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International Conference On DESIGN AND MANUFACTURING, IConDM 2013 Dry sliding wear behaviour of Ta/NbC filled glass-epoxy composites at elevated temperatures

N.Mohan^a*, C.R.Mahesha^a, Shivarudraiah^b, N. Rajesh Mathivanan^c, B.Shivamurthy^d

^aDepartment of Industrial Engineering and Management, Dr.Ambedkar Institute of Technology, Bangalore-560 056, India.

^bDepartment of Mechanical Engineering, University Visweswaraya college of Engineering, Bangalore-560 00, India.

^cDepartment of Mechanical Engineering, P.E.S. Institute of Technology, Bangalore-560 0056, India.

^dDepartment of Mechanical and Manufacturing Engineering, Manipal University, Manipal-576 104 India.

Abstract

In this work an attempt was made to evaluate wear loss, specific wear rate and coefficient of friction of Glass-Epoxy (G-E) composites with and without Tantalum Niobium Carbide (Ta/NbC) filler. A vacuum assisted resin transfer moulding (VARTM) technique was employed to fabricate the composite specimens. The fabricated wear specimens were tested by using pin-on-disk test rig at various temperatures viz., 30, 60, 90 and 120° C at normal applied loads of 10 N and 20 N. Sliding velocity of the disc of 1.5 m/s was maintained and test was continued for each sample up to a sliding distance of 5000 m. The wear loss in both the composites increases with increase in temperature and applied normal load. However, Ta/NbC particulate filler incorporated G-E composite exhibits lower wear rate and higher coefficient of friction as compared to unfilled G-E composite. The features of worn surfaces of the specimens were examined under scanning electron microscopy (SEM) and findings are analysed.

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Keywords: Glass fabrics; Epoxy; Wear; Tantalum Niobium Carbide.

Nomencl	ature		
ν	velocity (m/s)	Δ_V	Volume loss in m ³
μ	Coefficient of friction	L	Load in Newton
R_a	Surface roughness	d	Sliding distance in meters (m)
K_S	Specific wear rate		

^{*} Corresponding author. Tel.:+91- 080-23211505-Ext. 246 or 919060818100; Fax: +91- 080- 23217789. E-mail address: drmohannagaraj@gmail.com

1. Introduction

Polymer matrix composites (PMCs) have been increasingly used for numerous engineering purposes such as seals, gears, rollers, cams, wheel, clutches and bearings due to better tribological properties, wide varieties of availability and design flexibility. PMCs are produced without fillers and reinforcement using an epoxy resin as matrix, exhibit relatively high wear rates when dry-sliding against steel counter faces. This is basically due to the cross-linked molecular structure, which inhibits the formation of an efficient transfer film and results in a relatively high degree of brittleness. However, epoxy resins possess other favorable properties such as strong adhesion to many materials, good mechanical and electrical properties, relatively high chemical and thermal resistance. Also epoxy in moulded or cast form has excellent dimensional stability and low shrinkage [1-2]. Whereas Glass fabric reinforcements in PMCs generally improve the creep resistance,

stiffness and compressive strength and also result in enhanced wear resistance. Automotive applications is the new generation of control arm mountings or ball joints in the car chassis technology, in which higher loads and temperature are acting. In this case, PMCs will be operated at relatively high environmental temperature up to 120° C [3-5]. PMCs are promising in tribological applications due to the possibility of tailoring their properties with special fillers such as MoS₂, CuO, CuS, Al₂O₃, Gr, WC, Ta, Nb, and SiC etc. [6-7]. One of the limitations in the use of reinforced PMCs is their inferior thermo-resistant properties relative to metals and ceramics. During sliding, the mechanical energy is mainly dissipated as heat. This may lead to decomposition and deterioration of composite layups, which in turn often decreases the friction and wear performance. This limits the life time application of components made from PMCs. This is especially a problem in applications involving high contact pressures, high sliding velocities and high specific rate of energy dissipation. Hence, PMCs which are used in heavy duty applications at high temperature environment systems the heat decomposition and frictional heating are reduced by applying solid lubricating fillers [8]. Additionally, if the PMCs are subjected to harsh environmental conditions such as chemicals, or moisture, the characterization of the material's performance is more essential before application because of the viscoelastic nature of polymeric composites. Above glass transition temperature (Tg) fiber reinforced PMCs properties degrade significantly [9-10]. Hence it is necessary that the application temperature for a polymeric composite is below the Tg in order to assure that the mechanical and tribological properties of the material is satisfactory and epoxy resin is limited to a maximum service temperature about 120° C [11]. Crivelli Visconti et al. and Mohan et al. [12-13] in their study concluded that enhanced abrasion resistance can be obtained with the incorporation of hard powders (SiO₂, WC and Ta/NbC) filler into G-E composites. Most of the published studies on sliding wear behaviour of glass-epoxy composites under room temperature conditions. In the present work wear studies have been carried out for Ta/NbC filled G-E composite and unfilled G-E composite at different temperatures viz., 30, 60, 90 and 1200 C and reported.

