Influence of fillers on abrasive wear of short glass fibre reinforced polyamide composites

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Various composites of polyamide 6 filled with short glass fibre, polytetrafluoroethylene and metal powders viz. copper and bronze were formulated in the laboratory and characterised for their various mechanical properties such as tensile strength, tensile elongation, flexural strength, hardness and impact strength. Compositional analysis was done with gravimetry, solvent extraction and differential scanning calorimetry (DSC) techniques followed by tribo-performance evaluation in abrasive wear mode by abrading a sample against silicon carbide (SiC) abrasive paper in a single pass condition under various loads. It was observed that the fibre reinforcement deteriorated the abrasive wear resistance of virgin polymer. Combination of fibre and particulate filler was more detrimental in this respect. Efforts were made to correlate the wear performance with the appropriate mechanical properties. Under selected loading condition, wear as a function of product of hardness, elongation to break (e) and ultimate tensile strength (S) showed better correlation than Ranter-Lancaster plot. Scanning electron microscopy (SEM) was used to analyse the worn surfaces of the samples. © 2001 Kluwer Academic Publishers

1. Introduction

Abrasive wear is the most atrocious among all the forms of wear and contributes almost 50% to the total wear. Polymers and the fibre reinforced plastics (FRPs) are most widely used in highly abrasive systems such as conveyor aids, vanes, gears for pumps handling industrial fluids, sewage and abrasive contaminated water; bushes, seals and chute liners in agricultural, mining and earth moving equipments; roll neck bearings in steel mills subjected to heat, shock loading, water; and guides in bottle handling plants etc $[1-3]$. Polyamides and their composites are used in a variety of triboapplications because of their very good performance in adhesive and abrasive wear situations especially gears, bearings and tyres etc. Advances in technology are placing more and more demands on materials in terms of better performance in stringent operating conditions. Hence, research efforts are always focussed to tailor newer materials in terms of composites. However, tribo-properties are not intrinsic in nature and definetly depend on various environmental and experimental parameters [4]. The influence of fillers and fibres on tribobehaviour of composites cannot be predicted *a priori* and has to be tested in the laboratory.

It is always interesting to investigate structureproperty correlation and effect of fillers since it forms a platform for tailoring the composites with anticipation of desired properties. It has always been the most sought

area in tribology of composites also [5–23]. In this paper, a few composites based on polyamide 6 (PA 6) were formulated with short glass fibre (GF) and different fillers with a view to investigate their behaviour in abrasive wear situations. Though composites are generally used for adhesive wear applications, they are also exposed to abrasive wear situations depending on the environment of use e.g. abrasives entrapped from the dusty environment. Hence, it becomes more essential to study their abrasive were behaviour also. Their wear performance and the efforts to correlate it with various mechanical properties are reported in this paper.

2. Experimental

2.1. Materials selected

Details of the composites selected for this present work are shown in Table I. Polyamide 6 and 30% chopped GF composites were procured from GE Plastics (India) Ltd. Polytetrafluoroethylene (PTFE), bronze and copper powders were procured from local market. These components were mixed homogeneously in a tumble mixer before extrusion followed by injection moulding. All the composites were analysed for their compositions by gravimetry, solvent extraction and differential scanning calorimeter (DSC). Various mechanical properties were also measured and the data revealed are presented in Table II.

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TABLE I Details of the PA 6 based composites studied in this work

S , no.	Material	Designation
	PA ₆	А
2	PA $6 + 20\%$ GF	В
\mathcal{R}	PA $6 + 30\%$ GF	\subset
	PA $6 + 15\%$ GF $+ 3\%$ PTFE	
	$+6\%$ Copper powder	
	PA $6 + 15\%$ GF $+ 3\%$ PTFE	
	$+6\%$ bronze powder	E.

Figure 1 Schematic of abrasive wear testing machine.

