On the abrasive wear of some polyimides and their composites*

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The tribological properties are not intrinsic material properties; they depend on the experimental conditions. Composites that are very suitable for one type of application may not be useful for others. With this view, it was proposed to investigate the wear performance in extreme environmental conditions of some polyimide composites with proven properties in the adhesive wear mode. For this purpose the wear characteristics of ten such composites were investigated in extreme abrasive conditions under various loads. Fibre reinforcement and fillers (solid lubricants) with the exception of MoS₂ affected the wear performance in an unfavourable way. The increase in wear with increasing percentage of fillers was attributed to the fact that the composites exhibited lesser ultimate tensile elongations than those of their parent polymers. The load and abrading particle size were observed to be important parameters in these studies.

Keywords: polyimide composites, ultimate tensile strength and elongation, Ratner–Lancaster plots, reinforcement, particulate fillers

Introduction

Abrasive wear is probably the most common cause of mechanical damage in the engineering industries. Extensive work has been done on the influencing parameters and consequences in the abrasive wear of metals1–2. However, comparatively less attention has been given to the abrasive wear of polymers and their composites. Some of the applications requiring abrasion-resistant polymeric materials or composites include vanes and gears in pumps handling industrial fluids, sewage- and abrasive-contaminated water, roll neck bearings in steel mills subject to heat, coke, coal and mineral ores, guides in bottle handling plants, bushes and seals in agricultural and mining equipment, and sluice gate bearings3–4.

Most of the studies hitherto reported seem to be about investigating the influence of fibres on the abrasive wear of composites5–8, while the literature about the effect of particulate (powdery) fillers on the wear performance of composites in an abrasive environment is limited4–9,10. Data on the abrasive wear performance of commercially available composites are available from the manufacturers. However, hardly any data are furnished on the abrasive wear characteristics of such composites although these are important for understanding the wear behaviour in extreme abrasive conditions and in failure analysis.

From a survey of the literature, it is seen that the influence of additives on abrasive wear performance cannot be predicted a priori4–8,9. Although fillers and reinforcements increase the strength and stiffness of polymers, there is generally a reduction in the factor Se, where S and e are stress and strain at ultimate rupture during tension. Since the abrasive wear of the composites is related to (Se)–1, composites may not necessarily perform better than the parent polymers12–13.

In the light of this, two types of polyimides (thermoplastic and thermosetting), various composites filled with particulate fillers and/or short fibres were selected for investigating the influence of various parameters (such as fillers, load and abrading particle size) on abrasive wear. The studies were performed by abrading polymer pins in the single-pass condition against silicone carbide (SiC) abrasive papers of various grades. The single-pass condition was preferred for two reasons. Firstly, the additional effects originating from the transfer of polymer and/or filler to the paper is a minimum. Hence, a comparison among various materials and correlation of wear resistance to appropriate mechanical properties becomes possible. Secondly, the single-pass condition is closer to the situations in applications such as wear strips, chute liners and sleeve bearings and bushes in the liquid environment that wash away debris and prohibit film transfer4.

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Experimental conditions
Polyetherimide (pei) a thermoplastic polyimide commercially known as Ultem resin manufactured by the General Electric Company and its various composites (Table 1) and a thermosetting polyimide (pi) marketed under the trade name Vespel plastics by Du Pont and its various particulate filled composites (Table 1) were selected for the present studies. Both types of composites have proved promising in adhesive wear performance\textsuperscript{12,15}. We procured pei and its composites (except sample D) in the form of granules, which were then injection moulded into rods (5.3 cm length and 8 mm diameter) and plaques (4 cm × 2 cm × 0.4 cm). Pellets of pei composites cut from the rods had fibres normal to the sliding plane while pellets cut from the plaques had fibres parallel to the sliding plane. Composites of Vespel were supplied in the form of rods. Hence, pellets of the required size could be cut and machined from the rods. By using the proper sample holders, these polymer pins (7 mm diameter and 3 mm thickness) could be abraded against abrasive papers.

The abrasive counterface
Waterproof silicon carbide (SiC) emery papers of various grades (mesh sizes 600, 400, 320, 220, 180, 150 and 120) were used to study the effect of abrading particle size on wear. For studying the effect of load, abrasive papers of fixed mesh size (220) were used.

