# **Friction and wear studies of bulk polyetherimide**

# J. BIJWE, U. S. TEWARI,

*Industrial Tribology, Machine Dynamics and Maintenance Engineering Centre (ITMMEC), Indian Institute of Technology, New Delhi-110016, India* 

P. VASUDEVAN

*Centre for Rural Development and Appropriate Technology, Indian Institute of Technology, New Delhi- 110016, India* 

Polyetherimide, ULTEM 1000, was studied as a neat resin for investigating its friction and wear behaviour while sliding against mild steel, under unlubricated conditions on a pin on disc machine. Experimental parameters selected were different loads, sliding distances and counterface roughness. It was observed that unlike other semicrystalline highly linear thermoplastics and some thermoplastic thermosetting polyimides, this polyimide does not transfer a film on the counterface under the conditions of the study. It seems that it wears mainly by fatigue mechanism but crack propagation could not be observed in SEM micrographs of pin surfaces. The frictional coefficient was low as compared with other polyimides and varied with contact pressure from 0.4 to 0.22, although the initial value was high. The cycles required to initiate wear particles were around 90 kcycles at 13 N  $cm^{-2}$  contact pressure. Once the wear started, the specific wear rate was high  $(K_0 = 10^{-13} \text{ m}^3 \text{ N}^{-1} \text{ m}^{-1})$ .

# 1. **Introduction**

The increasing use of polymers in applications involving sliding motion makes wear studies imperative from technological and commercial stand points since it is well known that generation of heat at sliding contact of polymers can produce local decomposition, thus limiting the usefulness of polymers in sliding applications. Some heat resistant polymers developed in recent years are expected to be useful under severe sliding conditions. Polyimides, polyamideimide, polyether-ether ketone, polyphenylene sulphide and reinforced PTFE are some of the potentially useful materials selected for tribological studies [1-9]. However, these PTFE-based composites and thermoset polyimides can not be easily processed despite very good friction and wear properties. There is hence an increasing trend towards choosing processable thermoplastics [4-9].

The major portion of research on the friction and wear of polyimides has been done by Fusaro [10, 11]. He found that polyimides fall into two groups according to friction and wear properties. Group I polyimides generally have lower friction and higher wear than group II polyimides. The wear of both groups, occurs by an adhesive wear mechanism with larger particles being formed by Group I polyimides [10]. His results suggested that a linear non-crosslinked polyimide will have a higher wear rate than a cross-linked polyimide. He had noted that polyimides undergo a transition from high friction, high wear to low friction and low wear as the temperature is increased above  $40^{\circ}$  C [11]. Most of the studies done on polyimides are

focused on thermosetting polyimides. Hence, it was of interest to estimate tribological behaviour of thermoplastic polyimide.

Polyetherimide, commercially produced as ULTEM 1000 by General Electric Company, is a high performance engineering thermoplastic, exhibiting a combination of outstanding mechanical, thermal and electrical properties. In the structure of polyetherimide (Fig. 1), aromatic imide units provide stiffness and high heat resistance while ether linkages allow for good melt flow characteristics and processability [12, 13]. The main features of polyetherimide are the following.

(1) High mechanical strength and its retention at elevated temperature (flexural modulus 3300 x  $10^6$  Nm<sup>-2</sup>, outstanding for unmodified thermoplastic resin, exceptionally strong with tensile strength at yield in excess of  $105 \times 10^6$  Nm<sup>-2</sup> and flexural strength  $145 \times 10^6$  Nm<sup>-2</sup>).

(2) High temperture stability (Vicat softening point  $219^{\circ}$ C melting point  $380^{\circ}$ C.

(3) Inherent flame resistance with extremely low smoke evolution.

(4) Outstanding electrical (insulating) properties over a wide range of temperature and frequency.

(5) Good chemical resistance to aliphatic hydrocarbons, acids and dilute bases.

(6) Very good stability to UV and gamma radiations.

(7) Ready processability on commercial instruments.

This unique combination of properties made the



investigation of tribological behaviour of this polymer interesting, as this has not been reported in the literature.

