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A low friction and ultra low wear rate PEEK/PTFE composite

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Abstract

This paper presents a PEEK filled PTFE composite that exhibits low friction and ultra-low wear. It is hypothesized that a synergistic effect shuts down the dominant wear mechanism of each constituent. The friction coefficient and wear rate of this composite material on lapped stainless steel were evaluated for samples with PEEK wt.% of 0, 5, 10, 20, 30, 40, 50, 70 and 100 using a linear reciprocating tribometer. Tests were performed in filtered, standard laboratory conditions with a nominal contact pressure of 6.35 MPa, a speed of 50 mm/s and total sliding distances ranging from 0.5 km for the unfilled PTFE to 140 km for a 20 wt.% PEEK filled sample. The friction coefficients, averaged over an entire test, ranged from $\bar{\mu} = 0.111$ for a 50 wt.% composite to $\bar{\mu} = 0.363$ for unfilled PEEK. Wear rates ranged from $K = 2.3 \times 10^{-9}$ mm^3/(Nm) for a 20 wt.% PEEK sample to $K = 6 \times 10^{-4}$ mm^3/(Nm) for unfilled PTFE. Scanning electron microscopy revealed a unique interfacial connection between the PTFE and PEEK that is likely responsible for the ultra low wear rates observed in these experiments.

Keywords: Solid lubricants; Nanocomposites; Polymer; Composites; Low friction; Low wear; Friction; Wear; Space; Bearing

1. Introduction

Solid lubrication is an attractive option for the design engineer for a variety of reasons, including, simplicity, cleanliness, low expense, quiet operation, low maintenance, low outgassing and high temperature capability. Often, a design necessitates a low wear solid lubricant either for long life or to maintain proper kinematics. Very few materials, however, exhibit low friction and low wear, so the designer is forced to add cost and complexity to accommodate one or the other. There are significant efforts dedicated to the research and development of such solid lubricants; polymers and polymer composites are commonly used as solid lubricants (the state-of-the-art was reviewed by Zhang [1] and Friedrich [2]). Though not exhaustive, Fig. 1 contains plots of wear rate versus filler wt.% for many different polymer composites. The work reported here explores a particular type of polymer composite that has two constituents: polyetheretherketone (PEEK) and polytetrafluoroethylene (PTFE).

In its neat form, PEEK has high wear resistance, strength, operational temperature, friction coefficient ($\mu \geq 0.4$ in dry sliding), and low thermal conductivity. PTFE is a widely used and well-known solid lubricant that suffers from a high wear rate. Both PEEK and PTFE are very common matrix materials for solid lubricant composites, and there are a number of publications that include composites of PTFE and PEEK for tribological applications [3–9]. In the main, fillers are added to PEEK to reduce the friction coefficient and they are added to PTFE to increase the wear resistance.

PEEK is a high performance injection moldable polymer that finds applications in many components that require high strength and operating temperature. Like PTFE, PEEK is also resistant to many aggressive solvents and has a very low vapor pressure, making it suitable for vacuum applications. There are a number of commercially available PTFE-filled PEEK components marketed under a variety of trade names. Recently, Bijwe et al. [9] systematically studied the influence of PTFE inclusion in PEEK to determine an optimum loading of PTFE. The PTFE and PEEK were blended using a twin-screw melt mixer. In order to maintain the ability to injection mold parts, the PTFE volume fractions are limited; in this case between 0 and 30 wt.%. The results showed a monotonic decrease in friction coefficient, wear rate, and most mechanical properties with increasing PTFE content.

Another approach to forming composites of PEEK and PTFE is to follow the processing techniques of molding PTFE, which typically uses cold pressing and consolidation followed by sintering. Briscoe et al. [8] made samples of PEEK-filled PTFE

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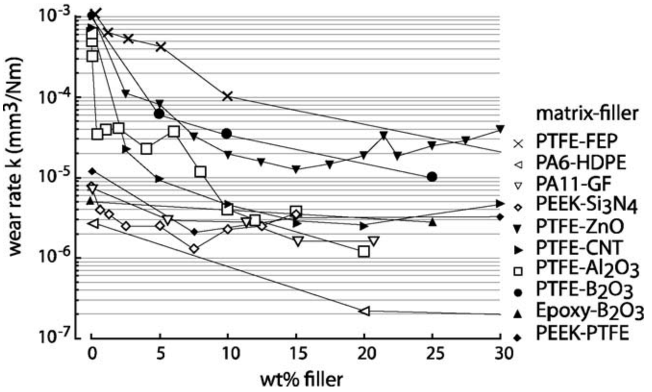