Abrasive wear studies
These were performed with a pin on a disc machine, which is described elsewhere\textsuperscript{10}. A disc fitted with an abrasive paper was rotated at a selected speed (5 cm s\textsuperscript{-1}). Pins were slid against a rotating paper in a circular path of diameter 7 cm. The total sliding distance in each experiment was 175 cm, while the loads were varied from 4 N to 12 N. Before starting the experiment, the pins were rubbed against a similar type of paper under the same experimental conditions until they showed uniform contact. Before and after the wear tests, the pins were cleaned in an ultrasonic cleaner with petroleum benzene, then dried and weighed until a constant weight was recorded. The pin surfaces were examined under an optical microscope to ensure that no emery particles were sticking to the pin surface. Wear was measured by loss in weight, which was then converted into volume loss using density data.

The specific wear rate ($K_a$) was calculated from the following equation:

$$K_a = \frac{V}{LD} \text{ m}^3 \text{ N}^{-1} \text{ m}^{-1}$$

where $V$ is the volume loss and $L$ is the load, while $D$ is the distance abraded.

SEM studies
In typical cases pin and paper surfaces were made conducting by silver sputtering, and observed with a scanning electron microscope (Philips 515).

Results and discussion
Wear data as a function of load for pei, pi and their composites are plotted in Figs 1 to 4. Figure 5 is a histogram indicating the comparative performance of composites in identical sliding conditions. The

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<th>Table 1 Details of the composites studied (manufacturer’s data)</th>
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<td>Material</td>
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<tr>
<td>Polyetherimide (Ultim) series (GEC) pei</td>
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<td>A</td>
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<td>Polymide (Vespel) series (DuPont)</td>
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* Analysed in the laboratory
** Prepared in the laboratory
1gf, glass fibre
correlations between wear and the appropriate mechanical properties for PEI and PI composites are shown in Figs 6 and 7. The relation between wear and abrading particle size is shown in Fig 8. In Fig 9 SEM micrographs of paper surfaces after wearing against PEI and PI are shown. The effect of abrading particle size and load on PI pin surface topography is shown in micrographs collected in Fig 10.

In Figs 1 and 2, the wear and specific wear rates of PEI and its composites are plotted as a function of load. For the composites C and E two fibre orientations were selected. Cn, Dn, and En represent composites with fibres normal and parallel to the abrading surface, respectively.

Fig 1 Abrasive wear of PEI and its composites as a function of load (velocity 5 cm s⁻¹, distance abraded 4 m and paper grade 220 mesh size). The subscripts n and p denote composites with fibres normal and parallel to the abrading surface, respectively.

Fig 2 Specific wear rates of PEI and its composites as a function of load (velocity 5 cm s⁻¹, distance abraded 4 m and paper grade 220 mesh size).

Fig 3 Abrasive wear of PI and its composites as a function of load (velocity 5 cm s⁻¹, distance abraded 4 m and paper grade 220 mesh size).

Fig 4 Specific wear rates of PI and its composites as a function of load (velocity 5 cm s⁻¹, distance abraded 4 m and paper grade 220 mesh size).
The abrasive wear characteristic is linear in all cases. However, for composites with a larger percentage of fillers, or more than one filler the wear increases rapidly with the increase in load (eg., samples D, E, P, R and Q).

The wear of the parent polymer is the least (with the exception of the composite S, which shows slightly less wear than that of the parent polymer) The order is as follows: 
A < C_o < C_o < B < D_o < E_o < E_o, 
for the pi series, and 
S < O < P < R < Q, 
for the pi series.

In all cases, except for the composite, S, wear increases as the amount of additives increases.

The abrasive wear rates of pei and composites are in the range $4 \times 10^{-11}$ to $2 \times 10^{-10}$ m$^{-1}$ N$^{-1}$ m$^{-1}$, while for pei and the composites the variation is over a wider range, $1 \times 10^{-11}$ to $1 \times 10^{-9}$ m$^{-1}$ N$^{-1}$ m$^{-1}$. The wear rate of pei is the least.

The specific wear rates show an increase with the increase in load for all the composites except for the composite D_o, where the trend is the opposite.