2.2. Abrasive wear studies

Abrasive wear studies were carried out on a pinon-disc machine, the schematic as shown in Fig. 1 and discussed elsewhere [24]. The polymer pin (10 mm \times 10 mm \times 4 mm) was abraded against the waterproof 1200 grade (grit size \cong 6 μ m) silicon carbide (SiC) paper for uniform contact and then cleaned with acetone, dried and weighed followed by abrading it in a single pass condition against the 80 grade (grit size \approx 175 μ m) SiC paper fixed on a disc rotating with a linear speed of 50 mm/s. After each traversal, a new paper was fitted until the total abrading distance was 3.26 m and range of applied load was 4–10 N. The pin was weighed again after cleaning with a brush to remove wear debris to calculate weight loss. The specific wear rate (K_0) was calculated from the following equation.

$$
K_0 = \frac{V}{Ld} \quad \text{m}^3/\text{Nm} \tag{1}
$$

where *V* is wear volume in m^3 ; *d* is sliding distance in metres, *L* is the load in newtons. Each experiment was repeated for three times. The average of two values, which were nearly same was taken. If all the three values differed significantly, the experiment was repeated for the fourth time.

2.3. Studies on SEM and DSC

Worn surfaces of the pins were observed with scanning electron microscope (SEM) after sputtering with silver coating. The melting point was determined using differential scanning calorimetry (DSC) (Perkin Elmer DSC 7) in inert N_2 atmosphere. The experiments were carried out in both heating and cooling modes with the rate of 10◦C/min.

3. Results and discussion

3.1. Abrasive wear studies

Details on the compositional analysis and mechanical studies are shown in Tables I and II respectively. Specific wear rate as a function of load for all the composites is plotted in Fig. 2. Wear volume as a function of various mechanical properties is plotted in Fig. 3. Micrographs of worn surfaces are shown in Fig. 4. Following are the salient observations revealed from the data on thermal and mechanical properties.

- Tensile and flexural strengths of PA 6 increased with increase in GF reinforcement i.e. for composites A to C which is according to general observation in FRPs. In case of impact property, however, 20% GF inclusion exhibited maximum strength. Addition of fillers such as PTFE and metal powders, however, deteriorated all these properties as seen in the cases of composites D and E.
- Tensile elongation to break drastically reduced with increasing amount of GF which is also in

Figure 2 Specific wear rate as a function of load: (a) for PA 6, (b) for PA $6 + 20\%$ GF, (c) for PA $6 + 30\%$ GF, (d) for PA $6 + 15\%$ GF + 3% PTFE + 6% Copper powder, (e) for PA $6 + 15%$ GF + 3% PTFE + 6% Bronze powder.

TABLE II Details of mechanical properties of the selected composites

Properties	Method	А	В		D	E
Hardness	Shore D	83	85	85	81	81
Impact Strength $(kg \, m/m)$	Izod Test	4.92	19.68	13.28	9.84	9.84
Flexural Strength (MPa)	ASTM D 790	42.10	66.98	106.83	79.70	72.80
Tensile Strength (MPa)	ASTM D ₆₃₈	38.58	52.03	84.75	49.09	46.59
Tensile elongation to break (%)	$---do--$	139.3	12.90	4.66	7.27	8.80
Se		5374	671	395	357	410

Figure 3 Wear volume as a function of various mechanical properties (a) as - - - - - - - - $(HSe)^{-1}$ and ——— (*Se*) $^{-1}$, (b) as $(e)^{-1}$.

tune with the general observation in the case of FRPs.

• The hardness value (*H*) increased with inclusion of GF reinforcement. Inclusion of PTFE in composites D and E, however, lowered it significantly.