Fig. 1. Results from wear tests for representative polymer matrix composites found in the literature. Wear rate is plotted vs. filler wt.%: PTFE–FEP [16], PA6–HDPE [17], PA11–GF [18], PEEK–Si_3N_4 [19], PTFE–ZnO [20], PTFE–CNT [21], PTFE–Al_2O_3 [21], PTFE–B_2O_3 [22], Epoxy–B_2O_3 [22], and PEEK–PTFE [2].

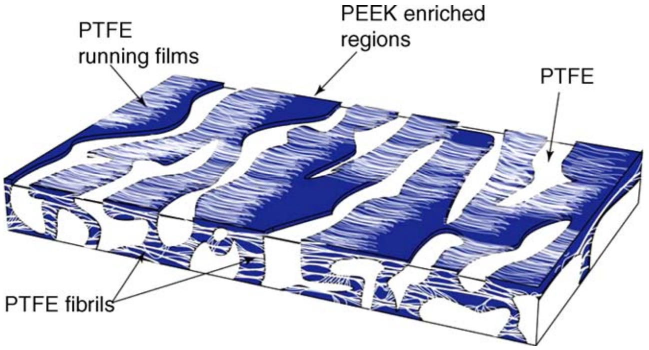

Fig. 3. An illustration of the targeted composite structure, where the major phase (PTFE) exists as neat regions interconnected via PTFE fibrils. The PEEK-enriched material encasing the neat regions of PTFE may also be interconnected. The PTFE running films thought to be responsible for lubrication are shown originating from neat PTFE as noted and illustrated.

from 0 to 100 wt.% PEEK; the powders were blended using a blade mixer, compressed, and sintered. The results showed a monotonic increase in wear rate with PTFE content and monotonic decrease in friction coefficient.

The differences between the study by Bijwe et al. [9] and Briscoe et al. [8] are likely related to materials, processing, and microstructure. In this study, we will process the composites following procedures that were developed for creating nanocomposites of PTFE and alumina. This process used jet-milling to break apart agglomerations, and the compression molding process involved sintering under hydrostatic pressure [10].

2. Materials and composite processing

The PTFE and PEEK are acquired in powder form. The powders are mechanically ground and sifted prior to blending and jet-milling. The PTFE powder used in this study is 7C Teflon powder from Dupont with agglomerated sizes between 100 and 1000 μm. The PEEK powder used in this study is a custom ground Victrex 450PF with a smaller agglomerated size prior to milling. Scanning electron images of the constituent powders are shown in Fig. 2; on average, the PTFE particles are 25 μm in diameter and the PEEK particles are 5 μm in diameter. Composite samples of 0, 5, 10, 20, 30, 40, 50, and 70 wt.% PEEK were created following the same approach as reported in [10]. The targeted structure is shown schematically in Fig. 3, and consists of PEEK enriched reinforcing material encasing regions of unfilled PTFE. Thin running films of PTFE are thought to develop during sliding to provide lubrication.

The milled powder ensembles are compacted with 15 MPa of pressure for 10 s. The pressure is lowered to 2 MPa and a temperature ramp of 120 °C/h is implemented until the hold temperature of 360 °C is reached. After 3 h at temperature, the sample is cooled at 120 °C/h. The mold produces 19 mm diameter × 25 mm long cylinders. Samples measuring 6.3 mm × 6.3 mm × 12.7 mm long are machined out of the interior of the compression molded cylinders using a numerically controlled milling machine. The samples are weighed on a precision analytical balance with a range of 220 g and a resolution of 10 μg; the bulk densities of the samples are calculated using these measurements.