Composites with fibres normal to the abrading surfaces show less wear than those with fibres in planes parallel to the surface.

Comparison of the wear performance of the composites B and C show that glass fibres are less detrimental to abrasive wear performance than particulate fillers such as pte.

When fibres are incorporated with particulate fillers, wear increases enormously, as seen in the cases of samples D and E. This indicated that fibre incorporation along with powdery fillers have a considerable adverse effect.

Thus, the most important feature brought out by this study is the negative influence of fillers/fibres on the abrasive wear performance of composites. A similar effect is also shown in the composites in the multipass condition. In the literature the mixed trend is observed in the case of effect of filler on the abrasive wear of polymer composites. Among thirteen composites reinforced with 30% carbon fibres, Lancaster has reported improved wear resistance for seven composites and a detrimental influence of fillers on six composites. A similar mixed trend is reported by Thorp for the series of composites of polyurethane, nylon and polyurethane filled with particulate fillers. There is much evidence that fillers or reinforcement leads to a deterioration in the wear performance. However, the composites with long and continuous fibres increase the abrasive wear resistance of the composites. The literature indicates that thermoplastic polymers show a better performance than thermosetting polymers in abrasive wear studies. In the present work the thermoplastic polyetherimide was also proved to be superior to thermosetting polyimides.

Another feature emerging from the Figs 2 and 4 is the sharp decrease in the specific wear rates of all the composites (except sample D) with an increase in load. This agrees well with Lhymn's mathematical model, accounting for crack propagation in fibre-reinforced composites where abrasive wear rates are inversely proportional to the normal load. According to Lancaster, the clogging effect (contamination of wear tracks with wear debris) is also possible in the single-pass condition, the extent of which strongly depends on the size and shape of the pin. This effect increases with the increase in load resulting in a sharp decrease in the wear rates. The opposite behaviour (an increase in wear rate with increase in load) of sample D, which was formulated in the laboratory, could arise from improper adhesion between filler and binder.

In the histogram (Fig 5) the comparative performance of all the composites abraded under both maximum (12 N) and minimum (4 N) loads is shown. Unfilled pei showed minimum wear while the composite Q (pi + 40% graphite) exhibited the highest wear.

![Fig 5 Histogram showing comparative abrasive wear performance of selected composites abraded under maximum (12 N) and minimum (4 N) load in the single-pass condition (velocity 5 cm s$^{-1}$, distance abraded 4 m and paper grade 220 mesh size)](image-url)
In Figs 6 and 7 correlation of wear with the relevant mechanical properties is shown. For the pei series, both the Rainer–Lancaster plot (wear against $S^{-1}$, where $S$ is the rupture stress and $e$ is the ultimate elongation at break) and a plot of wear as a function of $e^{-1}$, are not strictly linear (Fig 6). This shows that the abrasive wear mechanism depends on the nature of the filler incorporated. The composites B, C and E contained, respectively, particulate filler, fibre and mixtures of both. Though wear is roughly proportional to $(S e)^{-1}$ or $e^{-1}$, it was not perfectly linear, indicating that fibres and fillers interacted with the abrading surfaces in different ways. Fibres may pull out abrading particles or damage them while powdery fillers could change the abrading efficiency by the clogging effect or transferring a film of solid lubricant on the emery particles. Thus, for the composites with heterogeneous fillers, it is difficult to establish a predictable relationship between wear and the mechanical properties.

In the case of pi composites, although all of them contained powdery fillers only, the composite O showed a different behaviour from the rest. In all the correlations attempted (Fig 7), wear as a function of $H^{-1}, e^{-1}, S^{-1}$ and $(H e)^{-1}$, the point corresponding to Q did not agree. While with other samples (O, P, R and S) linearity was observed in the correlation between wear and the relevant property. It seems that when the filler percentage increased excessively (as in the case of sample O), a disproportionate amount of wear occurred.

The relation between wear and abrading particle size is plotted in Fig 8. For all the samples the wear increased continuously with the increase in grit diameter, although the extent of the wear rate differed from sample to sample. Above a typical grit size (52 μm) this increase in wear was either slow (for the samples A, B, C, O and S) or very rapid (for the samples E, P, R and O).