## **2. Experimental procedure**

## 2.1. Sample preparation

Mild steel with a Rockwell hardness RB 80, was chosen as one of the wearmates of a sliding system. These discs were ground with a grinding wheel in circular motion. For a smoother surface  $(R_{\rm s} \leq$ 0.09  $\mu$ m) discs were polished using different grades of diamond pastes and polishing cloth. The c.l.a surface roughness  $(R_a)$  of the discs was measured on Talysurf 6 (Taylor Hobson) by averaging over four locations 90 $^{\circ}$  apart on the track. C.l.a.  $R_{\rm a}$  is defined as arithmetic mean of the departures of the profile from the centre line. Polymer pins (7.3 mm diameter and 1 cm length) were machined from the injection moulded rods. Pins were generally prerubbed to get maximum contact with the counterface. Pins and discs were cleaned with

*Figure t* (a) General structure of poiyetherimide (PEI), (b) structure of ULTEM 1000.

acetone and dried before each experiment. After wearing, pins were cleaned with tissue paper and soft cloth. Since the polymer is slightly moisture sensitive, pins were allowed to cool in air to reach constant weight.

## 2.2. Tribological studies

Friction and wear studies were done on a pin on disc machine as shown in Fig. 2 which consists of a single polymer pin fixed in a holder and a disc rotating with selected r.p.m, against this pin. Different contact pressures were achieved by applying dead weights on the pin. Frictional track diameter was fixed  $(70 \text{ mm})$ . Frictional torque was measured with a strain gauge transducer and wear was measured by loss in weight which was then converted into volume loss  $(V)$  using density data.

The specific wear rate  $K_0$  was determined from the relationship,

$$
K_0 = \frac{V}{px} (m^3 N^{-1} m^{-1})
$$





*Figure 3* Infrared spectrum of PEI (-----) and its wear debris (---).

where  $V$  is the volume loss against sliding distance  $x$ at load  $p$ . All tests were carried out at ambient temperature.

#### **3. Characterization of the polymer**

Elemental analysis of PEI showed  $C = 75\%,$  $N = 4.01\%$ , and H = 3.7% which agrees well with calculated values. The infrared spectrum (Perkin Elmer 580 B Infrared spectrophotometer) of PEI is shown in Fig. 3. The sharp band at  $5.62 \,\mu m$  (1780 cm<sup>-1</sup>) and broader and stronger band at  $5.82 \mu m$  (1740 to  $1715 \text{ cm}^{-1}$ ) are the characteristic bands for imide structure. The strongest bands between 7.5 and 11  $\mu$ m are likely to be due to ether linkages. A very predominant band at about  $8 \mu m$  arises from diaryl ether group. This band is clearly indicated in the spectrum (Fig. 3). The series of bands which are related to methyl groups in complex structures [14] are compared with those observed in PEI spectrum as shown below

It is clear that PEI has methyl group arrangement as in type 2. Bands at 2960 and  $2870 \text{ cm}^{-1}$  which are for C-H stretching in methyl groups, are difficult to observe except when methyl concentration is high [14]. In the spectrum (Fig. 3), bands at 2970 and  $2875 \text{ cm}^{-1}$ are also present. The band at  $1450 \text{ cm}^{-1}$  is for  $-CH_3$ group since the bands which are characteristic of the  $CH<sub>2</sub>$  group at 2925, 2860 and 2720 cm<sup>-1</sup> for -CH bending are absent (Fig. 3). Thus, the proposed structure of polyetherimide is strongly supported by IR studies.

Thermal properties of the polymer were checked on a Du Pont 1090 analyser at a scanning rate of  $10^{\circ}$ C  $min^{-1}$ . Thermogravimetric analysis and DTG curves in nitrogen atmosphere are given in Fig. 4. It seems that the thermal stability of PEI is up to  $536^{\circ}$ C (in nitrogen atmosphere), after which, it starts degrading. Maximum degradation temperature is around  $558^{\circ}$ C, with 47.24% of the resin degraded. Differential scanning calorimetric studies of the polymer at a scanning





*Figure 4* Thermogravimetric analysis of PE1 in nitrogen atmosphere.

rate of  $10^{\circ}$ C min<sup>-1</sup> in static air is shown in Fig. 5. Glass transition temperature  $(T<sub>g</sub>)$  is seen to be around  $216^{\circ}$ C and the melting point around 380 $^{\circ}$ C (Fig. 5).

