Surface tailoring of carbon fabric reinforced PES composites with PTFE filler in micro and nano- sizes

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ABSTRACT

Effect of surface tailoring on mechanical and tribological properties of plasma treated carbon fabric (CF), used as reinforcement, to develop composites is reported in this paper. CF being inert does not have good adhesion with matrix. Cold remote nitrogen oxygen plasma (CRNOP) was used for surface treatment to enhance their adhesion with the matrix, polyethersulphone (PES) of higher molecular weight. Composites with twill weave carbon fabric (CF) (≈67-70 wt %) were developed using impregnation technique followed by compression molding. Further, the top two layers of composites with best properties were tailored with 2wt % PTFE fillers in micron and nano-scale range. Tribo performance of all composites was evaluated on UMT tribometers by sliding of composite pin against silicon carbide (SiC) paper; improved wear resistance (W_R) and reduced Coefficient of friction (μ). Worn surfaces of the pin and disc were studied with SEM to understand wear mechanisms.

Keywords: PES-CF composites, solid lubricants, plasma treatment, wear.

1. INTRODUCTION

Carbon fabric reinforced polymers (CFRPs) are being increasingly used as advanced composites for structural materials applications, especially in aircraft industry because of very high specific strength and modulus apart from their higher thermal conductivity, corrosion resistance etc [1]. In spite of excellent properties of CF such as very high strength, modulus, thermal conductivity, lubricity etc. it suffers with serious drawback. These are inert towards matrix and show little adhesion. Various surface treatments are reported to be successful to enhance adhesion either by mechanical interlocking or grafting various functional groups which interacts with matrix and improve adhesion. Plasma treatment is a well known technique to improve adhesion by incorporating functional groups on CF [2]. It is less destructive method and allows a greater control over the number of unwanted reaction pathways. It has been reported that such treatments proved beneficial for improvement in mechanical properties of some polymers and composites and also for tribological properties in adhesive and fretting wear mode [3, 4]. However, for abrasive wear studies such efforts are not reported.

Moreover, it is well known fact that addition of solid lubricants such as PTFE in polymers and composites proves beneficial in improving friction and wear properties. However, inclusion of such inert and low surface energy filler leads to deterioration in mechanical properties significantly.

Hence it is wiser to tailor the surfaces with solid lubricants rather than bulk. Not much is reported in this regard in the literature [5]. Hence in this work, efforts were also made to tailor the surface of composite with solid lubricant PTFE in two sizes viz. micro and nano. Nano particles were tried since these are reported to be more beneficial as compared to micro-size with equal loading [6, 7].

In current work, the influence of cold remote nitrogen oxygen plasma treatment on fabric and subsequently on its PES composites on physical, mechanical and abrasive wear properties is investigated. Further efforts of surface tailoring composite by using PTFE fillers in micron and nano-scale range and their subsequent influence on abrasive wear performance are also reported.

2. FABRICATION OF COMPOSITES

PES polymer GAFONE B3500 was used as matrix material. (properties as shown in Table 1).

Melt Flow Index (MFI) (g/10min) ASTM D 1238	Weight-average molecular (M_w)*	Polydispersiy (M_w/M_n)
8.5 ± 0.2	39,825	2.64

*Supplier's data

Table 1. Melt flow rate (MFI) and molecular wt of PES.

Carbon fabric of twill weave supplied by Fiber Glast USA was used as reinforcement for development of composites by impregnation technique. Cold remote nitrogen oxygen plasma (CRNOP) was employed for surface treatment of chemically inert CF to enhance its reactivity towards matrix materials [8].

Dichloromethane (CH_2Cl_2) was used as a solvent to prepare the solution of PES (20 wt %). Twenty fabric plies were then immersed for 12 hrs in a sealed steel container filled with viscous solution of PES in CH_2Cl_2. The prepregs were taken out carefully to avoid the disturbance in weave and dried in oven for 1 hour at 100^0C in a stretched condition. Twenty such prepregs were then stacked in the mould carefully to avoid misalignment. Compression molding was done at pressure of 7.3 MPa, temperature of 380-390^{0}C. The composites were then cooled in compressed condition and then cut with the help of diamond cutter as per ASTM standards for different mechanical and tribological characterizations.

The developed composites were designated as follows : C_U, C_T (subscripts U and T denotes untreated and plasma treated fabrics, respectively), P_N denotes virgin PES polymer and S_{tef} denotes the composite tailored with PTFE fillers.

2.1 Surface Tailoring

Surface tailoring of one composite (C_T, composite with best performance) was done separately by developing top two layers of composites with 2 wt % PTFE fillers (12µm and 30 nm). Prior to impregnation the fillers were well dispersed in solvent using high frequency probe sonification for 20 minutes.

3. CHARACTERIZATION OF COMPOSITES

The physical and mechanical characterizations of all composites as per ASTM standards are shown in Table 2.

