC composites as self and complementary friction partners with normal/parallel combination of laminates have not been considered yet.

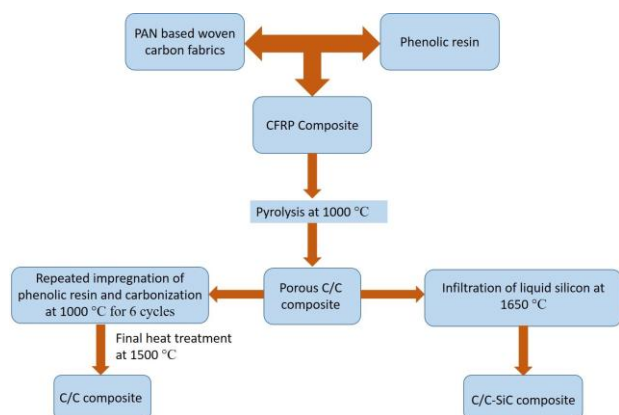
Tribological behavior of fiber reinforced composites also varies with the orientation of fibers with respect to the sliding surface [17-19]. Different orientations yield different results [20]. However, it is very difficult to generalize that which orientation will yield desirable tribological performance because it depends on the architecture of fibers in the composite, type of matrix and reinforcement, and tribological mechanisms for different orientation of fibers [20-23].

Tribological behavior of a material is different in unidirectional sliding and reciprocating sliding [24-26]. Sudden transition in friction regime doesn't occur in case of unidirectional sliding whereas in case of reciprocating sliding, transition in friction regime takes place at the beginning of each stroke [25]. The high energy sliding behaviour of C/C and C/C-SiC composites in complementary mated pairs with full conformity contacts and parallel orientation of laminates have been investigated in unidirectional sliding conditions [2]. The C/C and C/C-SiC composites have not been investigated for their tribological behavior in reciprocating sliding conditions with self and complementary mated pairs, and normal/parallel orientation of laminates. Therefore in this article, reciprocating sliding behavior of C/C and C/C-SiC composites have been investigated in self and complementary mated pairs with variation of normal load and laminate orientation. The underpinning mechanisms governing the tribological behaviour of C/C and C/C-SiC composites are also discussed.

## 2. EXPERIMENTAL

### 2.1 Materials

The schematic showing the procedure followed for the fabrication of two-dimensional (2D) cross woven fabric C/C and C/C-SiC composites is shown in Fig. 1. Firstly, polyacrylonitrile based carbon fiber mats were reinforced in phenolic resin matrix via vacuum assisted resin transfer molding process, which was followed by pyrolysis at 1000 °C. Densification of resulting porous C/C composites was performed by repeated impregnation of phenolic resin in vacuum, and repeated carbonization, i.e., for six cycles. After densification, heat treatment was done at a temperature of 1500 °C. C/C composites obtained after heat treatment contained ~50% carbon fibers by volume. The density of C/C composite was 1.8 g/cm<sup>3</sup>.

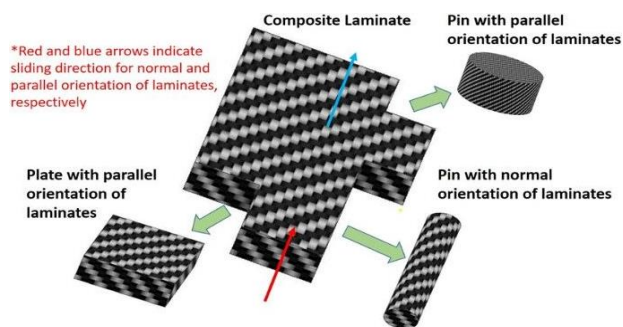


**Fig. 1.** Schematic showing fabrication procedure followed for C/C and C/C-SiC composites.

The C/C-SiC composites were fabricated by liquid silicon infiltration (LSI) technique. To fabricate C/C-SiC composites, porous C/C composites were used as preform. The siliconization of porous C/C preform was performed by heating it with silicon at a temperature of 1650 °C. Infiltration of silicon in C/C preform occurred through segmentation cracks and micro-delamination. Silicon reacted with carbon matrix and formed silicon carbide. The resulting C/C-SiC composite contained ~ 38% of silicon carbide and 2% free silicon by volume. The density of C/C – SiC composites was 1.95 g/cm<sup>3</sup>.