The counterfaces are plates of AISI 304 stainless steel measuring 38 mm × 25.4 mm × 3.4 mm. This material has a measured Rockwell B hardness of 87.3 kg/mm^2. The surfaces are prepared using a standard lapping finish and examined with a

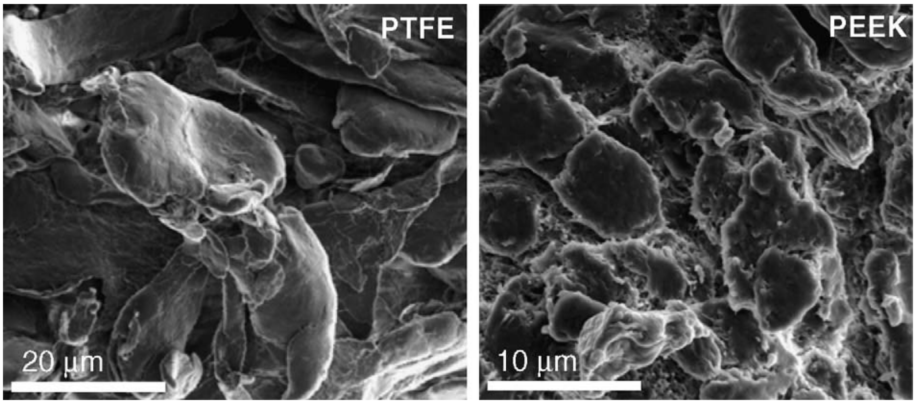

Fig. 2. Scanning electron micrographs of the constituent powders before jet-milling. Both materials are agglomerated prior to blending, and the blend material is compression molded to make this PTFE–PEEK composite. The PTFE and PEEK agglomerations have characteristic sizes ranging from 100 to 1000 and 10–40 μm, respectively. The disbanded PTFE and PEEK particles have characteristic sizes ranging from 5 to 25 and 2–10 μm, respectively.

scanning white light interferometer. Areas of 23 μm × 300 μm were measured for five regions of five counterfaces. The average and standard deviation of the root-mean-squared roughness calculations are $R_q = 161$ nm and $\sigma = 35$ nm, respectively.

3. Experimental procedure

A laboratory designed linear reciprocating tribometer was used to test the wear and friction of the samples; this testing apparatus and the uncertainties associated with the experimental measurements are described in detail in Refs. [11–13]. Although open to the air, the entire apparatus is located inside a soft-walled cleanroom with conditioned laboratory air of relative humidity between 25 and 50%.

The composite samples are mounted directly to a six-channel load cell and the counterface is mounted to a linear reciprocating stage. Prior to testing, the counterfaces are washed in soap and water, cleaned with acetone, sonicated for ∼15 min in methanol, and dried with a laboratory wipe. After dry machining, the composite samples are wiped down with methanol.

A normal force of 250 N is applied via a pneumatic cylinder/linear thruster assembly, and results in a nominal contact pressure of 6.25 MPa over the 40 mm^2 pin area. The load is continuously monitored and computer controlled using an electro-pneumatic valve. The reciprocating length is 25.4 mm with an average sliding speed of 50.8 mm/s. Instantaneous data is collected for normal load, friction force, table position and pin position at 1000 Hz over one cycle at specified intervals. The instantaneous data are averaged over two cycles and saved at specified intervals that depend on the length of the test.

Interrupted mass measurements are used to quantify wear rather than dimensional measurements of pin height because of sample creep and thermal expansion. These wear measurements are made periodically during each test. Steady state wear rates and uncertainties are reported in this study following techniques discussed in [13]. The uncertainty intervals on wear rate data represent the experimental uncertainty in the measurement while the confidence intervals on friction coefficient data represent the standard deviation of the friction coefficient for the entire test [14].

The volume loss of a preliminary 20 wt.% sample had a corresponding wear depth of 6 μm across the sliding surface after 100 km at 6.35 MPa of contact pressure. It is unclear what the statistical fluctuations in composition are over such a small volume of the composite (i.e. to what degree can the bulk composition be used to estimate the very near surface composition of these samples that experience ultra low wear). In order to estimate the magnitudes of the variation in composition as a function of volume, a diamond saw was used to cut several samples into parallel slices of varying thickness (the smallest sample made using this technique was approximately 10 mm^3). Here a batch is defined as a group of slices of equal volume cut from a single sample. The ratio of the standard deviation of the measured densities (σ_ρ) to the density of the composite (ρ_c) followed a power law relationship with the volume of the slice ($\mathcal{V}$) measured in cubic millimeters; this is given in Eq. (1). The equation for wt.% based on density measurements is given in Eq. (2).