As seen in the Fig. 2, specific wear rates for all the materials decreased with increase in load which is according to Lhymn's mathematical model [5] applicable when thermal effects are negligible due to low sliding speed. It was also seen that wear performance of the parent polymer deteriorated due to filler/fibre inclusion and was in the order $A > B > C > D > E$. Influence of fibres and/or fillers on the abrasive wear resistance of neat polymer is more complex and unpredictable and mixed trends are reported (Tables III and IV). Table III is for PA and its composites while Table IV is for materials other than PA. Lancaster [6] studied thirteen polymers reinforced with 30% short carbon fibre (CF) and reported that reinforcement enhanced wear performance of seven composites and that of six composites deteriorated. Earlier work by the authors [7–9] on the effect of various fillers such as GF, CF , PTFE, $MoS₂$, bronze powder etc. on abrasive wear behaviour of several thermoplastic materials viz. PA 6, PA 66, polyetherimide (PEI), polyimide (PI) and PTFE revealed an interesting trend. They found that all the particulate fillers and short fibres deteriorated the abrasive wear resistance of the virgin polymers, though the extent of influence depended on the polymers and the type of fillers. In the case of PA 66 reinforced with increasing amount of short CF, the wear behaviour continuously decreased with increasing percentage of fibres [7]. In the case of PI and PEI reinforced with short GF and solid lubricants, a similar trend was observed [10]. Sole *et al*. [11] reported that the mineral fillers such as talc, $CaCO₃$, BaSO₄ and fly ash deteriorated the abrasive wear behaviour of polypropylene (PP). The deterioration was due to the initiation and propagation of the cracks in the composite which promoted elastic and plastic mismatch between the matrix and the fillers. Mens *et al*. [12] observed that the incorporation of PTFE as filler in PA 66, Polyoxymethylene (POM) and PETP deteriorated the abrasive wear behaviour of the virgin polymers. Simm *et al*. [13] found that incorporation of bronze powder deteriorated the abrasive wear behaviour of epoxy. Briscoe *et al*. [14], however, reported the mixed trend for the abrasive wear of Polyetheretherketone (PEEK) filled PTFE and PTFE filled PEEK. Incorporation of PEEK in PTFE reduced the wear rate of PTFE while the wear rate increased in the later case. Few researchers, however, have also reported on the optimum percentage of fillers for maximum wear resistance. Lu *et al*. [15] for instance, investigated the abrasion of earth moving equipment parts made up of Al_2O_3 + PTFE + PPS and reported that the wear performance continuously increased up to 20% filler concentration Beyond that it worsened drastically. Similarly Yang *et al*. [16] reported about the optimum percentage of glass spheres in polydimethysiloxane being 30% for best wear performance. Bijwe *et al*. [17] also have reported 30% of short GF being the optimum percentage for highest wear resistance of PEI. Higher or lower concentration than this deteriorated the wear behaviour. In case of long glass, carbon and aramid fibre composites, also improvement in wear behaviour of PEEK and epoxy is reported [1, 18]. Liu *et al*. [19] and Ratner *et al*. [20] have also reported improvement in wear behaviour of UHMWPE and PP due to addition of quartz powder of two different sizes and $TiO₂$ respectively.

While discussing the reasons behind the filler effect (30% short CF in thirteen polymers) Lancaster [5] observed that the product of *S* and *e* (where *S* is ultimate tensile strength, *e* is ultimate elongation) is a very important factor which controls the abrasive wear behaviour of the composites. Reinforcement with the fibres and/or fillers always increases the tensile strength of the virgin polymer. The ultimate tensile elongation, however, generally reduces with increasing amount of fillers and fibres. Generally the product (*Se*) for the virgin polymer is higher than that for the reinforced composites. Hence, reinforcement usually leads to deterioration in the abrasive wear resistance. In earlier work by the authors [7–9] also, similar correlation between abrasive wear performance and the *Se* factor in the case of all polymer composites except one [17] was observed.

In the present studies, Fig. 3 indicates the relationship between the wear volume and the $(Se)^{-1}$ and $(HSe)^{-1}$

HNTN- 11746 20.0 KV $HD - 22$ PHOTO- 11748 EHT- 20.0 KV $HD - 23$ (e) (f)

Figure 4 SEM micrographs of worn surfaces of selected materials (load 10 N, speed 5 cm/s and 80 grade SiC Paper) (a) showing microploughing and microcutting of PA matrix, (b) showing broken fibre pieces in composite B, (c) and (d) showing severe damage due to microcracking, microcutting and pulverisation of the fibres, (e) showing fibre breakage and fibre pull out of composite D, (f) showing excessive wear of matrix in composite E.

(Fig. 3a) and *e*−¹ (Fig. 3b). In the case of elongation to break (*e*−1), no linearity was observed while in other cases (Fig. 3a) though reasonably good linearity was found in the case of the Ratner-Lancaster plot [wear rate vs (*Se*)⁻¹], wear rate vs (*HSe*)⁻¹ showed still better correlation indicating all the three factors viz. *e*, *S* and *H* (hardness) played prominent roles in controlling wear behaviour of the selected composites rather than the individual one. Composite E showed little excessive wear which could be probably because of less compatibility of bronze powder with the polymer matrix.