## **4. Results and discussion**

Wear data over different sliding times, distances, and loads are plotted in Figs 6 to 9 while variation in friction against time and load is shown in Figs 10 and ll. In Figs 6 and 7, wear data with different pin history have been plotted.

In the initial studies on PEI, it was observed that while sliding under high P and V values ( $P$ -102 Nm<sup>-2</sup>; 2.1 m sec<sup> $-1$ </sup> and time interval 30 min), pin did not show any significant wear within two intervals. (It should be noted that after every interval, pin was cooled and weighed.) However, in third interval, before its completion, friction coefficient suddenly reduced and catastrophic wear due to melting of polymer took place. The thick layer of molten material was transferred on the disc. The experiments were repeated on other PEI pins under comparatively low *PV* value. Every time, it was observed that after typical sliding distance depending on *PV* value, catastrophic wear took place. However, in these cases melting of polymer did not occur. Disc surface was covered with fine powdery wear debris. Such typical behaviour could be due to fatigue induced in the pins. Similar behaviour in case of UHMWPE (ultra high molecular weight

polyethylene) while sliding for long duration was observed and reported due to fatigue wear [15]. It was of interest to confirm this. From this point of view, two experiments were performed.

In first experiment, a pin was not prerubbed but machined carefully for full contact with the counterface. The weight loss after certain time intervals under the sliding conditions as shown in Fig. 6, was determined. It was observed that after the initiation of wear (after 42kcycles), it was first stabilized for some period and then again catastrophically increased later. In another experiment (Fig. 7), a pin prerubbed on steel disc for full contact with the counterface was used. It did not indicate much wear in initial period. However, after 12min (7kcycles) significant wear started. In all experiments, pins were used after wear had been initiated. A fresh disc with same  $R_a$  value was used in each experiment.

The following are the salient features in the friction and wear studies on PE1.

(1) Under high contact pressure  $(44 \text{ N cm}^{-2})$ wear was very high. Hence the maximum contact pressure was restricted to  $25 \text{ N cm}^{-2}$ .

(2) Certain number of cycles were required to initiate any significant wear of a fresh pin surface. The point at which wear initiated depended on load as well as history of the pin surface preparation.

(3) In the case of smooth counterface surfaces



*Fis:ure 5* Differential scanning calorimetry of PEI in static air.



*Figure 6* Wear plotted against time for PEI sliding against mild steel,  $R_a = 0.3 \,\mu \text{m}$ , r.p.m. 200, speed 0.73 m sec<sup>-1</sup>, contact pressure  $13$  N cm<sup>2</sup>.

 $(R_a \leq 0.08 \,\mu\text{m})$  friction was very high (almost double that for moderately smooth surfaces) and frictional track was full of deep grooves though the wear of the pin was insignificant.

(4) In the case of moderately smooth surfaces  $(R_a \simeq 0.3 \,\mu\text{m})$  friction coefficient observed was less than that for other polyimides but wear was high. A large amount of wear debris were collected on both sides of the track. However, there was no film transfer on the counterface. The IR spectrum of very fine powdery yellow debris was similar to that of parent polymer, thereby indicating no chemical degradation of the polymer during the tests (Fig. 3).

Polyetherimide wear  $-$  the wear of a polymer is generally caused by one or more of the following mechanisms: adhesive transfer, abrasive cutting and fatigue. When polymer slides on a very smooth and



*Figure 7* Wear plotted against time, for PEI (prerubbed on mild steel)  $R_a = 0.3 \,\mu\text{m}$ , contact pressure 5.2 N cm<sup>-2</sup>, speed 2.08 m sec<sup>-1</sup> r.p.m. 570.