$$\frac{\sigma_\rho}{\rho_c} = \left(\frac{0.1x10^{-3}}{\mathcal{V}} \frac{mm^3}{mm^3} \right)^{0.4} \tag{1}$$

$$\text{wt.\%}_{\text{PEEK}} = \frac{\rho_{\text{PEEK}}}{\rho_{\text{PTFE}} - \rho_{\text{PEEK}}} \left(\frac{\rho_{\text{PTFE}}}{\rho_{\text{c}}} - 1 \right) \times 100 \tag{2}$$

The confidence intervals on filler wt.% are calculated using the measured mass change in the sample after testing and the initial density of the composite, and represent one standard deviation of the expected filler wt.% of these worn surface regions. Although the most wear resistant composites are run the longest to maximize wear volume, they often have the greatest uncertainty in their near surface composition with many samples losing less than a cubic millimeter of material.

4. Results

The friction coefficients of the composites are shown in Fig. 4 as functions of the sliding distance and position along the wear track. The composites all had little fluctuations in friction coefficient along the wear track. The smooth shape along the wear track is characteristic to PTFE systems. Unfilled PTFE and PEEK have average friction coefficients of $\bar{\mu} = 0.135$ and $\bar{\mu} = 0.363$, respectively. The 20 wt.% composite has steady frictional behavior with an average friction coefficient of $\bar{\mu} = 0.12$. The composites containing less than 20 wt.% PEEK had initial friction coefficients equal to unfilled PTFE that gradually approached the friction coefficient of the 20 wt.% composite; the 70 wt.% composite behaved in a similar fashion. The composites with more PEEK than 20 wt.% (except the 70 wt.% composite)

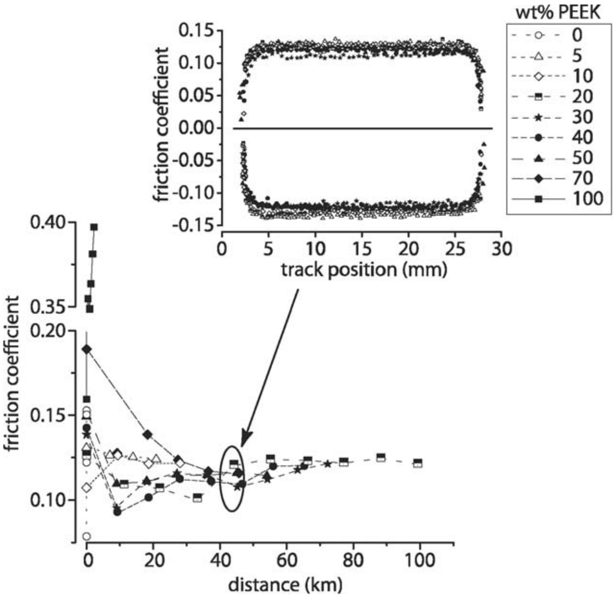

Fig. 4. Coefficients of friction vs. sliding distance for a number of composite samples varying from unfilled PTFE to unfilled PEEK. Filler wt.% reflects the prepared composition of the powder blends. Coefficient of friction vs. track position is also plotted for the composites after sliding for approximately 40 km. The 5 and 10 wt.% data was taken near the ends of the tests. Sample surfaces are 6.35 mm × 6.35 mm under 250 N of normal load and the reciprocation length is 25.4 mm at a speed of 50.8 mm/s.