## 2.2. Sample preparation

Samples were prepared in the form of pins and plates. The sample extraction strategy is shown in Fig. 2. The reciprocating wear tests were performed for self and complementary mated friction pairs of C/C and C/C-SiC composites, with normal and parallel orientation of laminates. The SEM images showing surface characteristics of composites with normal and parallel orientation of laminates are shown in Fig. 2 of our published article [27]. To perform tests for normal orientation of laminates, C/C and C/C-SiC composite samples were prepared in the form of pins having 9 mm diameter and 30 mm length (designated as C/C normal and C/C-SiC normal). For parallel orientation, composite pins were having 9 mm diameter and 4 mm thickness (designated as C/C parallel and C/C-SiC parallel). For CS material, C/C and C/C-SiC composite were prepared in the form of square plates having 25 mm side and 4 mm thickness (designated as C/C plate and C/C-SiC plate).



**Fig. 2.** Sample extraction strategy from fabricated composite plates.

## 2.3. Reciprocating wear testing

For self-mated pairs, pins of C/C and C/C-SiC composites were slid against C/C and C/C-SiC composite plates respectively. For complementary mated pairs, C/C pin was slid against C/C-SiC composite plate and C/C-SiC pin against C/C composite plate. To carry out tests for normal orientation of laminates, composite pins having normal orientation of laminates with respect to sliding plane were slid against composite plate having parallel orientation of laminates with respect to sliding plane. Similarly for parallel orientation of laminates, composite pins having parallel orientation of laminates with respect to sliding plane were slid against composite plate having parallel orientation of laminates with respect to sliding plane.

Firstly, C/C normal was slid against C/C plate for self-mated wear tests. Then, C/C parallel was slid against C/C plate. Same self-mated wear tests were performed for C/C-SiC composites for different orientation of laminates. For complementary wear tests, C/C normal and C/C parallel were slid against C/C-SiC plate, and similarly C/C-SiC normal and C/C-SiC parallel were slid against C/C plate.

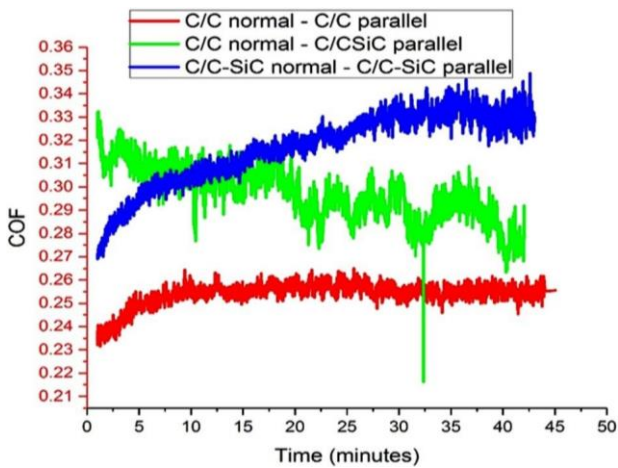
Wear tests were performed on reciprocating sliding wear test rig (TE 200ST, Magnum Engineers, Bangalore, India). Each test was performed at 5 Hz frequency, and for 13500 cycles. Stroke length was kept constant as 3 mm. Friction coefficient and wear loss were investigated. To investigate wear loss, samples (both pin and plate) were weighed before and after the test. The effect of normal load on reciprocating wear behavior was also investigated. The load was varied as 50 N (0.78 MPa normal pressure), 60 N (0.94 MPa), 70 N (1.10 MPa), 80 N (1.26 MPa) and 90 N (1.415 MPa). Relative humidity at the time of testing was 71 ± 3%, and the temperature of the ambient was 32 ± 2 °C.

To analyze the worn surface of composites and gain an understanding about the dominant wear mechanisms, worn surfaces were analyzed under scanning electron microscope. A ZEISS EVO RESEARCH, 20 kV scanning electron microscope was employed.