1. Experimental

2.1. Materials and Fabrication of composite specimens

A bidirectional E-Glass woven fabric 360 g/m² was procured from M/s. Reva Composites, Bangalore, India. The glass fabric of dimension 18 μ m diameter was used as reinforcement. Bifunctional epoxy resin (LY 5052) and room temperature curing cyclo aliphatic amine (HY 5052) (system) were obtained from M/s. HAM, India. The resin is a clear liquid with viscosity at 25 $^{\circ}$ C, 1000-1500 mPa and specific gravity 1.17g/cc. The hardener is a liquid and its viscosity is 40-60 mPa and specific gravity 0.94 g/cc. The commercially available tantalum niobium carbide in the ratio of 60:40 (Ta/NbC) (Density of Ta/NbC=10.8 g/cc, Size = 50-55 μ m) was used as filler. The powders were procured from M/s. Kennametal, Bangalore, India.

2.2. Fabrication of composite specimens

The composite fabrication consist of three steps: (a) mixing of the epoxy resin and filler using a mechanical stirrer, (b) mixing of the curing agent with the filled epoxy resin, and (c) fabrication of composites. In the first step, a known quantity of filler was mixed with epoxy resin using a high speed mechanical stirrer to ensure the proper dispersion of filler in the epoxy resin. In the second step, the hardener was mixed into the filled epoxy resin using a mechanical stirrer. The ratio of epoxy resin to hardener was 100:38 on a weight basis. In the last step, the epoxy resin was manually smeared onto the glass fabric and the resultant composites were fabricated using the VARTM process as described elsewhere [14] (Fig.2). The composites were cured at room temperature under a pressure of 14 psi for 24 h and it is post cured up to three hours at 100° C The glass fiber: matrix (epoxy): filler ratio was 60:36:4. The unfilled glass epoxy composites were designated as G-E and Ta/NbC filled G-E composites as Ta/NbC-G-E. The laminate of dimensions $300 \text{ mm x } 300 \text{ mm x } 2.6 \pm 0.2 \text{ mm}$ was fabricated and the specimens for the required dimensions were cut using a diamond tipped cutter. Density of the composites specimens was determined using a high precision digital electronic weighing balance of 0.1 mg accuracy by using Archimedes principle and the formulations measured density of the composites are listed in Table 1.

Table 1 Formulations of composite specimen with measured physical properties.

Specimen code	Glass fibre (wt.%)	Matrix (wt.%)	Filler (wt.%)	Density (g/cc)	Hardness Shore-D
Glass fabric-epoxy composite (G-E)	60	40	-	1.78	85
Ta/NbC filled G-E composite (Ta/NbC –G-E)	60	34	4	1.80	88

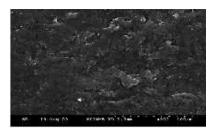


Fig.1 SEM image of Glass- epoxy composites after fabrication

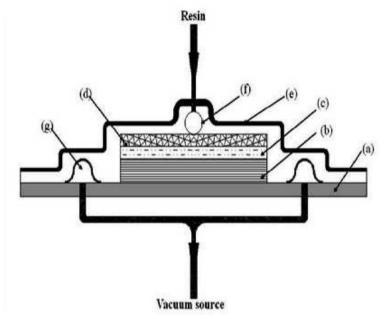


Fig. 2 Schematic of VARTM setup: (a) Granite molding tool, (b) Dry glass fabrics, (c) peel ply and breather material (d) distribution medium, (e) vacuum bag, (f) resin inlet, and (g) vacuum outlet

2.3 Techniques

A high temperature pin-on-disc (HT-POD) setup was used for the sliding wear tests as per ASTM-G99 (Fig. 3). The fabricated sample (5 mm x 5 mm x 2.8 mm) was fixed to a square slot (5 x 5 x 1.5 ± 0.2 mm) in the pin of dimensions 10mm dia and 25mm length and fitted in the pin holder mounted in the HT-POD lever-arm such a way that which comes in contact with a hardened alloy steel disc of the machine. The hardness value of the EN-32 alloy steel disc 55 HRC and surface roughness (Ra) of $0.65\mu m$. the test was carried out at normal load of 10 N, sliding velocity of 1.5 m/s and at varying temperatures viz., 30, 60, 90 and 120° C. For every sample the surface was cleaned with a soft paper soaked in acetone and compressed air before and after testing. The specimen weight is recorded using digital electronic balance 0.1 mg accuracy. The difference between initial and final weight of the specimen was a measure of slide wear loss. A minimum of three trials were conducted to ensure repeatability of test data. The frictional force is measured by attaching a force transducer on the machine. The friction coefficient was recorded continuously and gets displayed on a computer interfaced with the high temperature pin-on-disc (HT-POD) machine. Experimentation was repeated for anther set of samples at 20 N applied normal load. The tests conditions adopted in the present study are listed in Table 2.The wear behaviour was measured by the loss in weight, which was then converted into wear volume using the measured density data. The specific wear rate (Ks) was calculated from the equation;

$$K_S = \frac{\Delta V}{L \times d} m^3 / Nm$$

Where, Δ_V is the volume loss in m³, L is the load in Newton, and d is the abrading distance in meters.