Properties	P_N	C_U	C_T
Fiber weight (%) (ASTM D2584)	-	67.5	71.5
Void fraction (vol.) (ASTM D2734)	-	0.47	0.44
Density (g/cm^3) (ASTM D792)	1.37	1.52	1.58
Tensile strength (MPa) (ASTM D638)	83	744	747
Tensile modulus (GPa) (ASTM D638)	3.22	65	74
Strain at break (%) (ASTM D638)	>60	1.1	1.06
Toughness (MPa) (ASTM D638)	3.2	4.1	4.4
Flexural strength (MPa) (ASTM D790)	112	692	736
Flexural modulus (GPa) (ASTM D790)	2.8	54	63
ILSS (MPa) (ASTM D2344)	-	42	46

Table 2. Physical and mechanical properties of CF-PES composites reinforced with virgin and CRNOP treated CF.

3.1 Abrasive wear studies

Abrasive wear studies were carried on tribometer (UMT-3MT), CETR, USA. The tests were carried out in Pin-on-Disc configuration (Fig. 1) on S33HE high torque rotational motion drive loading with 1-100N dimensional force sensor with the resolution of 5mN in a single pass rotational motion.

For bedding purpose, square sized composite pin (10 mm × 10 mm × 3.5 mm) was abraded against silicon carbide (SiC) abrasive paper of 800 grade for uniform contact. The bedded sample after cleaning ultra centrifugally followed by drying was fitted in a holder and was then abraded against SiC abrasive paper of 120 grade (grit size ~118 µm) fixed on the rotary drive with the speed of 8 rpm. The total sliding distance of 1.15 m was achieved by abrading the pin in 5 rotations. Experiments were conducted at different loads (10, 20, 30 and 40 N). After the experiment, pin was again cleaned, dried and weighed with an accuracy of 0.0001g. The experiment was repeated for two times and the average value of weight loss (difference in weight) was used for specific wear rate calculations.

The average coefficient of friction (µ) was recorded with respect to time with UMT test viewer software and average value of µ from the data on five tracks recorded for each experiment was considered. The specific wear rate K_0 was calculated from the following equation;

$$K_0 = \Delta m / \rho L d \ (m^3/Nm) \ldots\ldots\ldots (1)$$

Where Δ m is the weight loss in kg, ρ the density in kg/m^{3,} L the load in N and d the distance abraded in m. The fabric was always parallel to the abrading plane and warp fibers were parallel to the sliding direction.

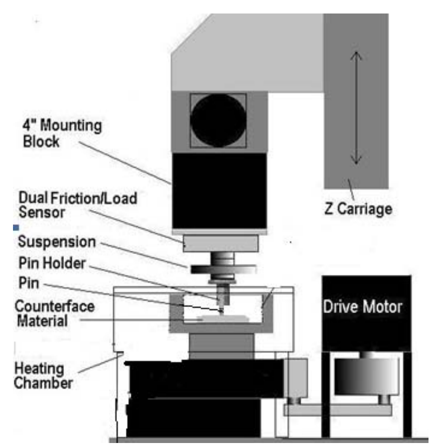

Figure 1. Schematic of Pin-on-Disc Configuration

4. RESULTS AND DISCUSSION

Mechanical properties of pristine polymer and composites are shown in Table 2. High mol wt polymer showed slightly superior properties. Composites based on treated fabric showed better properties than those with virgin/untreated CF. confirming enhancement in fiber-matrix adhesion due to plasma treatment. Most of the properties such as ILSS, flexural strength and modulus, and tensile modulus showed improvement due to enhanced fiber matrix bonding. Specific wear rates (K_0) and Coefficient of friction (μ) as a function of load for selected composites are plotted in Fig. 2 (a, b). Following were salient observations.

- Both wear rate and μ decreased with load for all selected materials.
- CF reinforcement led to significant decrease in friction and wear, both.
- Treated fabric composites led to better performance.
- Surface tailoring by PTFE proved to improve both friction and wear performance in abrasive wear mode, though not significantly.
- There was marginal difference in the performance of micro and nano-sized PTFE when placed on the surface, nano being slightly more effective. Improper dispersion of nano-filler could be one of the reasons for this behavior.

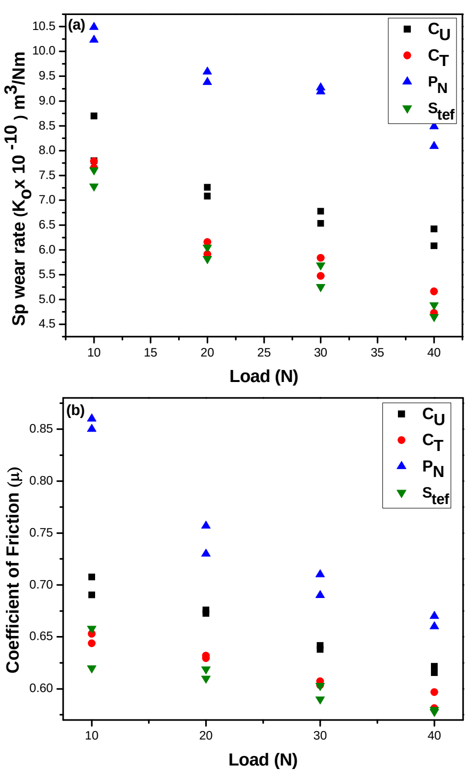

Figure 2. Specific wear rate (K_0) and coefficient of friction (μ) as a function of load for pristine polymer, P_N; composites C_U and C_T and surface tailored composite $S_{tef.}$

It was observed that μ decreased gradually with load from 10 to 40N for all the composites (from 0.9 to 0.5), which is a common trend for polymer composites [9]. At 10N load, the highest μ was recorded for virgin PES (0.84) followed by C_U (0.69), C_T (0.63) composites and surface tailored composite (0.6).

