Friction and wear characteristics of robust carbon–carbon composites developed solely from petroleum pitch without reimpregnation

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Abstract: Friction and wear characteristics correlating the fiber reinforcement percentage of carbon–carbon (C/C) composites solely developed from petroleum pitch matrices were investigated. This study exhibits the tribo-characteristics of C/C composites developed in a single-step carbonization process for varying loads for the first time without a reimpregnation process. A pin-on-disc tribometer with a sliding speed of 0.5 m/s and loads of 5, 10, and 20 N with a flat tool grade stainless steel pin as a static partner was employed. Further, polarized light optical and scanning electron microscopes (SEM) were utilized for a morphological analysis. Elastic modulus and strength were determined by a compression test. A result analysis is conducted to analyze sliding wear accompanied with minor abrasion. The composites with a high percentage of reinforcement exhibit credible wear resistance and mechanical robustness.

Keywords: carbon; sliding wear; microstructure; composite; friction; pitch; characterization

1 Introduction

Carbon–carbon (C/C) composites are emerging engineering materials traditionally introduced as chief materials utilized in aerospace industries primarily for thermal shielding of reentry spacecrafts and aerospace vehicles [1]. As carbon fiber is now economical and there is an abundance of carbonaceous precursor material, C/C composites are increasingly gaining attention [2]. C/C composites are formed by dispersing load bearing entities, i.e., reinforcement of carbon fiber in a carbon matrix [3]. Upon carbonization, bulk C/C composites are obtained that exhibit superior properties and high rigidity compared with other composites [4]. C/C composites are known for their extraordinary tribo-mechanical response.

Their friction studies primarily comprise of low wear rates and a low friction coefficient [5–6]. Evidently,

upon loading and sliding, a significant amount of energy is introduced to a composite system, and therefore, wear mechanism and estimations become important. C/C composites being used nowadays generally comprise of phenolic resins and have been researched extensively [7–12]. The use of resins as a matrix introduces few problems, for example, processing conditions require continuous attention, and heating and cooling mechanisms are gradual and sophisticated [13, 14]. In contrast, a pitch matrix exhibits flexibility as it is isotropic in nature and can bear high heating and cooling rates. A very few references are available in the field of C/C composites entirely made from pitch without a reimpregnation process [15]. The friction behavior of C/C composites is thus far unique among those of other engineering materials. C/C composites developed from pitch are turbostratic carbon; hence, their atomic arrangement

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Nomenclature

k	Wear rate as per Archard's equation	T	The absolute temperature (°C)
N	Dead load in newtons	D	Sliding distance
μ	Coefficient of friction	u	Sliding speed
R4	Re-impregnated four cycles		

is comparable to that of graphite. Although graphite is highly crystalline, the turbostratic phase lags the long-range arrangement of the graphitic layers; in other words, the semi-graphitic arrangement is dominant. In the present work, the technique used to fabricate samples helps to remove the barrier of multiple impregnation cycles with conclusive sample densities in the range of 1.59–1.72 g/cc. A process developed to reduce the reimpregnation cycles is carried out by acid treatment of the pitch generally using HNO_3 as a binder and the thermal treatment of the pitch at 380 °C in zero air that is further used as a base matrix. The friction studies of such a single-step fabrication process of C/C composites is crucial as the processing method affects the microstructure and mechanical properties of the whole material itself. Such turbostratic carbon is highly robust and resistive to extremely high temperatures. Their friction characterization is generally based on micro abrasion even under dry conditions. The friction typically outstands other types of composites because both the phases are predominantly carbon and are favorably compatible with each other [16, 17]. The work discloses the friction and wear mechanism of high-performance C/C composites fabricated using a single-step carbonization process via pin-on-disc method; further, the effect of reinforcement weight percentage on various tribological aspects for different loads under dry sliding of 8,000 m.

2 Experimental

2.1 Friction and wear test

Studies were conducted on a CSM pin/ball-on-disc tribometer at room temperature ($T = 21 \pm 1$ °C). The sliding speed (u) was maintained at 0.5 m/s and the stainless steel pin with a diameter of 8 mm covering a distance (D) of 8,000 m was used. Wear track radii

were fixed at 5, 10, and 15 mm for 5, 10, and 20 N loads, respectively. For weight loss measurement, LIBROR AEG 220, a Shimadzu precision-weighing machine with a resolution of 0.01 mg was employed. The coefficient of friction μ was obtained automatically from the instrument. The characteristic schematic of the complete setup is represented in Fig. 1.