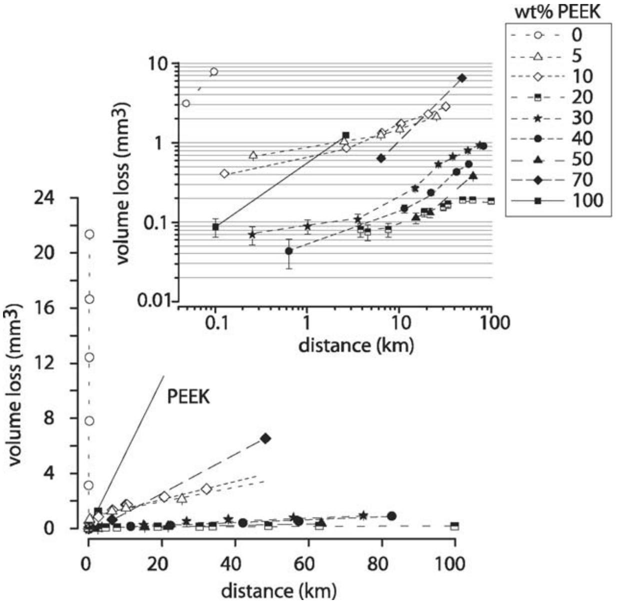

Fig. 5. Volume loss vs. sliding distance for a number of composite samples varying from unfilled PTFE to unfilled PEEK. A log–log plot is inset for easier visualization of the many orders of magnitude variation in wear rates. Filler wt.% reflects the prepared composition of the powder blends. Volume loss is calculated from mass measurements and the calculated density. The uncertainty intervals on volume loss are noted and calculated from [13]. The sample surfaces are 6.35 mm × 6.35 mm under 250 N of normal load and the reciprocation length is 25.4 mm at a speed of 50.8 mm/s.

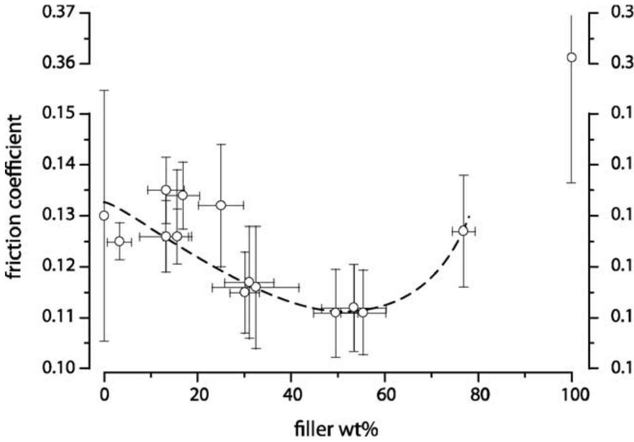

Fig. 6. Friction coefficient for the composites plotted as a function of PEEK filler wt.% as calculated using the measured sample density. The vertical confidence intervals represent the standard deviation of the friction coefficient over the entire test, while the horizontal confidence intervals are calculated following Eqs. (1) and (2). Sample surfaces are 6.35 mm × 6.35 mm under 250 N of normal load and reciprocation length is 25.4 mm at a speed of 50.8 mm/s (the total sliding distance varied).

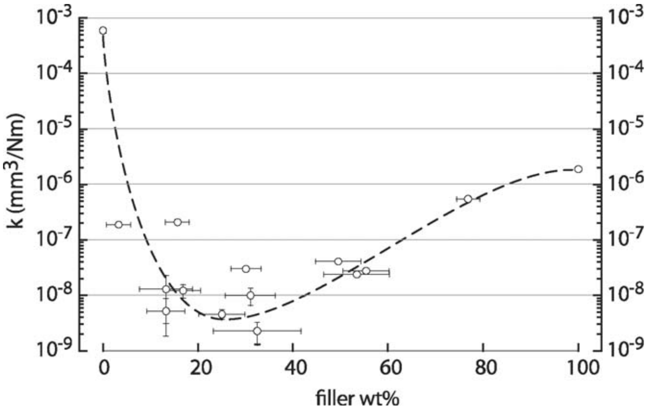

Fig. 7. Wear rates calculated from interrupted mass measurements for the composites plotted as a function of PEEK filler wt.% as calculated using the measured sample density. The uncertainty intervals on wear rate are calculated according to [13], while the horizontal confidence intervals are calculated following Eqs. (1) and (2). Sample surfaces are 6.35 mm × 6.35 mm under 250 N of normal load and reciprocation length is 25.4 mm at a speed of 50.8 mm/s (the total sliding distance varied).

had low initial friction coefficients that increased to a steady state friction coefficient equal to that of the 20 wt.% composite.