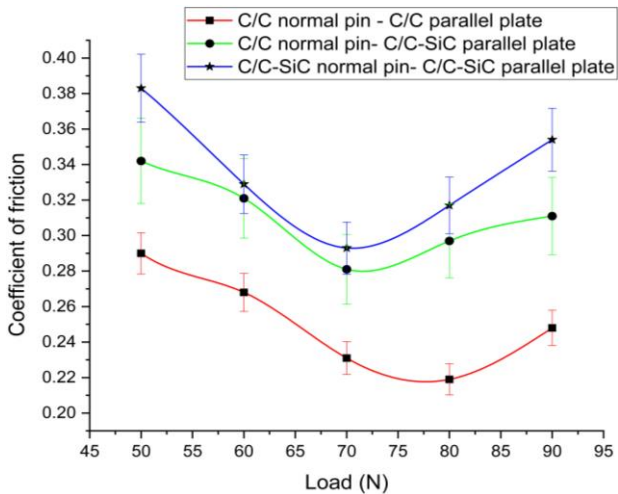
### 3. RESULTS

#### 3.1. Normal/parallel combination

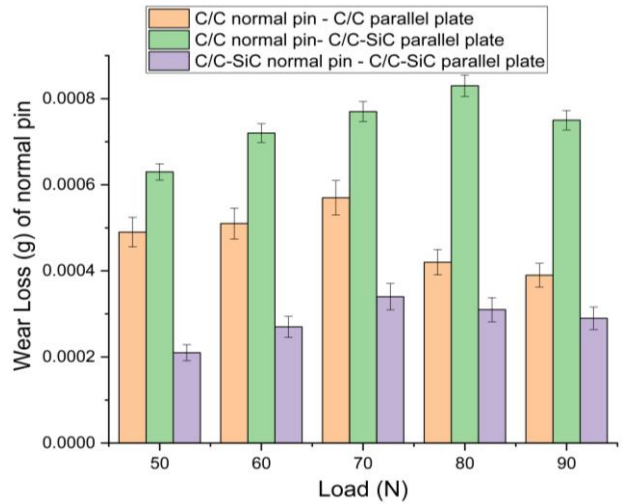
Fig. 3a shows the variation of COF with time when normal/parallel self-mated and complementary-mated combinations were tested. It was observed that COF of C/C-SiC self-mated pair increased with time whereas, COF of complementary mated pair decreased with time. The self-mated C/C-SiC pair showed higher COF value as compared to C/C self-mated and C/C and C/C-SiC complementary mated pair. The COF of C/C self-mated pair was almost stable and lower as compared to C/C-SiC self-mated and complementary mated pairs.



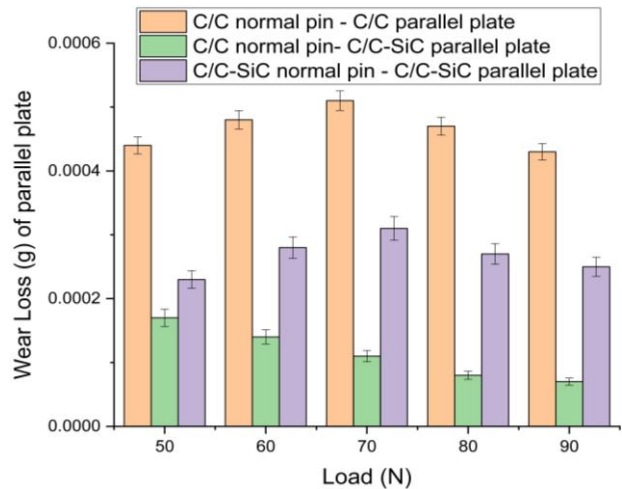
(a)



(b)



(c)



(d)

Fig. 3. Variation of (a) COF with time (at 80 N load), (b) COF with load (c) wear loss of normal pin, and (d) wear loss of parallel plate, in case of normal/parallel combination.

Fig. 3b shows the variation of COF with load when the composite pin was loaded with normal orientation of laminates and composite plate was loaded with parallel orientation of laminates.

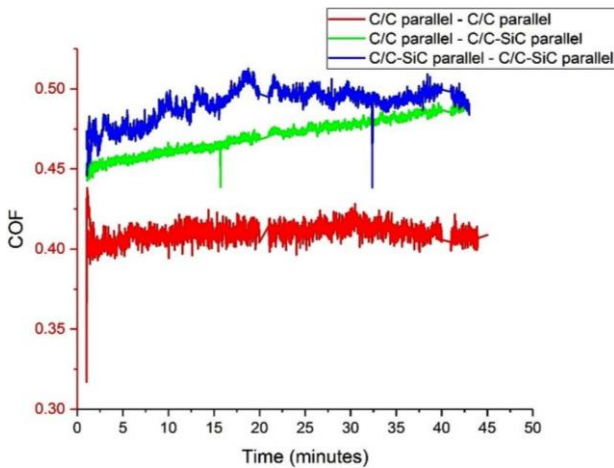
It can be observed from Fig. 3b that in case of normal/parallel combination, C/C-SiC self-mated pair exhibited highest COF, and C/C self-mated pair exhibit lowest COF at high loads. COF of C/C and C/C-SiC complementary pair lied in-between the COF of C/C and C/C-SiC self-mated pairs. COF first decreased with increase in load, and showed increase at high loads both complementary and self-mated pairs. Fig. 3c shows the variation of wear loss of composite pin (normal orientation) with load. It can be observed that C/C pin exhibited highest wear loss in complementary-mated pair whereas C/C-SiC pin exhibited lowest wear loss in self-mated pair.