*Figure 8* Wear behaviour of PEI for various sliding distances,  $R_a = 0.3 \,\mu\text{m}$ , speed 2.08 m sec<sup>-1</sup>, contact pressure 5 N cm<sup>-2</sup>.

hard surface, wear occurs by adhesive transfer. On a rough surface with sharply pointed asperities, abrasive cutting is the dominant wear mode. For surfaces with more gently rounded asperities which do not cause stresses in the polymer large enough to cause cutting, many stress cycles (repeated traversals) are required to produce wear particles, This mode is called fatigue wear. In the present studies, when the surfaces were very smooth ( $R_a \leqslant 0.08 \,\mu\text{m}$ ), the friction coefficient was very high. Instead of the pin surface, the metal surface was worn out. Possibly strong adhesion occurs in the case of such a smooth surface due to maximum asperity contacts, ultimately resulting in an increase in contact area. Since the pin surface was hard enough to resist adhesive wear, the metal surface was grooved. At higher pressure and speed;  $(28 \text{ N cm}^{-2}, 2.1 \text{ m})$  $sec^{-1}$ ), due to very high frictional heating, the pin surface was damaged, producing sparks while sliding and ultimately the pin failed. This failure can be seen in the SEM photograph.

For moderately rough surfaces  $(R_a \simeq 0.3 \,\mu\text{m})$ fatigue wear seems dominant. In similar work, the experimental conditions being almost the same, similar features have been reported [3] for different polyimides. The incubation time (time required to initiate wear) observed was explained due to contact stresses which were below the magnitude required to generate wear particles. It could also be due to the existence of a contaminated film which is worn away during this period and then giving rise to wear particles [3]. However, in the case of PEI an explanation based on contaminated film on metal disc may not be valid since once the pin starts wearing, it produces wear particles on any fresh disc surface immediately. Hence, the fatigue phenomenon seems to be the basic factor incubation time. For three polyimides, reported incubation time ranged from 100 to 600 cycles [3]. Under selected experimental conditions, PEI showed better fatigue resistance (40 to 90 kcycles) (Fig. 6), In fact, once wear was indicated (after 40kcycles) it was stabilized and there was no appreciable wear again till 90kcycles. This could be due to surface fatigue first in the layer which was just in contact with



*Figure 9* Affect of load on wear behaviour of PEI  $(R_a = 0.3 \,\mu\text{m}, \text{sliding speed } 2.08 \,\text{m} \,\text{sec}^{-1}, \text{sliding}$ distance 1 km.

the sliding surface. Once this layer gets worn away, there is no wear. After 90kcycles, it becomes bulk phenomenon and continuous wear is recorded after each interval. At this stage, the specific wear rate was in the range of  $10^{-13}$  m<sup>3</sup> N<sup>-1</sup> m<sup>-1</sup>.

As shown in Fig. 7, incubation time depended on the history of the pin surface preparation and load conditions. Since this pin was already prerubbed against m.s, it started wearing after 7 kcycles (12 min).

Once the pins start wearing, they show a wear against sliding time relationship as indicated in Fig. 8. In this case, a continuous increase in wear is observed with sliding distance, indicating nonoccurrence of a film transfer. In contrast, in the case of polymers where film transfer occurs, wear increases steeply in the beginning and once the transfer film stabilizes surface topography, not much further wear is noted [16, 17].

In the specific wear rate against contact pressure relationship, (Fig. 9), it seems that specific wear rate shows slight increase with the contact pressure and is in the range of  $10^{-13}$  m<sup>3</sup> N<sup>-1</sup> m<sup>-1</sup>. Whereas for most other polymers and composites which transfer films  $K_0$ values decrease with the increase in contact pressure and are in the range of  $10^{-13}$  to  $10^{-16}$  m<sup>3</sup> N<sup>-1</sup> m<sup>-1</sup> which are dependent on several experimental parameters.

It is known that relatively ductile polymers such as polyethylene, polypropylene and polytetrafluoroethylene form transfer films which effectively smoothen the counterface and decrease wear. Polymers with relatively small elongations, however, such as polystyrene and ABS, do not easily form a beneficial layer and hence a degree of counterface roughness only increases wear. A feature of glassy polymers and, indeed, elastomers is that they do not transfer coherent films of relatively degraded material to the counterface. However, when frictional heating is substantial, the glass transition temperature of the polymer is exceeded and gross transfer of molten material to the counterface occurs. Sliding wear of such polymers, e.g. PMMA, is caused by crazing and brittle failure. When there is no thermal softening of surface layers, the wear debris remains large, thick and lumpy. While