2.2 Fabrication of composites and sample preparation

Cylindrical-disc shaped C/C composites with a diameter of 50 mm and the thickness of 6 mm were fabricated using chemically modified pitch as a binder and thermally treated pitch as a base matrix material. Crude petroleum pitch was treated with HNO_3 to increase the softening point and to impart thermal stability in the pitch such that it can be used as a binder. Thermally modified pitch was obtained by high temperature treatment of crude pitch at 380 °C in zero air environment to remove volatiles from the pitch. Chemically and thermally modified pitch were ground separately using a planetary mill to attain 40 μm consistency. For the hot compression molding of the C/C composites before carbonization, 30 g of

Fig. 1 Schematic diagram of the pin on disc wear setup.

thermally treated pitch and 6 g of chemically modified pitch were mixed with the reinforcement and then compressed in a mild steel mold. The powdered mixture with a different amount of chopped carbon fiber was consolidated at 220 °C, followed by carbonization at 1,000 °C with a 2.5 ml/min flow of nitrogen gas. Further details of the method are presented in an earlier study [18]. The primary characteristics and naming of the samples are summarized in Table 1. With 25% weight fraction of reinforcement, a composite exhibited the last best consolidation. A sample with 30% of reinforcement exhibited dominance of fiber pull outs and an extremely poor performance in mechanical tests. Hence, a set of five samples was fabricated and subjected to wear studies. Figure 2 shows the CAD representation of a consolidation setup and composite samples.

In abbreviation CpC, “Cp” stands for carbon matrix i.e., petroleum pitch while preceding “C” represents chopped carbon fiber reinforcement. Numeric 5, 15, 10, 20, and 25 represent the weight percentage of chopped fiber reinforcement.

3 Results and discussion

3.1 Coefficient of friction

Figure 3 shows a graph between the coefficient of

friction (μ) and the reinforcement weight percentage for varying loads. Evidently, as the fiber weight increases, there is a relative increment in the coefficient of friction. The increasing pattern suggests that, as the fiber percentage increases, resistance to the motion also increases, hence the coefficient of the friction increases. Sample CpC25 contains the highest weight percentage of carbon fiber reinforcement which gives rise to the surface asperities, and henceforth, the roughness. These asperities are rigid by nature and are highly orientated hard segments of crystalline carbon, i.e., carbon fibers. Such covalently bonded carbons are extremely stable with a high degree of crystallinity and coherence in atomic arrangement that offer an extraordinary modulus and excellent hardness. For that reason sample CpC25 exhibits a coefficient of friction as high as 0.038, 0.075, and 0.156 at 5, 10, and 20 N, respectively. For a sample containing 5% reinforcement, i.e., CpC5, the matrix is dominating; hence, the smooth surface offers low friction coefficient values, which are 0.028, 0.065, and 0.133 at loads 5, 10, and 20 N, respectively. This is also attributed to the fact that a matrix upon carbonization transforms into turbostratic or semi-graphitic carbon that provides a lubricating effect and is primarily observed in composites exhibiting a low fiber weight percentage.

Table 1 General characteristics of fabricated carbon carbon composites.

No.	Sample code	Weight percentage of reinforcement	Density (g/cc)	Practical density (g/cc)	Rockwell hardness (HRC)
1	CpC5	5	1.593	1.581	53
2	CpC10	10	1.612	1.625	56
3	CpC15	15	1.670	1.683	63
4	CpC20	20	1.672	1.710	67
5	CpC25	25	1.711	1.725	71

Fig. 2 (a) CAD representation of the mild steel die, (b) CAD representation of the hot compression molding, and (c) composite sample before and after the wear test.

Fig. 3 Coefficient of friction at varying loads.

3.2 Weigh loss

Weight loss data, summarized in Table 2, help in interpreting the stages of material loss during the tests. Figs. 4–6 represent the weight loss of composites at

various loads; a pattern is attained in the graphs indicating that a low percentage of reinforcement results in high material loss and vice versa. After covering a significant distance, the weight loss is perceived to be almost independent of the distance for composites CpC15, CpC20, and CpC25. Specimens become stable and their weight loss with respect to distance remain unaffected (Figs. 4–6). This behavior is observed in all the three systems after covering a distance of 6,200 m, because upon removal of the superficial surface of low-order carbon matrices, the reinforcement appears to interact with the pin. Further, carbon matrices and reinforcement exhibit high bonding and compatibility with each other along with an extremely high wear resistance owing to their phase similarities [19–21].