Wear loss first increased with increase in load and experienced decrease at higher loads.

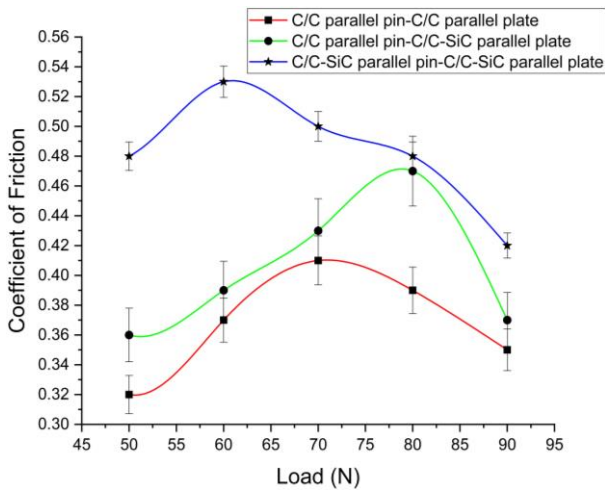
It can be observed from Fig. 3d that C/C plate exhibited highest wear loss when used in self-mated pair whereas C/C-SiC plate exhibited lowest wear loss when used in complementary-mated pair. Wear loss of C/C-SiC plate decreased with increase in load when used in complementary-mated pair.

### 3.2. Parallel/parallel combination

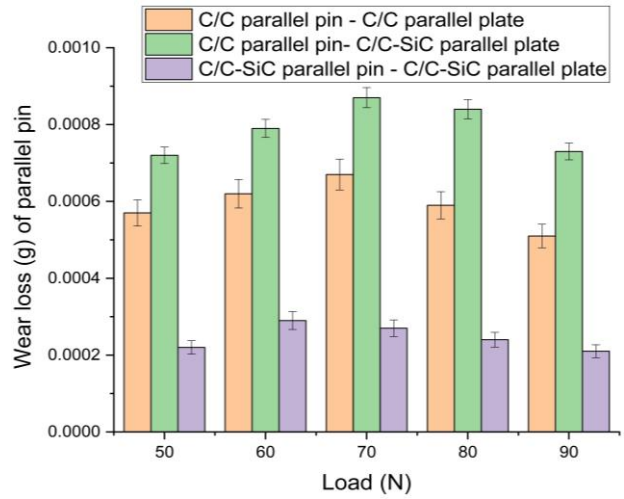
It can be observed from Fig. 4a that COF exhibited by self-mated C/C-SiC pair was higher as compared to C/C self-mated and C/C and C/C-SiC complementary-mated pairs, when loaded in parallel/parallel combination. COF of complementary mated pair increased with time whereas for C/C self-mated pair, it was almost stable. The COF of C/C-SiC self-mated pair first increased with increase in time and got stabilized after some time.



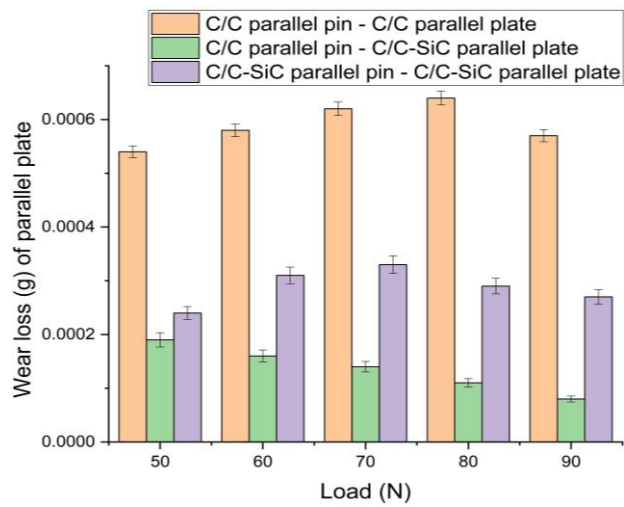
(a)



(b)



(c)



(d)

**Fig. 4.** Variation of (a) COF with time (at 80 N load), (b) COF with load, (c) wear loss of normal pin, and (d) wear loss of parallel plate, in case of parallel/parallel combination.