in the case of ductile polymers, only linear, symmetrical and semicrystalline polymers without any pendant groups seem to exhibit low friction and the occurrence of thin oriented transfer film on the counterface as sliding commences. The tribological behaviour of other semicrystalline polymers e.g. LDPE, PP and PA (polyamides) is different. Their wear debris are thicker and lumpy indicating that their molecules are not as elongated as in highly semicrystalline polymers [16]. Little work has been carried out on glassy polymers. They are essentially brittle materials in tension but the hydrostatic stresses generated in the contact region will convey significant ductility. The ductile brittle transition pressure for polymethylmethacrylate and polystyrene at 20 $^{\circ}$ C is 3 × 10<sup>7</sup> Pa. Significant plastic deformation has been reported for such systems [18].

PEI is an amorphous but ductile polymer with high impact strength. It has bulky groups in the chain backbone. Hence it shows mixed wear behaviour.



*Figure 10* Frictional behaviour of PEI at various contact pressures (sliding speed 0.73 m sec<sup>-1</sup> and ( $R_a = 0.3 \,\mu$ m). (1, 2.43 N cm<sup>-2</sup>; 2,  $5.20\,\mathrm{N\,cm^{-2}}$ ; 3, 24.50 N cm<sup>-2</sup>).



*Figure 11* Frictional behaviour at various contact pressures, (speed  $0.73 \text{ m}\text{ sec}^{-1}$  $R_a = 0.3 \,\mu\text{m}$ , sliding time = 10 min). (o kinetic, x static).

Though ductile, it does not wear by a film transfer irrespective of counterface material, and roughness, sliding velocity, distance and load. As in the case of glassy (brittle) polymers, however, its wear debris is not large, thick and lumpy, but in the form of very fine powder. Its ductile-brittle transition pressure is possibly higher than PMMA and PS, hence significant plastic deformation could not be seen at normal loading condition except in case of pin failure (Fig. 16).

Thus it seems that generally adhesion bonds to the counterface are weaker than those within the polymer and shearing occurs basically at the interface without any film transfer. When, however, the counterface is smooth meaning that the adhesive bonds are stronger, severe plastic deformation and brittle fracture occurs. Thus, though amorphous, this polymer does not wear by crazing or brittle fracture within observed experimental conditions. This may be due to its very good impact strength, hardness and reasonably good ductility.

In the case of PEI composites, pins and disc surfaces worn out in extreme load and sliding distance conditions, when analysed with the SEM, revealed that (i) gross transfer of molten polymer took place on the disc surface, (ii) severe melt flow of polymeric material could be seen on the pin surface, (iii) cracks parallel as well as transverse to the sliding direction were observed on the pin surfaces [19]. According to Jain *et al.* [4] these transverse cracks as observed in the case of filled PAl, were due to fatigue wear of the polymer.

For polyetherimide friction, the coefficient of friction depends on the material, counterface and its topography, load, sliding time and speed. As seen in Fig. 10 as sliding commences,  $\mu$  falls and stabilizes to a certain value. Generally, such behaviour is observed in highly crystalline, linear polymers which transfer a thin and highly oriented film due to which the frictional coefficient decreases [16]. In the case of brittle material like PMMA, Jain *et al.* [20] observed that transfer occured in the form of large number of fragmented irregular particles which had filled the asperity cervices in a number of locations and were scattered all over the surface increasing the roughness of the disc slightly in the direction of sliding. After further sliding, fragments of polymeric materials filled the cervices and a uniform coating of the deposited material was formed. This decreased the surface roughness to a minimum value. In the case of PEI, though it does not transfer a film, very fine powdery material accumulates on the wear track of the counterface. This wear debris fills the crevices modifying the surface topography to the extent that it decreases the friction coefficient to a stable value. However, in the case of a smooth surfaces ( $R_a \leq 0.08 \,\mu\text{m}$ ), a high frictional coefficient was observed in the case of mild steel, stainless steel, brass and an aluminium counterface without any film or material transfer. On the contrary, the surface became grooved. The same trend is also observed for PEI composites. This must be because of maximum asperity - asperity contact leading to more adhesion to the counterface.



