Wear 270 (2011) 791–799

Contents lists available at ScienceDirect

Wear

journal homepage: www.elsevier.com/locate/wear

Exploring potential of Micro-Raman spectroscopy for correlating graphitic distortion in carbon fibers with stresses in erosive wear studies of PEEK composites

Mohit Sharma[a], Jayashree Bijwe[a,*], Kuldeep Singh[a], Peter Mitschang[b]

[a] Industrial Tribology Machine Dynamics and Maintenance Engineering Centre (ITMMEC), Indian Institute of Technology Delhi, Hauz Khas, New Delhi, India
[b] Institut für Verbundwerkstoffe GmbH, Verarbeitungstechnik, Erwin-Schrödinger-Str., Geb. 58, Kaiserslautern, Germany

ARTICLE INFO

Article history:
Received 16 August 2010
Received in revised form 21 January 2011
Accepted 10 February 2011
Available online 17 February 2011

Keywords:
Solid particle erosion
Plasma treatment of fibers
Polymer-matrix composite
Micro-Raman analysis

ABSTRACT

Cold remote oxygen nitrogen plasma (CRNOP) treatment was used to enhance reactivity of carbon fabric (CF) towards PEEK. The composite with treated fibers exhibited significantly better mechanical properties due to enhanced fiber–matrix adhesion as evidenced from SEM studies. It was of interest to examine the effect of the treatment on erosive wear performance with variation in angle of impingement and under elevated temperatures. The treatment proved successful in imparting wear resistance to the composite as compared to that with untreated CF. Efforts were made to study correlation between mechanical properties and erosive wear behavior at high temperature. Micro-Raman spectroscopy (MRS) was used to analyze the effect CRNOP treatment on CF and also to study stresses introduced during the erosion of CF-PEEK composites at different angles of impingement. Fairly good correlation was observed in wear rate and strain produced on the surfaces of fibers. SEM was used to understand wear mechanisms.

1. Introduction

Erosion is a serious problem in various situations such as gas turbines, rocket nozzles, cyclone separators, valves, pumps, boiler tubes etc. Erosive wear studies on polymer composites have attracted a lot of attention in the recent years [1–3]. The main impetus for these studies has come through their extensive use in a variety of applications as structural materials in pipe-lines carrying sand slurries in petroleum refining industries; gear cases for locomotives, surfing boats, conveyor belts, helicopter blades, tyres, radomes, pump impellers in mineral slurry processing etc. [4].

Now-a-days, specialty thermoplastics such as polyetherether-ketone (PEEK), polyethersulphone (PES), polyimides (PIs), polyetherimide (PEI) etc. are widely being used to develop high performance composites based on various fiber reinforcements such as carbon, glass, aramid etc. to improve performance properties; mainly mechanical and tribological ones. PEEK, a high performance semi crystalline polymer is known for having outstanding thermal stability, mechanical properties, resistance to wear and chemicals; hence rated as one of the most favored specialty polymer matrices for developing high performance composites [5]. Carbon fibers being multifunctional are most favored as reinforcement for enhancing performance properties. Instead on unidirectional fibers, bidirectional (BD) reinforcement in the form of fabric has proven additional advantages of ease in handling during processing, apart from imparting strength in two directions. Performance properties of BD composites mainly depend on the type of matrix, fabric (its amount, type, orientation with respect to loading direction, weave etc.), fiber–matrix interface and processing technology. Their erosive wear performance depends on type of erodent, its shape, size, flux rate and operating parameters such as angle of impingement of erodent, its velocity, temperature of composite, humidity etc. [6]. Angle of impingement is the most widely studied parameter in the literature [7] while temperature effect appears to be sparingly studied parameter [8]. Among the available literature on tribology of BD polymer composites, a little is reported on the erosive wear behavior [9,10]. Though a lot is reported on tribology of PEEK and its short fiber composites and unidirectional carbon fibers [11], not adequate data are available on erosive wear of carbon fabric PEEK (CF-PEEK) composites [12–14]. Hence in this work, erosive wear studies on CF-PEEK composites with variation in angle of impingement using fused alumina as erodent were performed under various temperatures. Since CF is known for its inertness towards a matrix, these were treated with a recent technique with cold remote nitrogen–oxygen plasma (CRNOP) prior to development of composites. A lot is reported on the classical and modified plasma treatments for influencing performance properties of composites [15,16] but not much on its exploitation for enhancing tribological performance of composites. Few papers are available in this respect for adhesive

---

* Corresponding author. Tel.: +91 11 26591280; fax: +91 11 26596222.
E-mail addresses: jbijwe@hotmail.com, jbijwe@gmail.com (J. Bijwe).

0043-1648/$ – see front matter © 2011 Elsevier B.V. All rights reserved.
doi:10.1016/j.wear.2011.02.002

Table 1
Properties of twill weave carbon fabric[a].

Carbon fabric properties	
Density (g/cm^3)	1.85
Area (g/cm^2)	198
Tex	22
Denier	198
Crimp (%)	0.70
Count	26
Warp/inch	16
Weft/inch	16
Thickness (cm)	0.34
Bending length (cm)	5.9
Tensile strength (MPa)	0.147
Elongation (%)	1.85

[a] Supplier's data.

or fretting wear performance [17–19] but not on erosive wear behavior. For CNROP treatment on CF, papers are not available in tribology, though on mechanical properties [20]. The composites from our earlier work [20] were selected for erosion wear studies in this paper.