Fig. 4b shows the variation of COF with load when the composite pin and composite plate were loaded with parallel orientation of laminates. It can be observed that COF first increased with increase in load and showed decrease at higher loads, which was in opposite trend as exhibited by normal/parallel combination. C/C-SiC self-mated pair showed highest COF whereas C/C self-mated pair exhibited lowest COF. The difference between COF of C/C-SiC self-mated pair and C/C - C/C-SiC complementary mated pair was less at high loads.

It can be observed from Fig. 4c that C/C parallel pin showed highest wear loss when complementary mated with C/C-SiC plate. The C/C-SiC parallel pin showed lowest wear loss in self-mated pair. It can be observed from Fig. 4d that wear loss of C/C-SiC

plate decreased with increase in load in case of complementary-mated pair. However, for C/C and C/C-SiC self-mated pairs, wear loss first increased and showed decrease afterwards.

#### 4. DISCUSSION

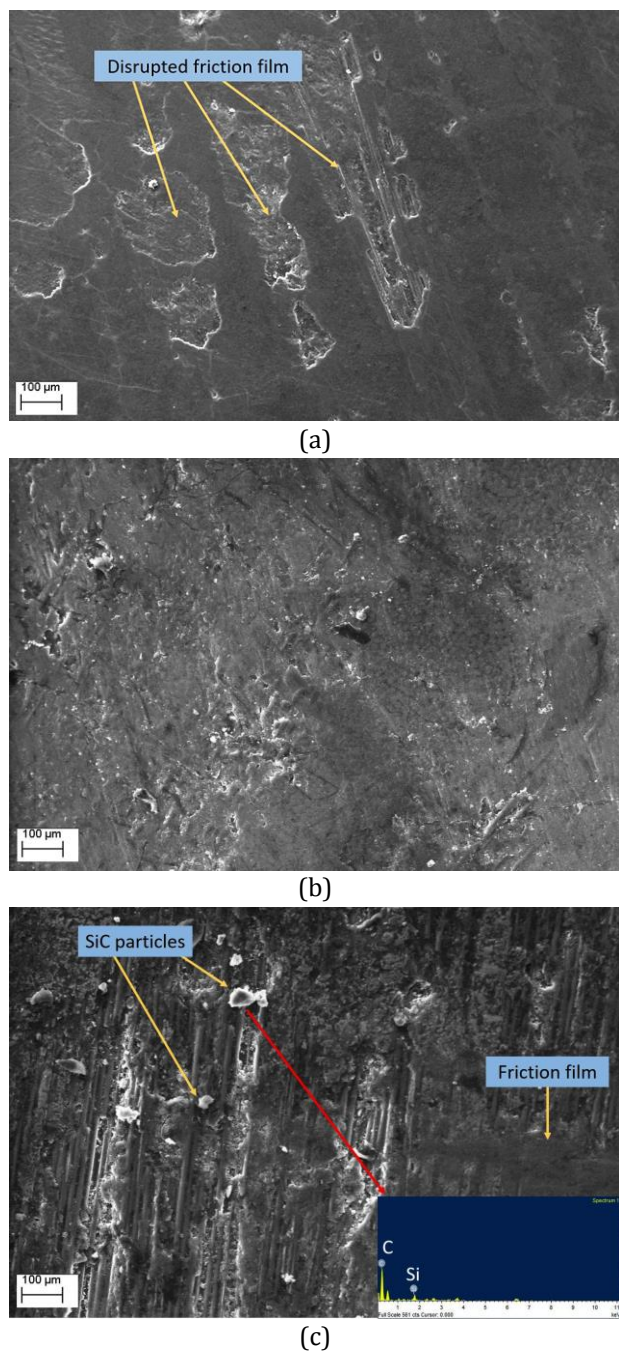
COF of C/C self-mated pair in normal/parallel combination was lower as compared to C/C-SiC self-mated and C/C-SiC and C/C complementary-mated pairs, as shown in Fig. 3b. This was attributed to the formation of flaky wear debris in case of C/C composites [2] in self-mated pair, which easily formed friction film on the surface of composite, and reduced COF. However, in case of C/C and C/C-SiC complementary mated pair, the hard SiC particles penetrated the surface of C/C composite due to high hardness of SiC as compared to C/C composite [28], and caused grain abrasion. This led to higher COF of complementary mated pair in the initial cycles, as shown in Fig. 3a. However, due to generation of carbon wear debris, friction film was formed after some cycles which led to decrease in COF. The disruption of friction film also occurred due to presence of hard SiC particles. Furthermore, reciprocating sliding occurs in confined region due to small stroke length. Thus wear debris was not ejected from in-between the interacting surfaces, and formation of friction film also took place. This simultaneous formation and disruption of friction film led to more fluctuations in friction curve of C/C and C/C-SiC complementary mated pair. In case of C/C-SiC self-mated pair, highest COF was observed which increased with time. The highest value was attributed to its high superficial hardness which provided resistance to sliding as micro-peaks which were meshed with micro-valleys experienced shearing, breaking, deformation and cutting under braking energy [29].