In Fig. 5 the volume loss of each sample is plotted versus the sliding distance. A log–log plot has been included for clearer visualization of the data. The unfilled PTFE and PEEK had steady state wear rates of $K = 6.0 \times 10^{-4}$ and $K = 1.9 \times 10^{-6}$ mm^3/(Nm), respectively. The composite samples were all 10×–1000× more wear resistant than the unfilled PEEK sample. The samples with less than 20 wt.% PEEK have initial transient regions of high wear followed by a gradual decrease in wear that approaches steady state. The 20 wt.% sample performed best, having a small transient period that was followed by a steady state during which no change in mass was detected for the last one million sliding cycles (the scale has a resolution of 10 μg). The samples having more than 20 wt.% PEEK wore nearly linearly from the onset of sliding; the wear rates generally increase with increasing PEEK content.

Fig. 6 shows the time-averaged friction coefficients for all of the tested samples plotted versus filler wt.% as calculated from density measurements. The lowest friction coefficient is $\bar{\mu} = 0.111$ with 50 wt.% PEEK. The friction coefficients tend toward that of unfilled PTFE as the wt.% of PEEK goes below 50 wt.%. Above 50 wt.% PEEK, the friction coefficient increases, but remains less than that of unfilled PTFE at 70 filler wt.%.

Fig. 7 shows the steady state wear rate plotted versus filler wt.% as calculated by density measurements. At PEEK concentrations greater than 30 wt.%, the wear rates increase monotonically with PEEK content. At PEEK wt.% less than 30, the wear rates approach the wear rate of unfilled PTFE as filler content is reduced.

5. Discussion

Nearly all of the composites had lower average friction coefficients than unfilled PTFE, which is in contrast to what might be expected from a linear rule of mixtures approach. The mechanism for this reduction in friction coefficient is believed to originate from thin running films of PTFE that are drawn out over the PEEK enriched regions. These running films then slide against a transfer film that develops on the surface of the stainless steel counterface.

The friction coefficient of the composite material was lowest at approximately 50 wt.% PEEK. As the PEEK content is increased from unfilled PTFE, friction is lowered because the shearing of low strength running films is over an increasingly stiff material with less real area in contact. At some filler wt.% the spacing of the PTFE reservoirs and size of PEEK reinforced regions become such that the running films cannot completely lubricate the PEEK. Adhesion and fracture can liberate pieces of PEEK, which can abrade the composite, further increasing the frictional forces.

Like friction, wear resistance also fails to follow any rule of mixture explanation. The best performing composite sample (20 wt.% prepared, 32 wt.% PEEK calculated with density) is 260,000 times more wear resistant than unfilled PTFE and 900 times more wear resistant than unfilled PEEK. It wore a total of 0.24 mm^3 under 250 N of load after the first 50 km of dry sliding and then reached a steady state where no change in mass was detected for the last 50 km. This corresponds to the dissipation of 1.5 MJ of frictional energy over a 40 mm^2 surface without detectable (to 10 μg resolution) material removal. The wear resistance mechanism is unclear; it was hypothesized that a co-continuous network structure of PTFE and PEEK would prevent delamination in PTFE and the reduced friction coefficient would reduce the fracture induced wear of PEEK. One of the unique aspects of this composite is that the PEEK particles are small compared to the PTFE particles, potentially facilitating a network structure similar to that described in the nanoparticle filled research with PTFE and alumina [10,15].

At filler loadings less than 32 wt.% the transient region of wear is longer and more pronounced as the filler content approaches zero. There are a number of possible explanations for this behavior: (1) there is insufficient PEEK to create a network, (2) the increased area of PTFE and increased spacing of PEEK prevents the interruption of damage to the PTFE, and (3) the increased real area of contact causes more PTFE to be engaged at the interface. The low wt.% samples' wear rates slowly approached those of the more wear resistant samples; this suggests that some surface accumulation of PEEK had occurred.

To investigate this effect further, five identically prepared 15 wt.% PEEK composite samples were tested under the standard conditions for one of five numbers of sliding cycles: 2×10^4, 1×10^5, 5×10^5, 1×10^6 and 3×10^6. The instantaneous wear rate and friction coefficient are plotted as functions of reciprocation cycles in Fig. 8 for this series of tests. The wear rate approaches those of the optimum composition as the number of reciprocation cycles builds.