*Figure 12* SEM micrograph of disc surface after the wear initiation.



*Figure 13* Energy dispersive X-ray analysis (EDAX) spectrum of wear tracks before  $($ ——) and after cleaning  $(-,-)$  with solvent.





The dependence of  $\mu$  on load is shown in Fig. 10, The initial friction seems to be higher than the dynamic due to mechanical interlocking of the asperities. Both frictional coefficients decreased with the increase in contact pressure. This could be explained as follows. Polymeric materials have low bulk moduli and therefore shear strength of these materials is affected by the hydrostatic pressure which changes the intermolecular spacing. This change in flow strength of the material with the normal load affects the size and contact area and hence the frictional coefficient more sensitively than in the case of metals [16]. The coefficient of friction for PEI is comparatively low if compared to the literature values. Tanaka *et al.* [5] observed  $\mu \approx 0.5$  for Vespel polyimide which seemed to decrease slowly with increase in load. For PEI,  $\mu$ is around 0.4 which decreased to 0.2 as the load increased. It seems that Fusaro's prediction about non-crosslinked linear polyimides held good for PEI except for the nature of wear debris and wear mechanism.

#### **5. SEM studies of worn surfaces**

Scanning electron microscopy (SEM 515, Philips) was used for investigating worn disc and pin surfaces and wear debris in typical cases. Results, in the form of micrographs and EDAX spectra are shown in Figs 12 to 16. In Fig. 12, the disc surface on which wear was just initiated and light deposition of fragmented wear debris had started (as in Fig. 7) has been photographed. The surface seems to be covered with a number of parallel wedge shaped asperities due to the grinding process. The EDAX spectrum of a disc surface where no particles were sticking did not show any indication of nitrogen which is one of the main constituents of a polymer unit (Fig. 13). This indicated non-existence of polymeric film on the surface. For further confirmation this surface was cleaned carefully with dichloromethane, a solvent for PEI and then observed with SEM. The EDAX spectrum of this cleaned surface as shown in Fig. 13 is identical to the previous spectrum. Thus, it is clear that material transfer was in the form of fragmented irregularly shaped particles and not a film.

In Figs 14 to 16 typical SEM micrographs of pin surfaces and wear debris are shown. A pin surface prerubbed on a steel disc is shown in Fig. 14 indicating smooth topography. The effect of load variation on surface topography while sliding is shown in Fig. 15. When contact pressure is low, the surface is comparatively smooth and shows longitudinal furrows in the sliding direction due to microploughing. The track in



*Figure 15* SEM studies of (a) worn pin surface, load  $5 \text{ N cm}^{-2}$ ; (b) worn pin surface, load  $25 \text{ N cm}^{-2}$ .



*Figure 16* Failure of PEI pin sliding against smooth aluminium surface  $(P = 28 \text{ N cm}^{-2}$ ,  $V = 2.08 \text{ m sec}^{-1}$ ,  $R_a$  of counterface  $0.06 \,\mu m$ ).

the bottom may be due to abrasion of a particle entrapped while sliding (Fig. 15a). In Fig. 15b the ploughing action is severe due to high contact pressure. This is indicated by thick furrows in the direction of sliding. Pin failure due to high PV factor is seen in Fig. 16. High frictional heat while sliding against smooth aluminium counterface, means that the pin surface was chiseled in places. It seems that severe melt flow of the polymer has occurred which is evident in the upper part. The whole surface seems highly plastically deformed and interspersed with many ploughed furrows parallel to the sliding direction. At the edges of the groove, severe plastic deformation with brittle fracture of the polymer material due to abrasive cutting by aluminium particles is visible.

## **6. Conclusions**

Tribological studies done on the neat polyetherimide (Ultem 1000) revealed the fact that it had a comparatively lower frictional coefficient than other polyimides which decreased with increase in load. Although it is a ductile polymer, it did not wear by the transfer mechanism. It has very good resistance to fatigue wear. However, once the catastrophic wear started, its specific wear rate  $(K_0)$  was high  $(10^{-13} \text{ m}^3)$  $N^{-1}m^{-1}$ ). Fatigue wear seemed to be the dominant wear mechanism.

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