It was observed that in case of normal/parallel combination of laminates, COF firstly decreased with increase in load and experienced increase at higher loads. In case of C/C self-mated pair, friction film formation took place which decreased the COF with increase in load. However, when the load was increased further, the high value of stresses and its reversals at consecutive strokes led to the disruption of friction film which resulted in discontinuous friction film at the surface of composite, and hence increased COF at higher loads. Fig. 5a shows the discontinuous friction film formed at the surface of composite pin with normal orientation of laminates.

In case of C/C normal - C/C-SiC parallel complementary-mated pair, the asperities of the C/C-SiC parallel plate caused ploughing of C/C normal pin. At low loads, friction film was formed due to generation of carbon wear debris. But, the ease of disruption of formed friction film due to increased grit abrasion at high loads led to increase in COF. Some cracks on the surface of C/C normal pin were also observed due to repeated flexion in opposite directions. These cracks loosened the matrix, and led to detachment of matrix material. Fig. 5b shows C/C normal pin tested with C/C-SiC parallel plate at 80 N load. Some cracks due to repeated flexion can be observed.

The C/C-SiC self-mated pair showed highest COF values. SiC particles got detached from the matrix as reciprocating sliding caused repeated flexion in opposite direction, and induced cyclic stresses in opposite directions. SiC particles are hard to cut or pulverize even at high loads. Thus, SiC particles prevented the direct contact of interacting surfaces and thus reduced COF. However as the load was increased further, the loose SiC particles embedded in the surface and caused abrasion which increased COF. Fig. 5c shows the surface of C/C-SiC normal pin tested in self-mated pair at 80 N load. SiC particles can be observed (inset of Fig. 5c shows EDS map of wear particle which depicts presence of C and Si i.e., SiC). A segment of friction film can also be observed which resulted from pulverization of debris from carbon matrix.

The friction behavior in parallel/parallel combination was different from friction behavior in normal/parallel combination. The parallel/parallel combination exhibited higher COF values as compared to normal/parallel combination. Tribological behavior in case of parallel/parallel combination is different from normal/parallel combination as area proportion of fibers in interacting surfaces varies with orientation of laminates which effects overall COF due to disparate local friction coefficients [30]. The proportion of fiber filaments touching counter surface also varies with the load, and laminate orientation which affects COF [31]. In case of parallel/parallel combination, the interaction of fiber/fiber interaction was more as compared to normal/parallel combination. Fibers provide more resistance to sliding due to their fragmentation/breakage during tribo-interactions. Thus COF in case of parallel/parallel combination was more as compared to normal/parallel combination.



**Fig. 5.** SEM images showing worn surfaces of pins tested at 80 N (a) C/C normal pin tested with C/C parallel plate, (b) C/C normal pin tested with C/C-SiC parallel plate, and (c) C/C-SiC normal pin tested with C/C-SiC parallel plate (inset showing EDS map of wear particle which depicts presence of C and Si i.e., SiC).

In case of parallel/parallel combination, COF of C/C self-mated pair didn't considerably increased with time whereas, COF of complementary-mated pair increased with time. The increase in friction value of complementary-mated pair was attributed to increase in interaction (with time) between SiC particles in C/C-SiC parallel plate and carbon fibers in C/C

parallel pin, as surface wore out. However in case of C/C and C/C-SiC self-mated pairs, penetration of asperities was less as the hardness of both the interacting surfaces were same.