The size effect is an important feature observed in the abrasive and erosive wear of materials. Above a critical particle size, wear either shows a small increase or no increase at all with increasing grit diameter. This effect has been observed extensively in the case of metals. In the case of polymers and their composites, however, a small increase is reported in this respect, and the reasons behind this effect are not fully understood. Misra and Finnin, after reviewing this phenomenon for metals, concluded that this is the result of a shallow surface layer that exhibits a higher flow stress than that of the bulk material when the metals are abraded or eroded. This theory cannot be applied to polymers since work hardening does not take place in such materials. In studies with two metals and one polymer,
so that as the size of abrasive particle decreases the wear mechanism undergoes a transition from cutting to delamination wear.

In our earlier studies\textsuperscript{16, 20} about the wear of a number of composites (in the single-pass or multipass condition) as a function of grit size, it was observed that no unique pattern emerged. For some polymer samples above a typical grit size (52 \( \mu \)m) the wear increased more or less rapidly as in the case of the present investigations. It is likely that the wear mode changes from deformation to cutting above a typical range of grit diameter. This transition in wear mechanism may be strictly true for bulk polymers only. In the case of composites, however, various types of fillers/fibres also play an important role in the wear process. Hence, though the wear mode changes from delamination to cutting, the wear of the composites shows either a slow or rapid increase in wear rather than showing a saturation effect.

SEM studies of abraded surfaces are shown in Figs 9 and 10. In Fig 9 micrographs of abrasive papers (220 grade, 52 \( \mu \)m grit diameter) are shown after wear against pei and pi under a load of 8.5 N. The noticeable

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure8.png}
\caption{Abrasive wear as a function of abrading particle size: (a) for the pei series, (b) for the pi series (velocity 5 cm s\(^{-1}\), load 8.5 N).}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{figure9.png}
\caption{SEM micrographs of paper surfaces (220 mesh size) worn against (a) pei and (b) pi at a load of 8.5 N.}
\end{figure}

\textsuperscript{19} Sin et al. observed this typical size effect. According to them, depending on the \( w/r \) ratio (where \( w \) is the width of groove and \( r \) is a measure of the sharpness of the particle), the material surface deforms or is cut
The difference between the two types of wear debris is that the debris in the case of PEI is less in amount and shows plastic failure, while in the case of PI the debris has a fibrous nature and at some spots small patchy films are transferred on the tips of emery particles. In the multipass condition, this difference could be seen distinctly. SEM studies seem to support the view that the failure mode of the material is not the same in all cases. It was expected that, since PEI is more ductile than PI (Table 1), this would reflect in their failure modes. However, the SEM studies in this case and elsewhere showed that PI wear debris are more fibrous than those of PEI.

Figure 10 contains micrographs of PI pin surfaces abraded under different loads and particle size conditions. Micrographs 10(a) and 10(b) are for the pin abraded under a load of 8.5 N against 600 grade emery paper, while micrographs 10(c) and 10(d) show a pin surface abraded under a 12 N load against 120 grade paper.

The pin surfaces are full of deep furrows (which can be seen in magnified views) generated in the direction of abrasion owing to the 'ploughing' action. The severity of the abrasion and the depth of the furrows are greater in the micrographs 10(c) and 10(d) because of higher loads and particle sizes. In these micrographs (10(c) and 10(d)) the cutting action seems to play a prominent role, rather than the ploughing action.

Conclusions

Based on the studies of various polyimide composites with different fillers under varying conditions of load and abrading particle size, the following conclusions emerge:

All fillers and fibres (except for MoS₂ filler) reduced the abrasive wear performance of the parent polymers. In fact, wear increased rapidly with filler content. Wear was roughly proportional to the factor \( (S/\varepsilon)^{-1} \), where \( S \) and \( \varepsilon \) are the rupture stress and elongation at break, respectively. The composites that showed a very good performance in adhesive wear studies against smooth metals, were observed to be totally unsuitable in abrasive wear performance. The abrading particle...
size was observed to be an important parameter. The normal size effect as in the case of metals was not observed. However, above a critical grit size, wear increased more or less rapidly depending on the material.

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