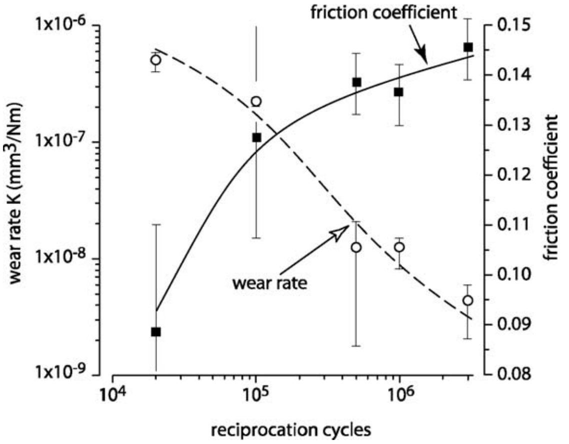

Fig. 8. Instantaneous wear rate and friction coefficient plotted as functions of reciprocation cycles. The uncertainty intervals on wear rate are calculated according to [13] for the last two mass measurements in each test, and the confidence intervals on friction coefficient represent the standard deviation of friction measurements made throughout the test as described in [14]. Sample surfaces are 6.35 mm square under 250 N of normal load and reciprocation length is 25.4 mm at a speed of 50.8 mm/s.

At PEEK concentrations above 32 wt.% the wear rate increased monotonically with 30 PEEK content. Qualitatively, optical microscopy of these samples revealed a smooth surface with increasing sizes and amounts of fractured regions in the composite with increasing wear rates and wt.% above 32 filler wt.%. It appears that at high PEEK concentrations, the increased number of large regions of PEEK leads to a fatigue/fracture wear mechanism. This behavior is also consistent with the idea that as PEEK content increases, the size of the PEEK domains and the spacing of the PTFE reservoirs increase, and may prohibit the full lubrication of the PEEK. As these regions of PEEK are fractured, third body abrasion can lead to damage of the transfer films and plowing of the composite.

The use of small PEEK particles and fibrillation of PTFE during jet-milling allows a specific microstructure to form as the PEEK particles coalesce around fibrils during melt. Fig. 9 shows digital enhancement of an optical micrograph of a 20 wt.% PEEK composite sample after 20 h at the maximum processing

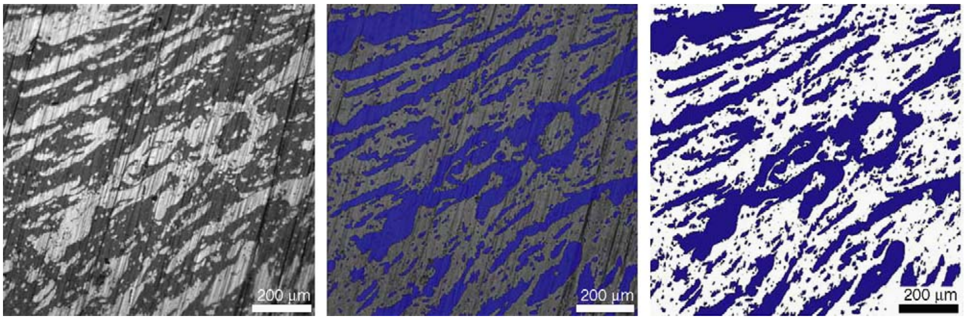

Fig. 9. Optical micrograph illustrating the microstructure of a 20 wt.% PEEK filled PTFE composite after processing at 360 °C for 30 h. The sample was sanded flat and polished with 2000 grit silicon carbide wet abrasive paper prior to imaging. Left: original image with PEEK as the light colored phase; center: PEEK is highlighted in blue; right: The background PTFE is removed for clarity. There is a clearly networked region of PEEK containing regions of PTFE in the center of the image. For interpretation of the references to colour in this figure legend, the reader is referred to the web version of the article.

