Friction and wear behavior of GNPs functionalized carbon fiber reinforced polymer matrix composites under high-frequency reciprocating conditions

V K Srivastava, Parshant Kumar, Till Quadflieg and Christoph Greb

1 Department of Mechanical Engineering, Indian Institute of Technology (Banaras Hindu University) Varanasi-221005, U.P., India
2 Institute for Textile Technology and Engineering, RWTH Aachen University, Otto-Blumenthal Stresse-1, 52074 Aachen, Germany

E-mail: vijayks210@gmail.com

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Abstract
The effect of normal load on the reciprocating sliding behavior of plain weave woven carbon fibers (CF) and graphene nanoplatelets (GnPs) coated unidirectional carbon fiber strands (GCF) reinforced polymer (CF-GCF/epoxy) composites was investigated. The normal load was varied from 50 N to 90 N at the interval of 10 N. The high frequency of reciprocating motion (5 Hz) was considered. Nearly 13,500 cycles were employed with a stroke length of 1.5 mm. The tensile behavior of CF-GCF/epoxy composite was also investigated keeping longitudinal direction of GCF along the loading direction. The fractured samples and worn surfaces were analyzed under scanning electron microscopy. It was observed that the surface of GCF was rough which increased its interlocking tendency with the matrix and hence increased the tensile strength. The friction coefficient increased with increase of load up to 80 N and decreased afterward, whereas wear loss exhibited increasing trend with increase in load. However wear loss showed a slight decrease in slope beyond 70 N load. Fiber fragments in wear debris were observed at high loads. Although the friction film was also observed at high loads, the abrasion furrows were not significantly visible. The plasticity of matrix and the formation of friction film depicted the friction and wear behavior of CF-GCF/epoxy composites.

1. Introduction

Polymer composites have been widely used as friction parts in the aerospace sector due to their low density, self-lubrication properties, high wear resistance, good mechanical properties, excellent irradiation resistance, chemical inertness, and ease of manufacturing [1]. In the wide plethora of polymer composites, fiber reinforced polymer composites (FRPs) have attracted a great attention as friction materials due to their stable friction behavior in various environments [2]. Among various FRPs, carbon fiber reinforced polymer composites (CFRPs) have aroused a considerable research interest in both industrial and scientific communities due to their high modulus and strength, ease of recycling and low density [3]. The interfacial adhesion at the interface of carbon fiber and polymer matrix plays an important role in depicting the mechanical and tribological properties of CFRPs. The highly stable, non-polar and smooth graphitic surface of carbon fiber may lead to low adhesion at fiber/matrix interface. Thus surface functionalization of carbon fiber is usually performed to achieve high level of interfacial adhesion.

The superficial modification of carbon fiber by carbon-based nanomaterials may enhance its adhesion with polymers. For instance, the addition of graphene oxide sheets in the sizing solution of fibers may strongly enhance the interfacial adhesion between carbon fiber and polymer matrix and thereby, their mechanical properties [4]. Also, the incorporation of graphene nanoplatelets (GNPs) at the fiber-matrix interphase affects the mechanical and electrical properties of the composite [5]. The van der Waals interactions between different graphene layers in GNPs tend them to stack and aggregate. Thus, local stresses are developed when an external force is applied to it. Srivastava et al. [6] used photoelastic analysis to study the stress field around GNPs coated...
carbon fiber strand reinforced in polymer matrix, and concluded that a strong fiber/matrix interface could be obtained after functionalization of carbon fiber with GnPs, due to which its properties are strongly affected. Mechanical, thermal and tribological properties of FRPs are affected by the strength of interphase [7]. An optimal adhesion strength may lead to a reduction in the ejection tendency of fiber during tribological interactions [8]. Lee et al [9] showed that the fracture toughness of fiber/matrix interface is higher than that of either of its constituents, and the fracture in the reinforcement is not favorable, the generated wear debris will be small which may result in improved wear resistance. Thus, to enhance the wear resistance of carbon fiber composites, GnPs can be coated on it which increases the interfacial adhesion and eventually wear resistance. However, the incorporation of GnPs at the interphase region increase the overall cost of the final product. Thus to reduce the cost, hybrid composites with two types of fibers could be employed to serve the purpose. Several authors have investigated hybrid composites for various applications [10, 11]. Carbon fibers (CF), as mentioned earlier, has gained a significant attention in industrial and scientific communities. Thus, a hybrid composite of carbon fibers and GnPs coated carbon fiber strands can be investigated for its tribological behaviour. The frequency of reciprocating sliding motion affects the tribological behavior of FRPs. Mathew et al [12] observed a sharp increase in wear loss at high frequencies (>2 Hz) as compared to low frequencies (<2 Hz) in case of textile composites. Sarkar et al [13] observed that frequency affects both wear loss and friction coefficient. Some authors [12, 13] also showed that the applied load have a significant impact on the tribological response of fiber reinforced composites. Thus, in the light of above, the reciprocating sliding behavior of woven carbon fibers and GnPs coated carbon fibers (CF-GCF) reinforced polymer matrix composites have been investigated in the present article, at high-frequency conditions. The main objective of this study is to understand the dominant friction and wear mechanisms, and the applicability of CF-GCF hybrid polymer composites for high-frequency tribological applications.