The composites are resistant to sliding wear owing to the synergistic tribo-response. From this behavior, it is evident that reinforcement with stronger mechanical properties bind more strongly with the matrix and prevent material loss than those without.

Table 2 Detailed tribo-characteristic of composites at different loads of 5, 10, and 20 N.

Sample code	COF			Net weight loss (over the sliding distance $D = 8,000$ m) (g)			Wear volume (mm^3)		
	5 N	10 N	20 N	5 N	10 N	20 N	5 N	10 N	20 N
CpC5	0.028	0.065	0.133	0.0140	0.0185	0.0204	8.788	11.613	12.806
CpC10	0.031	0.067	0.134	0.0104	0.0130	0.0182	6.451	8.064	11.290
CpC15	0.033	0.072	0.146	0.0041	0.0050	0.0134	2.455	2.994	8.023
CpC20	0.034	0.072	0.156	0.0025	0.0039	0.0079	1.495	2.332	4.724
CpC25	0.038	0.075	0.156	0.0020	0.0023	0.0051	1.168	1.344	2.980

Fig. 4 Weight loss of samples at 5 N load.

Fig. 5 Weight loss of samples at 10 N load.

Fig. 6 Weight loss of samples at 20 N load.

3.3 Wear rate

Wear rate (k) helps resolve the wear and theories of predicting models, further, it correlates material loss with respect to distance [22, 23]. Wear rates are calculated using Archard's equation represented pictorially in Fig. 7. Even though the relation was established for an adhesive type of wear, it is valid and extensively used for modeling abrasive, sliding, erosive, and other types of wear and fretting mechanisms. Samples were weighed at the intermediate sliding distances during the wear test. Specimen CpC5 exhibited the maximum wear rates: 10.98 E-04 , 14.51 E-04 , and $16 \text{ E-04} \text{ mm}^3\cdot\text{m}^{-1}$ at 5, 10, and 20 N, respectively. High wear rate is observed because of the lack of reinforcement, hence the matrix is free to wear off and is not held stable by fibers unlike in sample CpC25. Severe wear rate, which is 16 E-03 , is observed

Fig. 7 Wear rates of various composites.

in sample CpC5. On the contrary, specimen CpC25 exhibits minimum wear rates: 1.46 E-04 , 1.68 E-04 , and $3.72 \text{ E-04} \text{ mm}^3\cdot\text{m}^{-1}$ at 5, 10, and 20 N, respectively. This is supported by the fact that the presence of reinforcement, i.e., the load bearing phase holds the matrix all together and prevents material loss. Table 3 and Fig. 7 indicate that at high loads, there is a corresponding increment in the wear rates.

3.4 Polarized light optical micrographs

Polarized light optical microscopy is an essential imaging technique that helps carry out an in-depth morphological analysis of optically active materials. Figures 8 and 9 are Pol-optical micrographs of the CpC20 and CpC10 samples, respectively. The region marked as "1" in the images is the normal surface, whereas region "2" is the worn surface. In Fig. 8, the highly reflecting fibers are distinctly visible, representing resilience and endurance against the sliding wear. The optical examination indicates that the reinforcement in the sample offers higher wear resistance

Table 3 Wear rates at different loads of various composites.

Sample	Wear rate E-04 (mm^3/m)		
	5 N	10 N	20 N
CpC5	10.98	14.51	16
CpC10	8.06	10.08	14.11
CpC15	3.06	3.74	10.02
CpC20	1.86	2.91	5.9
CpC25	1.46	1.68	3.72

Fig. 8 Polarized light optical micrograph of sample CpC20.

Fig. 9 Polarized light optical micrograph of sample CpC10 representing voids from chipping along with mild abrasion.

than the matrix alone; further, with an increment in reinforcement, the wear resistance also increases. Furthermore, it is interesting to notice in Fig. 8 that the wear is high where the reinforcement is less. The optical micrograph of specimen CpC10 in Fig. 9 shows the presence of minor scratches caused by removal of the worn debris rubbed against the surface, suggesting the minor mechanism of wear by abrasion. Carbon is a material that offers extremely strong mechanical properties such as hardness. Carbon matrix and fiber reinforcement are the hard segments of C/C composites and during the rubbing of worn debris against the composites. In such a situation, the worn debris acts as an abrasive and contributes to the scratches, which are vivid in the micrographs. The major wear mechanism appears to be caused by the sliding of the surfaces, as it is evident from the smooth regions, material loss, and reflective surface of the wear tracks.