In the literature, no fixed correlations with material properties are reported in such situation. Various researchers have found variety of factors such as *e*, *S*,

S . no.	Resin	Fibre/filler	$wt\%$	Wear rate due to fillers	Correlation of abrasive wear with	Ref.
1	PA 6, PA 66, PVC (Polyvinylchloride), PTFE, PP, Epoxy, PMMA (Polymethylmethacrylate), Polyester, PC (Polycarbonate), Phenolic, PE (Polyethylene), Acetal copolymer,	CF	30	Increased for six polymers and decreased for seven polymers	$(Se)^{-1}$	[6]
\overline{c}	PA 66	CF PTFE	10, 20, 30 and 40 15	Increased	$(Se)^{-1}$	$[7]$
3	PA 6, PEI, PI, and PTFE	GF PTFE Graphite $MoS2$, Carbon Bronze powder	16, 20 and 25 10 and 15 15, 20 and 40 5, 15 25 55	Increased		[8]
4	PA 66, POM and PETP	PTFE	15	Increased		$[12]$
5	PA 6, PMMA, PE, POM, PA 66, PP, PTFE, PC, PTFCE (Polytrifluorochloroethylene), PPO Poly(phenylene oxide), and PVC				cohesive energies.	$[21]$
6	PA 6, PTFE, PP, POM, HDPE (High density Polyethylene), PVC, and PMMA				Ploughing component of friction, hardness, tensile strength and elongation to failure of polymers.	$[22]$

TABLE IV Details of the reported literature on abrasive wear performance of polymer composites other than polyamides

H, cohesive energy, ploughing component of friction, fracture energy of composites and probability factor for microcracking as abrasive wear controlling factors and are correlated in Tables III and IV. Giltrow [21] for example, correlated the cohesive energies of the various thermoplastics with their abrasive wear behaviour. He found that the rate of abrasive wear of the thermoplastic polymers was inversely proportional to the square root of their cohesive energy. Vaziri *et al*. [22] developed the empirical relationship which correlated the abrasive wear rate of various polymers with their hardness, tensile strength, elongation to break and ploughing component of friction. Friedrich *et al*. [23] on the other hand, correlated the abrasive wear behaviour of the poly(ethylene terephthalate) (PET) and their composites with their hardness, macrofracture energy and the probability factor for microcracking.

In the present studies, however, the product (*Se*) was maximum for neat polymer (Table II) which showed the highest wear resistance. This product decreased due to inclusion of fillers which resulted in deterioration of abrasive wear performance.

3.2. Scanning electron microscopic studies

Micrographs 4a–f are for surfaces of polymer pins worn under a load of 10 N. Micrograph 4a for neat polymer indicated plastic deformation of matrix

when microploughed and microcut under load. Micrograph 4b ($PA + 20\%$ GF) and micrographs 4c and d $(PA + 30\% \text{ GF})$ indicated different extents in severity of wear. Few fibre pieces after microcutting lying on the surface (4b) can be seen. A magnified view indicated that such broken pieces can be easily peeled off in further abrasion contributing to higher wear. In the case of the worn surface of composite C (micrograph 4c), the number of fibres appearing on the surface is greater and damage to fibres due to microcutting is also more indicating higher wear which is in accord with the observation. In the magnified view fibre piece (4d), fibre on the verge of pulverization due to microcracking followed by microcutting mechanisms can also be seen. Micograps 4e and f are for worn surfaces of composites D and E respectively. Overall damage to the surface and fibres is higher compared to the earlier composites explaining the higher wear of these composites.

4. Conclusion

Various composites of PA 6 filled with different percentages of short glass fibres and fillers were formulated and characterised for their compositional, thermal and mechanical properties followed by evaluation in abrasive wear condition. It was observed that the mechanical properties except elongation to break increased with increase in percentage of GF. Inclusion of fibre/filler, however, deteriorated abrasive wear peformance of the parent polymer. The higher the percentage of the filler, the higher was the deterioration in wear behaviour and this was correlated to reduction in *Se* factor. Good linearity was observed in the case of wear rate vs $(Hse)^{-1}$ plot, which indicated all the three parameters i.e. hardness (*H*), ultimate tensile strength (*S*) and elongation to break (*e*) controlled the wear behaviour of the composites.

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