2. Materials and methods

2.1. Materials
Carbon fiber strands were coated with graphene nanoplatelets/epoxy resin mixture which contained 1 wt% of graphene nanoplatelets. A spray deposition technique was used for coating and the diameter of the fiber strand was ∼0.5 mm. Each fiber strand contained numerous carbon fibers having ∼8 μm diameter. After coating carbon fiber strand, hand layup method was used for the fabrication of woven fabric composites. A standard DGEBA (diglycidyl ether of bisphenol-A) epoxy resin was chosen as matrix material. The plain weave cross-woven (0°/90°) high strength carbon fiber mats, and the carbon fiber strands were reinforced in epoxy resin.

2.2. Fabrication of composites
Hand layup method was used for the fabrication of composites. Firstly, a standard DGEBA epoxy resin (PL411) and a curing agent (hardener, PH861) were thoroughly mixed in a container in the ratio of 10:1 by weight. Afterward, two cross-woven carbon fiber mats were laid up with epoxy resin mixture applied in-between the layers and then, the third layer of unidirectional GnPs coated carbon fiber strands was laid. The lay-up scheme for CF and CF-GCF is shown in figure 1(a.i). The distance between the GCF strands was maintained to be 1.5 mm. After laying 8 layers of CF mats and 3 layers of GCF strands, a pressure of 10 KPa was applied on the top face to prevent any accumulation of epoxy resin and dry patches of fibers. The CF-GCF/epoxy composite was left for curing in compressed condition for ∼20 h. After that, it was left for curing in ambient environment for ∼24 h. The surface of the final composite plate is shown in figure 1(a.ii). After curing, samples were prepared from the composite plate.

2.3. Sample preparation and testing
Samples were prepared for tensile and tribological tests. For tensile testing, ASTM D3039 standard was followed. The room temperature tensile tests were performed on universal testing machine at a cross-head speed of 2 mm.min⁻¹. To perform reciprocating wear tests, samples were prepared in the form of cylindrical pins having a diameter of 9 mm. The ASTM G133 standard was followed for linearly reciprocating wear tests. To prepare cylindrical pins, the composite samples were firstly cut in the form of square section of required dimensions using diamond cutter. The square sections were then manually formed to circular shape using emery papers of different grades, i.e. 400, 1000 and 1200. The composite samples used for reciprocating wear testing were formed in such a way that all the CF mats were lying parallel to the contact surface and GCF strands were lying in the antiparallel direction as shown by schematic in figure 1(b). Chrome steel was used as a counter surface material having hardness of 62 HRC. Reciprocating wear tests were performed on a reciprocating friction monitor (TE 200ST, Magnum Engineers, Bangalore, India). The schematic illustration showing reciprocating friction
monitor is shown in figure 2. The stroke length was kept constant as 1.5 mm. The frequency of reciprocating motion was set to 5 Hz, and 13 500 cycles were employed. Wear loss and friction coefficient were investigated. The normal load was varied from 50 N to 90 N in the steps of 10 N. All the tests were performed at an ambient temperature of 29 ± 3 °C.

2.4. Scanning electron microscopy
To gain an understanding of the possible wear mechanisms, samples were analyzed under a scanning electron microscope. A ZEISS EVO 18 RESEARCH (20 kV) scanning electron microscope was employed to analyze the worn surface.

3. Results
3.1. Tensile testing
Figure 3(a) shows the stress-strain curve of the CF-GCF/epoxy composite under axial loading conditions. A dip in stress was observed as indicated by point A. After the dip, the stress value increased again. The ultimate tensile strength (UTS) was observed to be 240.28 MPa. The strain at fracture was observed to be 0.113, and the value of fracture stress was 46.07 MPa. The composites without GCF were also fabricated and it was found that tensile strength of carbon fabric reinforced epoxy composite (CF/epoxy) was lower than that of CF-GCF/epoxy composite as shown in figure 3(b).
3.2. Reciprocating wear tests

Figure 4 shows the variation of friction coefficient with time when the loads were 50 N and 70 N. These plots show the representative plots for all the tested loads. It can be observed that the fluctuations in friction coefficient were less. At low loads, initial friction coefficient was low and increased with time, whereas opposite occurred at high loads. Figure 5(a) shows the variation of friction coefficient with normal load. It can be observed that friction coefficient increased with increase in normal load up to 80 N, and decreased at 90 N. The increase in friction coefficient was sharp when the load was increased from 60 N to 70 N. The variation of wear loss with the normal load is shown in figure 5(b). The almost linear relationship of load and wear loss was observed up to 70 N after which, the slope of wear loss decreased slightly.