3.5 Highlighted optical micrographs

Figures 10 and 11 represent a part of the worn area of samples CpC25 and CpC5, respectively, thereby making it is easy to differentiate the worn region in the composite samples by coloring the different planes with different reflectivity. The software works on the principles of reflectivity of surface; the blue region, which is the normal surface, offers the diffused reflection, while the region worn by sliding provides

Fig. 10 Highlighted optical micrograph of polished wear track of sample CpC25.

Fig. 11 Highlighted optical micrograph of wear track of sample CpC5 showing minor polishing and major erosion along the wear track.

the specular reflection colored in red. Region 2 in Fig. 10 represents the polishing of the wear track caused by sliding. The blue region in Fig. 11 along the wear track represents the material loss and it is significant in composite CpC5. In contrast, the red region caused by surface polishing is significant in composite CpC25, as shown in Fig. 10. The colored sections help visually understand the resilience of the composite, which indicates that the composite CpC25 suffered lower wear than that of CpC5. Their response is attributed to the synergetic effect of the matrix and reinforcement interaction that is high for composites with a high weight percentage of carbon fiber.

3.6 Scanning electron micrographs

The scanning electron micrographs show the wear tracks; the worn region is marked, and the wear debris are highlighted in bright circles. As depicted in Fig. 12, the track appears smooth and scar free with some patches, signifying the chipping as a result of continuous sliding. The high reflectivity of the track is a result of the removal of the less ordered and low density carbon leaving behind the stable turbostratic phase. Additionally, in Fig. 12, no significant deep wear marks are observed in comparison with Fig. 13; this may be attributed to the high hardness of the specimen and the presence of a higher percentage of

Fig. 12 SEM micrograph of sample CpC25 representing the smooth surface caused by sliding, and wear voids caused by removal of matrix flakes

Fig. 13 SEM micrograph of sample CpC5 showing significant material loss.

reinforcement [24–26]. Upon analyzing the microstructure, it was observed that in Fig. 12 the fibers are well impregnated in the matrix and the synergetic effect can be witnessed. The enlarged circular region shows the signatures of the fiber matrix bonding, which contemplates the endurance against the wear. Although deep scars are observed in Fig. 9, because the sample under study is CpC5 at 20 N load, which has the least weight percentage of the reinforcing phase, the wear becomes prevalent.

Also, the hardness of sample CpC5 is comparatively lower than that of the other composite samples. This makes the pin scar the surface during the test. In composite CpC5, the fiber percentage is less, hence it exhibits a combination of erosion and sliding wear.

Deep scars and ploughing are caused because of the erosion of the matrix by the edges of the pin, whereas chipping is primarily caused due to the sliding of the pin against the composite. Further analysis revealed that the region of the fiber matrix interaction was not as good as that in composites CpC25 and CpC20, which is the most likely cause for the low resistance against wear in the prolonged test. However, it is interesting that deep scars and ploughing, hence wear by erosion accompanied by sliding wear, are only observed in composite CpC5. C/C composites are similar to graphite and exhibit self-lubrication as these samples are semi-graphitic carbons. Therefore, the static wear partner merely slides on the surface without any major wear of the samples containing a higher percentage of the reinforcing phase, and simultaneously smoothens the surface with minimalistic wear. The wear tracks appear to be highly stable dynamically with no substantial material loss, as verified by the weight loss study. However, a similar argument cannot be made for samples CpC5 or CpC10.

3.7 Wear mechanism: microstructure of the worn debris and wear track

Grooves by ploughing suggest that the composite CpC5 exhibits wear by erosion accompanied by sliding wear, whereas the delamination of the surface layer, polishing, and chipping are distinctly seen in composites CpC25 and CpC20. This can be observed in

the optical micrographs, i.e., Figs. 10 and 14 which demonstrate the domination of the wear mechanism by sliding. For further confirmation of the wear mechanism, a scanning electron micrograph of the worn debris and the optical micrograph of the track were recorded, as illustrated in Figs. 14 and 15. The presence of the sharp debris of the matrix in Fig. 14 indicates the dominance of the erosive wear mechanism in specimen CpC5. The sliding wear accompanied by minor abrasion is evident from the polishing of the surface and scratches in composite CpC15, as depicted in Fig. 15. Similitude is also observed in composites CpC10, CpC20, and CpC25 as evident from Figs. 9, 8, and 12, respectively.