Carbon materials, carbon fibers and other sp^2 bonded amorphous carbons are strong Raman scatterers. The technique enables to distinguish between various structural organizations in these materials [21,22]. The exploitation of this technique has not been extensively done in tribology for various analyses. A very few papers are available on reporting adhesive wear studies of composites with carbon nano-tubes in carbon–carbon (C–C) composites in which the characterization with Raman spectra indicated that the graphitic degree of worn surface increased due to the strain produced by shear deformation on the friction surface [23]. In another paper in which CNT doped C–C composites were tested on Ball-on-disc configuration, however, spectra recorded for worn and unworn surfaces did not reveal any changes leading to the speculation that the CNT doped on the surface might be swept away due to severe wear or changed to amorphous carbon [24]. Thus the potential of MRS in correlating wear with stresses induced in fibers and subsequently operating parameters or wear mechanisms is not yet established convincingly.

Though MRS is expected to quantify amount of stresses in the carbon fibers, no efforts are yet reported for exploiting this in tribological studies. Hence in this paper, correlations between stresses due to treatment and erosion with changes in graphitic disorientation are reported and well correlated. Finally efforts are made to study correlation between high temperature erosion rates and high temperature mechanical properties of composites.

2. Experimental

2.1. Procurement of materials

PEEK powder (150 XF) was supplied by VICTREX, USA. The 3K (2 × 2) twill weave multifilament continuous tow carbon fabric was procured from Fibre Glast Developments Corporation USA. Table 1 shows the supplier's data for carbon fabric used as reinforcement.

2.2. Surface treatment of CF

The facility of cold remote nitrogen oxygen plasma (CRNOP) available at University of Science and Technology Lille, France; was used for surface treatment of chemically inert CF to enhance its reactivity towards matrix and details are discussed elsewhere [25].

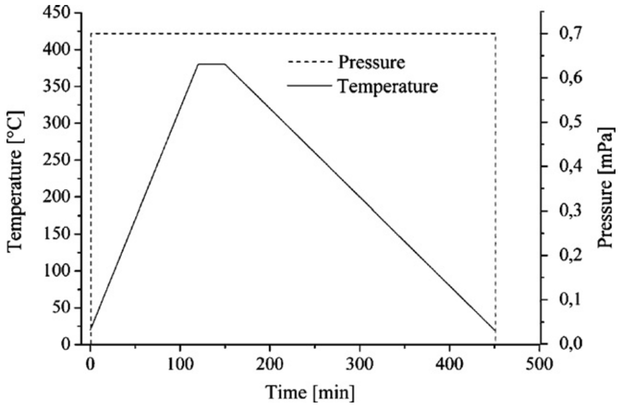

Fig. 1. Pressure and temperature profile for composites manufacturing.

2.3. Fabrication of composites

Since PEEK is soluble only in some very corrosive solvents, conventional method of solution impregnation is not applicable for developing CF-PEEK composites. Hence in the present work, powder sprinkling followed by static compression molding method was adopted. Composites were developed at IVW (Institut für Verbundwerkstoffe) GmbH, Germany; on static press in which the heating and cooling cycles are employed in the same tool. Prior to the manufacturing process, the piece of fabric (400 mm^2) was placed on the bed of known weight of PEEK powder on the platen followed by placement of an alternate sequence of fabric and powder. The amount of powder was optimised in such a way that the fibers in the matrix could be ≈70% by wt. in the final composite. Fourteen such layers were arranged between the heating/cooling plates and moulded using the temperature and pressure profiles as shown in Fig. 1. The maximum temperature of 380 °C was allowed to reach in 120 min, was held for 30 min and then cooled down to 20 °C at a cooling rate of 1.2 K/min. The operating pressure was maintained at 0.7 MPa during the entire process.

In all, two composites designated as $P_{K\ CFU}$ and $P_{K\ CFT}$ (P_k for PEEK; subscripts CF for carbon fabric, U for untreated and T for CRNO (0.5%) P treated fabrics) were developed and studied.

2.4. Characterization of the composites

2.4.1. Physical characterization

The physical and mechanical characterization of two composites was done a per ASTM standards (Table 2). In physical characterization, density of composites was determined as per ASTM D792 method. Since PEEK is insoluble in non corrosive solvents, ignition loss method (ASTM 2584-02) was used to calculate fiber weight fraction in PEEK composites which was 68 wt.% (55 vol.%).

Table 2
Physical and mechanical properties of CF-PEEK composites.

Properties/materials	P_K[*]	$P_{K\ CFU}$	$P_{K\ CFT}$
Fiber weight (wt.%) ASTM 2584-02	–	68.2	67.9
Void fraction (vol.%) ASTM 2734	–	0.56	0.58
Density (g/cm^3) ASTM D792	1.3	1.43	1.44
Tensile strength (MPa) ASTM D638	100	576	627
Tensile modulus (GPa) ASTM D638	3.7	57	60
Strain at break (%) ASTM D638	15	1.0	1.1
Toughness (MPa) ASTM D 638	–	3.0	3.4
Flexural strength (MPa) ASTM D 790	170	622	726
Flexural modulus (GPa) ASTM D790	4.1	51	78
ILSS (MPa) ASTM D2344	–	43	46

[*] Supplier's data based upon ISO methods (PK for PEEK; subscripts-CF for carbon fabric, U for untreated and T for treated).