CF reinforcement (untreated and plasma treated) significantly decreased the specific wear rates. At 40N load, the K_0 reduced almost two times due to treated CF reinforcement. When the performance of treated and untreated fabric composites was compared, enhancement was around 15 % in wear resistance (W_R) due to treatment. The trends confirmed that the plasma treatment has beneficially improved the W_R of composite and are in accordance to the mechanical strength properties.

K_0 decreased with increase in load in general and was in accordance to the Lhymn's equation. The increase in W_R due to reinforcement could be due to lubricating effect of CF [10].

4.1 SEM Studies

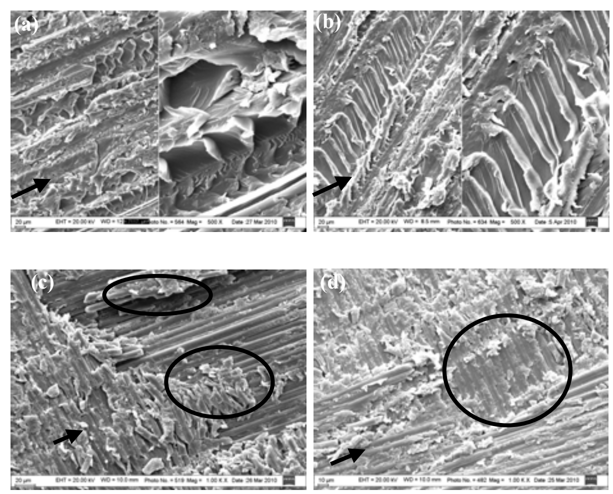

Figure 3. SEM micrographs (X1000) of abraded surfaces of composites worn under loads (a) P_N at 10N, (b) P_N at 40N (c) C_U at 10 (d) for C_T at 10N.

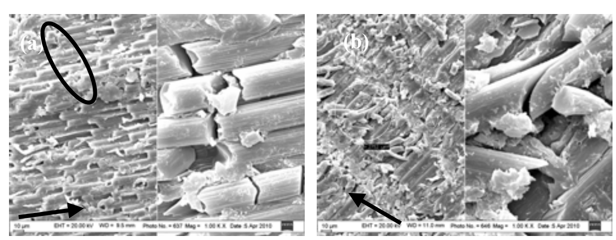

Figure 4. SEM micrographs (X1000) of abraded surfaces of composites worn under 40N load showing various failure mechanisms; (a) Micro cutting and multiple fractures to the fibers lying in perpendicular direction of abrasion; (b) fiber fracture/slicing-lengthwise, micro cracking and debonding.

SEM micrographs of the groves of virgin polymer P_N under extreme loads (10 and 40 N) are shown in Fig. 3 (a and b).

Groves of fatigue striations were observed. Such feature are reported for thermoset polymers during abrasion [11]. For amorphous polymer such as PES, failure is more by fatigue cracks (parallel multiple cracks in the direction perpendicular to stress). The wide furrows in the direction of sliding due to micro-ploughing are also visible. The extent of micro-ploughing by SiC particles and plastic deformation which was the main cause for wear was load dependent. As seen in Fig 3(b), the grooves are deeper and wider. Effect of treatment on fiber matrix adhesion can be seen in Fig. 3(c) and 3(d). Fig. 3(c) shows the less fiber matrix adhesion and most of the fibers are almost without any appreciable amount of matrix. On the contrary, treated fibers show good adhesion. This enhanced adhesion was responsible for less damage to the fibers and hence less wear. Fig. 4(a, b) shows various failure mechanics of fibers in worn composites.

5. CONCLUSIONS

Based on the studies on composites developed with carbon fabric treated with cold remote nitrogen plasma, following conclusions were drawn.

- CF reinforcement showed significant enhancement in all mechanical properties except elongation to break.
- Plasma treatment of CF proved beneficial for improvement in fiber-matrix adhesion leading to more enhancement in mechanical properties.
- CF reinforcement proved beneficial for reducing friction and wear of PES significantly. Treatment of fabric proved more beneficial in this aspect also.
- Surface tailoring of composite is a new technique and offers best combination of mechanical and tribological properties apart from saving the cost of solid lubricant which would have been otherwise required in the bulk. However, in case of abrasive wear, though some improvement was observed due to micro and nano-sized PTFE particles on surface, it was not so significant. Perhaps in adhesive wear mode, the effect would be manifold.

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