4. Discussion

The surface of the GNPs coated carbon fiber strand was rough (as shown in figure 6) which resulted in an increase in the interlocking tendency of fiber strand with epoxy matrix [5]. The stiffness of carbon fiber and epoxy are significantly disparate. Thus, the crack generally initiates at the fiber/matrix interface. However, in case of GCF, the functionalized surface led to the deviation of cracks at the interphase region along parallel and perpendicular to the interface, which prevented the sudden failure of the composite [14], and resulted in dip(s) in stress-strain curve, as shown by point A in figure 3(a). This increased the strain to failure.

Figure 7 shows a bunch of carbon fibers in the fractured strand. It can be observed that there was not a single plane of crack propagation. The change in plane required more stress level for the propagation of crack. That is why slight increase in stress was observed even after first dip (point A) in the stress-strain curve.
The reciprocating sliding behavior of CF-GCF/epoxy composite revealed that friction coefficient increased with increase in time at low loads, whereas opposite was observed at high loads, as shown in figure 4. The increase of friction coefficient with time may be attributed to the plasticization of matrix due to rise in temperature, as the time elapsed. The plasticization increased the adhesion tendency of matrix material to the
counter surface which resulted in an increase in friction coefficient. The removal of matrix material due to adhesion can be observed in the composite sample tested at 50 N load, as shown in figure 8.

After delamination of matrix material due to adhesion, fibers were exposed to the counter surface. However, it was depicted that the direct contact of exposed fibers and counter surface did not occur at low loads. The cracks in the matrix were also observed, as shown in figure 8. These cracks were induced due to repeated sliding over the same region in the opposite direction. The direction of sliding changed after each stroke (i.e. forward and backward stroke). Thus the sign of shear stress developed in the contact area was changed after each stroke which led to the generation of cracks, usually normal to the direction of sliding.

The friction coefficient slightly decreased with time at high loads. However, the initial friction coefficient was higher at high loads as compared to low loads. The initial high value of friction coefficient was attributed to deeper penetration of asperities. Braking energy was also increased with increase in load which resulted in an early rise of temperature [15]. Hence matrix was plasticized at the early stage which led to adhesion with the counter surface and delamination from the surface of the composite, as shown in figure 9(a). Therefore, CF-GCF/epoxy composite showed high friction coefficient in the initial cycles. After a few numbers of cycles, friction coefficient reduced slightly. This reduction was attributed to the generation of wear fragments of the carbon fibers. The direct contact of counter surface with the exposed fibers after matrix delamination resulted in fracture of fibers in small fragments, as shown in figure 9(b). The fiber fragments acted as third body particles. Due to confined area sliding in reciprocating motion, the fractured fiber fragments repeatedly rolled in between the interacting surfaces. Some of the fiber fragments even pulverized at high loads and formed a friction film on the surface (as shown in figure 9(b)) due to which the friction coefficient decreased, as the time elapsed.

Figure 8. SEM image showing the worn surface of CF-GCF/epoxy composite tested at 50 N load.

Figure 9. SEM image of worn surface of CF-GCF/epoxy composite tested at 70 N load showing (a) Delamination wear, and (b) Fiber fragments.
The friction coefficient increased up to 80 N load. This increase was attributed to the increase in braking energy which may lead to an early rise in temperature and hence, increased the adhesion component of friction coefficient. Although abrasion component of friction coefficient also increased due to deeper penetration of asperities at high loads, they were not significantly visible on the worn surface. Despite abrasion furrows, some interconnected ligands were observed, as shown in figure 10. These ligands seem to fill the grooves which were generated due to abrasion. The origin of ligands may be attributed to the plastic flow and thermal expansion of matrix due to rise in contact temperature with increasing load. When the load was increased to 90 N, the friction coefficient got reduced. This decrease was attributed to the ease of formation of friction film at high loads due to readily generation of fiber fragments and confined area sliding which led to pulverization of fiber fragments. The observed friction film and fiber fragments at 90 N load are shown in figure 11.

The wear loss varied linearly with load up to 70 N after which, a slight decrease in slope was observed. The increase in wear loss was attributed to the increase in penetration of asperities and braking energy as explained earlier for friction coefficient. The slight decrease in slope was due to the formation of friction film which reduced the adhesive wear.

5. Conclusion

Based on the experimental observations on the tensile, and reciprocating wear and friction tests of woven carbon fibers and GNPs coated carbon fibers reinforced polymer composites, following conclusions were drawn from the present study.
1. The coating of GNPs increased the surface roughness of carbon fiber strands which increased the interlocking tendency of fiber strand with the matrix.

2. The crack changed plane in the strand itself, which required more stress level for its propagation, and hence increased tensile strength.

3. The increase in adhesion tendency due to the early rise in temperature at high loads tend to increase the friction coefficient in initial cycles. However, the opposite was observed at low loads.

4. Due to the pulverization of wear fragments and formation of friction film, the friction coefficient decreased when the load was increased beyond 80 N.

5. Wear loss followed a linear trend with an increase in load up to 70 N load after which, it decreased slightly due to formation of friction film.

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ORCID iDs

V K Srivastava  https://orcid.org/0000-0001-6765-6985
Parshant Kumar  https://orcid.org/0000-0002-7182-1645

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