Composites in parallel/parallel combination exhibited opposite trend as shown by normal/parallel combination. In case of C/C parallel/parallel self-mated pair, fibers interacted with each other and fiber breakage occurred, as can be observed in Fig. 6a. Thus, as the load was increased, more fiber filaments came in contact with each other which increased the COF value [31]. However as the load was increased further, the carbon fiber fragments and carbon particles from matrix in the wear debris pulverized and formed a friction film which decreased the COF. In case of normal/parallel combination, fiber breakage was very less.

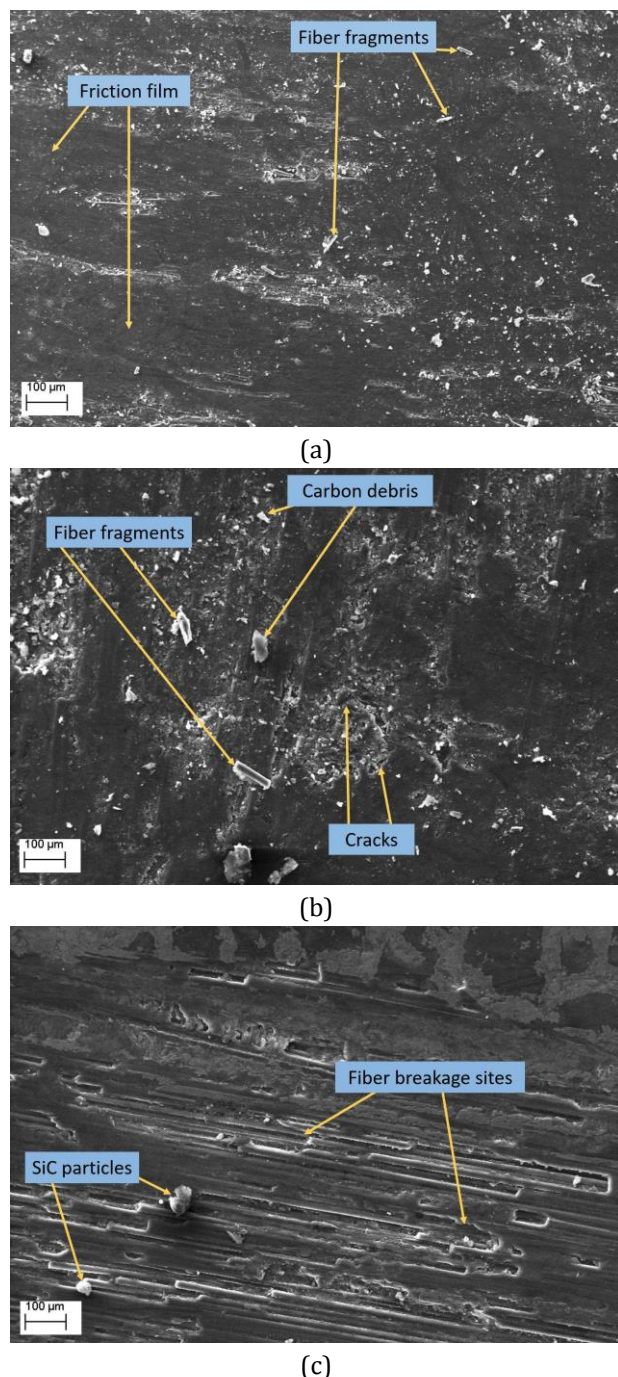
Fig. 6a shows C/C parallel pin tested with C/C parallel plate at 70 N load. Fiber fragments in wear debris can be observed. Formed friction film on the surface can also be observed.

The increase in COF of C/C parallel and C/C-SiC parallel combination was attributed to increase in penetration depth of SiC particles in C/C composites, as the load was increased. SiC also interacted with carbon fibers in C/C composites and led to breakage of fibers. As breakage of fibers required more braking energy, this increased COF with increase in load.

Fiber fragments in wear debris can be observed in Fig. 6b. Some carbon particles from matrix can also be observed. These carbon particles made friction film at high loads which decreased COF at higher loads.