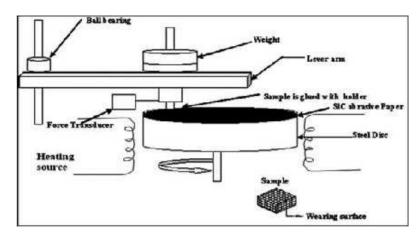


Fig. 3 A Schematic representation of high temperature pin-on-disc machine setup



Fig. 4 Photograph of dry sliding pin on disc wear test rig (i) Heating Coils and Pin and disc, (ii) pin holder with screw, (iii) Specimen with holder and (iv) Fixed steel disc

Table 2 Details of the sliding wear test conditions.

Test conditions	Parameters
Dimension of the specimen (mm³)	5 x 5 x 2.8
Applied load (N)	10 and 20
Angular speed (rpm)	716.5
Sliding diameter (mm)	40
Sliding Distance (m)	5000
Sliding Velocity (m/s)	1.5
Temperature (⁰ C)	30, 60, 90 and 120
Steel disc hardness and roughness (HRC and Ra)	55, 0.65

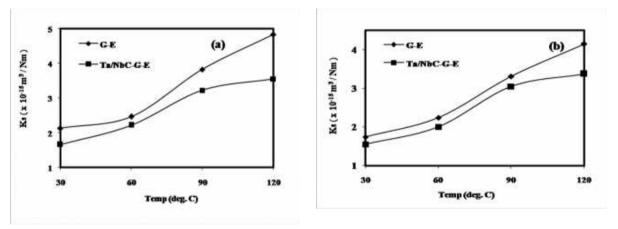
3. Results and Discussion

3.1 Wear volume loss and specific wear rate

The wear loss of composites increases with increase in applied load and temperature. Due to thermal softening, initially both type of composites the epoxy matrix was detached from the composite surface and after certain sliding distance shear deformed polymer matrix containing broken pulverized matrix powder which spreads on the counter surface. This remains there some time as a transfer layer on the steel counter face. In case of unfilled G-E composite the broken pulverized glass particles can act as a third body abrasive leading to enhanced roughening of the counter surface. Hence, the soften epoxy matrix dig into the surface of the composite specimen and enhances the wear loss. While in the case of Ta/NbC filled G-E composite, pulverized debris from the surface of the composite consists of Ta/NbC form a layer of transferfilm which acts as effective barrier to prevent the large scale fragmentation of epoxy. This phenomenon is more effective at lower temperature, but at higher temperature due to softening effect of polymer reduces the debris formation and will not create film and failure of surface more on plastic deformation and deep scratches. Hence, the wear loss of unfilled G-E composite is much higher than those of Ta/NbC filled G-E composite at elevated temperatures.

The lowest wear loss of 1.5 x 10⁻⁴ g was observed in Ta/NbC filled G-E composite at 30⁰ C temperature and highest wear loss of 4.3 x 10⁻⁴ g was observed in unfilled G-E composite at 120⁰ C temperatures and at applied load of 10 N. Furthermore, higher wear loss of 7.4 x 10⁻⁴ g was noticed in unfilled G-E composite at 120⁰ C and at applied load of 20 N. As compared to Ta/NbC filled composite, unfilled G-E composite shows more wear loss at all conditions. In case of Ta/NbC filled Glass-epoxy composite, wear loss increased with the increase in applied load and temperature. This is due to energy barrier created at the specimen surface is greater due to the reinforcement of Ta/NbC particles into the matrix. Hence at even higher loads and higher temperature, energy generated by third body particles at counterface is not sufficient as a result particles cannot get penetrate deeper into the matrix material and only a microfracture of matrix material. The wear data of the composites reveal that the wear loss strongly depends on the applied load and temperature.

The plot of specific wear rate (Ks) as a function of temperature is as shown in Fig. 5 a and b. The specific wear rate increases with increase in temperature and decreases with increase in applied load. The lowest Ks $(1.5 \times 10^{-15} \text{ m}^3 / \text{Nm})$ for Ta/NbC filled Glass-epoxy composite and highest Ks $(4.8 \times 10^{-15} \text{ m}^3 / \text{Nm})$ for unfilled Glass-epoxy composite were observed. As compared to unfilled G-E composite the Ta/NbC filled composite shows low specific wear rate at all condition and which acts as a anti wear additive. The results of the present study at normal temperature sliding condition is matching with several authors who have discussed the role of hard powders as effective wear resistant filler in tribological studies [12-14].