Fig. 14 SEM image of the worn debris of CpC5 showing chunks of eroded matrix.

Fig. 15 Optical micrograph of worn track of CpC15 showing sliding wear and insignificant scratches caused by mild abrasion.

3.8 Comparison between cyclic impregnated and non-impregnated C/C composite

For comparison purposes, the best performing C/C composite was chosen from the set, i.e., CpC25, and the results were compared with the C/C composite developed by four cycles of reimpregnations. The composite was named as R4CpC25, where “R4” indicates that reimpregnation was conducted four times. While the coefficient of friction nearly remained the same, there was a significant increment in the wear rates. It was observed that there was an increment of 3.42%, 4.76% and 7.25% in the wear rates at 5, 10 and 20 N, respectively. The data are presented in Table 4. The increasing trend in the wear rates is the consequence of the layer by layer growth of the composite interface after each cycle. During carbonization, the porosity and the vacant spaces generated in the composite are later filled by the impregnation pitch [27]. This is a result of the weak layer by layer formation of the composite, which causes high wear rates. In composites developed without the reimpregnation process, the monolithic carbon matrix dispersed with the carbon fiber reinforcement offers superior wear resistance than the composite developed by the cyclic reimpregnation process.

Table 4 Comparative data of the friction and wear tests of CpC25 and R4CpC25.

Load (N)	CpC25			R4CpC25		
	COF	Wear volume (mm ³)	Wear rate E-04 (mm ³ /m)	COF	Wear volume (mm ³)	Wear rate E-04 (mm ³ /m)
5	0.038	1.168	1.46	0.029	1.210	1.51
10	0.075	1.344	1.68	0.070	1.412	1.76
20	0.156	2.980	3.72	0.161	3.195	3.99

4 Mechanical response of various composite samples

C/C composites are known for their robust mechanical response. Therefore, mechanical studies were performed to evaluate the compressive strength and modulus of different C/C composites. Samples were cut in a rectangular shape with 10 mm × 10 mm × 6 mm dimensions ($L \times B \times H$) and loaded with uniaxial

compressive load with 0.2 mm per minute crosshead speed [28]. The compressive strength was the highest for sample CpC25, i.e., 80.07 MPa, and was the lowest for CpC5, i.e., 25.45 MPa. The data is enlisted in Table 5. Similitude was observed in the elastic modulus which is represented in box individually for each composite in the stress–strain response under compressive loading i.e., Fig. 16. The elastic modulus is the highest for composite CpC25, i.e., 10.8 MPa, whereas that of CpC5 is 3.34 MPa. The loading capability and toughness of the composites demonstrate an increasing trend with the increasing fiber percentage.

Table 5 Mechanical response of composite samples under compression test.

Sample code	Compressive strength (MPa)	Elastic modulus (MPa)
CpC5	25.45	3.34
CpC10	31.23	4.54
CpC15	43.00	5.77
CpC20	74.66	10.6
CpC25	80.07	10.8

Fig. 16 Compression behavior of composite samples; block values represent the elastic modulus.

5 Hardness, density, and wear rates

To summarize the overall response of the C/C composites, a relationship plot defining the physical, mechanical, and tribological aspects, i.e., the wear rate of the composites, was plotted for different samples at various loads. The data plots are represented in Figs. 17(a)–17(c) at loads of 5, 10, and 20 N, respectively.

The three systems exhibit a similar trend and mechanical behavior (hardness and physical properties). The density of the composites increases and as a result,

Fig. 17 Plot wise presentation of wear rate, hardness, density, and fiber weight relationship at (a) 5 N, (b) 10 N, and (c) 20 N.

the tribological behavior, i.e., the wear rate, becomes detrimental.

6 Conclusions

This study confirms that the coefficient of friction increases with an increasing load and fiber reinforcement. On the contrary, the wear and wear rates become detrimental with an increase in the fiber percentage. It is also established that the composites with a higher percentage of reinforcement are mechanically robust. Fibers oriented in the arbitrary direction hold effectively well with the matrix and impart wear resistance. Intermediate minor abrasive wear is also observed because of the removal of the matrix debris; however, its contribution appears insignificant with the overall dominance of the sliding wear. Composite CpC25 performs the best among all the systems, and offers superior wear resistance when compared to R4CpC25. The wear rates of CpC25 are 3.42%, 4.76%, and 7.25% lesser than those of R4CpC25 specimen at 5, 10, and 20 N, respectively.

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