When C/C-SiC composites are prepared through LSI technique, silicon is infiltrated into C/C composites through segmentation cracks and micro-delamination. Thus the area proportion of SiC in C/C-SiC composites in parallel orientation was more as compared to normal orientation of laminates. In C/C-SiC parallel self-mated pair, SiC from one surface tried to abrade other by grain abrasion. Due to its high hardness, both the interacting surfaces of friction couple resisted sliding. As the load was increased, SiC penetration was more which increased COF. Fiber breakage due to SiC

penetration also increased COF. However the eruption of SiC particles from the friction surface prevented the direct contact of interacting surfaces which decreased COF at higher loads. Fig. 6c shows C/C-SiC parallel pin tested in self-mated pair with parallel/parallel combination. Fiber breakage with SiC particles at the surface of composite can be observed.



**Fig. 6.** SEM images showing worn surfaces of pins tested at 70 N (a) C/C parallel pin tested with C/C parallel plate, (b) C/C parallel pin tested with C/C-SiC parallel plate, and (c) C/C-SiC parallel pin tested with C/C-SiC parallel plate.

Wear loss of friction mate having parallel orientation of laminates was less as compared to the mate having normal orientation of laminates in normal/parallel combination. The higher wear loss of composite with normal orientation may be attributed to the repeated flexion of opposite signs in consecutive strokes which led to loosening of matrix and generation of cracks parallel to the laminae. Thus, the matrix material was erupted from in-between the lamina, and hence higher wear loss was observed for normal orientation.

Wear loss of normal pin and parallel plate generally showed first increase with increase in load, and decrease afterwards. However, almost same behavior was exhibited by composites whether it is normal/parallel combination or parallel/parallel combination. In case of C/C self-mated normal/parallel combination, the tendency of friction film formation is high due to generation of carbon wear debris, but the extent of grit abrasion also increases with increase in load. Thus, rise in wear loss was observed initially, with increase in load. However, frictional energy was also increased at higher loads, due to high friction values. Thus more heat was generated which ease the pulverization of debris and formation of friction film due to which wear loss decreased. In case of C/C normal pin and C/C-SiC parallel complementary mated pair, SiC rolled in-between the interacting surfaces. The ejection of SiC particles was easy at high loads. Although it decreased COF, but due to its movement in opposite directions, it rolled over the interacting surfaces. It generated local compressive stresses whose magnitude varies within a cycle. This led to eruption of material from the surface. This increased wear loss as the load was increased. As the load was increased further, the embedment of SiC particles in between the sites of cross fibers in parallel orientation of laminates formed a smooth surface. The embedment of SiC particles in the surface of C/C composites increased the COF but due to high hardness of C/C-SiC composites, they were not easy to be abraded. Thus wear loss was decreased. The rolling of SiC particles in-between the interaction surfaces and its embedment, and high wear resistance of C/C-SiC composites due to their high superficial hardness described the wear behavior of C/C-SiC self-mated pair in normal/parallel combination. Wear behavior of parallel/parallel combination was same as friction behavior. The mechanisms elaborating friction and wear were same because both the interacting surface had parallel orientation of laminates.



## 5. CONCLUSION

The objective of this work was to investigate the reciprocating sliding behavior of C/C and C/C-SiC composites in self- and complementary-mated pairs for different orientation of laminates of friction mates and applied normal load. Following are some conclusion based on present study.

1. The COF exhibited by C/C and C/C-SiC complementary mated pair lies in between the COF of C/C and C/C-SiC self-mated pairs. Moreover, the wear loss of C/C composites in complementary mated pairs was higher as compared to C/C-SiC composites. Therefore, the C/C and C/C-SiC complementary mated pair may be selected for tribology related weight sensitive applications with C/C composite relatively thicker than C/C-SiC composite.
2. The normal/parallel combination exhibited lower COF as compared to parallel/parallel combination of laminates under normal pressure less than  $\sim 1.41$  MPa. The lower COF for normal/parallel combination was ascribed to the ease of formation of flaky wear debris in case of normal laminates, and less proportion of fiber/fiber interaction which resulted less generation of fiber fragments.
3. The normal/parallel combination exhibited opposite trend for COF as compared to parallel/parallel combination. The friction behaviour was governed by the synergism between morphology of wear debris, and proportion of different constituents of the composites at the contact surfaces which changes with the orientation of laminates. Furthermore, generation of wear debris, and formation and disruption of friction film were also governed by the load because the severity of repeated flexion in opposite direction, and induced compressive stresses depend upon the applied normal load in case of reciprocating sliding.

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