 $Fig.\ 5\ Specific\ wear\ rate\ versus\ temperature\ of\ G-E\ and\ Ta/NbC-G-E\ composites\ at\ (a)\ 10\ N\ and\ \ (b)\ 20\ N\ load.$

3.2. Coefficient of Friction

The main mechanism of frictional energy dissipation will be shear of a few nanometers thin zone of the composite at the interface. If the sheared layer of composite film may be deposited on the smooth counter surface which acts as a transfer layer and subsequently the frictional energy dissipation mainly takes place either in the transfer film or in a very thin layer [15-18]. The detached thin layer from the matrix was directly contact with the counter surface. The measured coefficient of friction (μ) of the composites is given in Table 3. For unfilled G-E composite μ decreases as increase in temperature and load. The Ta/NbC filled G-E composite shows higher value of μ and it increases with increase in

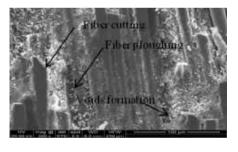
temperature and decreases with increase in applied load. This indicates that μ depends on the temperature and applied load. As temperature increases, more heat generates and consequently more fiber damage occurs. The hard particles in epoxy matrix acts as a third body and uniformly distributed on the surface of the specimen and they can easily roll in between the counterface. Hence, Ta/NbC filled composite shows lower wear rate and high coefficient of friction. In G-E composites due to increase in both surrounding and interface temperature, rupture of adhesive bonds occurs in between the counterface and specimen and fibre glass particles dig into the specimen surface giving high wear rate and lower coefficient of friction.

Table 3 Coefficient of friction	(μ) in G-E and Ta/NbC -G-E com	nosite specimens under all evn	erimental conditions
Table 3 Coefficient of friction	(µ) III G-L and Ta/Tibe -G-L com	posite specimens under an exp	CHIIICHIAN CONGINONS

Load (N)	Coeffici	ent of friction (µ)						
	Те	mperature 30°C		60°C	90°C		120°C	
	G-E	Ta/NbC-G-E	G-E	Ta/NbC-G-E	G-E	Ta/NbC-GE	G-E	Ta/NbC-G-E
10	0.39	0.65	0.34	0.69	0.29	0.72	0.21	0.79
20	0.36	0.59	0.32	0.53	0.26	0.50	0.19	0.45

3.3 Worn Surface Morphology

In glass fabric reinforced-epoxy composites the process of material removal in dry sliding condition is dominated by four wear mechanisms, viz., matrix wear, fiber sliding wear, fiber fracture and interfacial debonding. The matrix wear occurs due to plastic deformation and fiber sliding wear occurs due to fiber rubbing, fiber rupture, fiber cracking, and fiber pulverizing [19-22]. The SEM photographs of worn surfaces of unfilled and Ta/NbC filled composite samples at 10 N and 20 N load, constant sliding velocity of 1.5 m/s and for an sliding distance of 5000 m at 120° C are shown in Figure 6 and 7. The Worn surface of unfilled G-E composite (Fig.6a and 7a) shows both fiber and matrix breakage/removal and fiber breakage and voids formation due to thermal softening of the matrix. In Ta/NbC filled G-E composite (Fig.6b and 7b) hard Ta/NbC in matrix well bonds between fiber and matrix and these hard particles are well protects the fiber from damage and worn surface was relatively smooth and less damage to matrix was observed and as compared to lower load at higher load shows severe damage of both the composites.



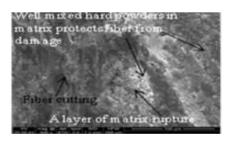
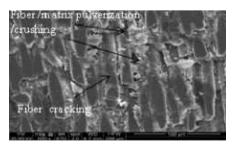


Fig. 6 SEM image of (a) G-E and (b) Ta/NbC-G-E composites at 10 N load and at 120°C





(a)

(a)

Fig.7 SEM image of (a) G-E and (b) Ta/NbC-G-E composites at 20 N load and at 120°C

(b)

4. Conclusion

- Tribological behaviour of unfilled and Ta/NbC filled G-E composites strongly depends on the experimental test parameters such as temperature and applied load. The enhancement on the wear resistance of Ta/NbC filled G-E composite is associated with less fibre / matrix loss.
- The specific wear loss increases with increase in the temperature.
- The change in the coefficient of friction with temperature does not follow a fixed pattern for both the composites.
- The SEM images of the specimen reveals fibre fracture, fibre breakage, matrix cracking, matrix wear, void formation and fibre exposure.

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