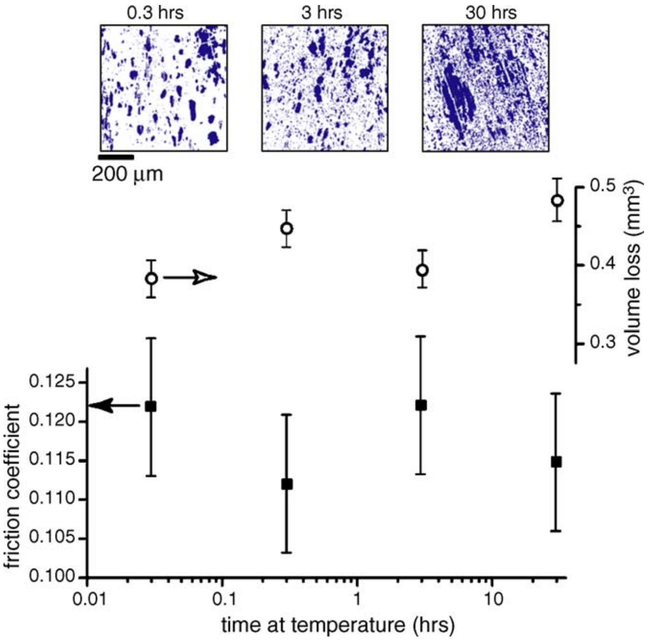

Fig. 10. Friction coefficient and wear volume at the completion of 1.25 million cycles (the total sliding distance was 63.5 km and the applied load was 250 N) plotted vs. the maximum temperature hold time (h) during compression molding. Optical micrographs with computational identification of the visible PEEK microstructure (following Fig. 9) after sectioning are shown above the graph. There is a clear increase both the size and number density of visible PEEK domains (all composites have the same 20 wt.% of PEEK); the wear rate and friction coefficient appear independent of time at temperature.

temperature of 360 °C. The sample was sectioned with a diamond saw, wet abraded with 600 grit silicon carbide paper, and polished on a lapping wheel with 2000 grit silicon carbide paper. Continuous regions of PEEK can be seen spanning up to 1 mm in length. It is clear that many 5 μm particles must have coalesced during melt to form such a complex microstructure. The effect of this time dependent coalescence on the tribological properties was studied by processing four identically prepared 20 wt.% powders with times at melt of 0.03, 0.3, 3 and 30 h. These samples were tested under the standard conditions for 1,250,000 cycles of sliding. Fig. 10 shows the worn volume and average friction coefficient plotted as functions of the time at melt during processing at the completion of 1,250,000 wear cycles. These results show little dependence of friction and wear to time at melt during processing. These samples were sectioned with a diamond saw, wet abraded with 600 grit silicon carbide paper, and polished on a lapping wheel with 2000 grit silicon carbide paper for optical microscopy observation of the microstructure. The results of these observations are shown in Fig. 10; the PEEK was computationally identified and highlighted in blue. Despite the lack of difference in tribological properties of these samples, optical microscopy did show some development of the microstructure with increasing time at temperature. As the time at process temperature increases, both the size and number density of the visible domains of PEEK increase.

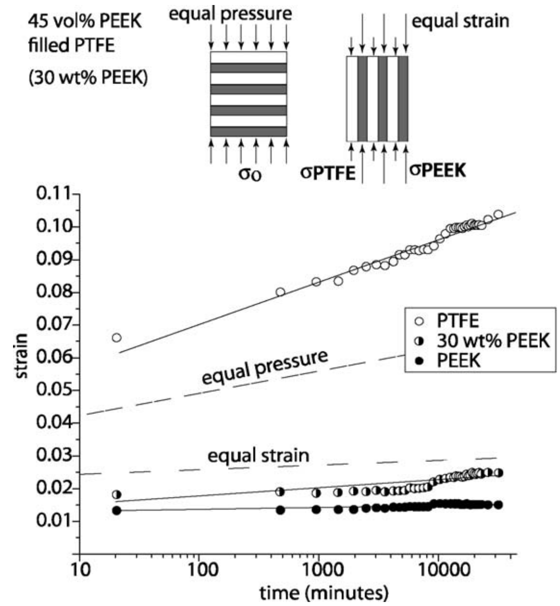

Fig. 11. Results of uniaxial compression tests of neat PEEK and PTFE as controls, and a 30 wt.% (45 vol.%) PEEK/PTFE composite. The responses of equal strain and equal pressure 45 vol.% composites were calculated based on the measured neat material responses, and are plotted with the data for comparison.

Several groups have published work with PTFE–PEEK composites, all finding statistically significant tribological improvements over the neat materials. However, the 10×–1000× improvement in the wear resistance of this particular form of the composite indicates that there is some fundamental microstructural difference in these composites (as discussed previously modifications of the gross networked structure showed no effect on the tribological behavior). This difference was believed to be a unique microstructure that results from the combined effects of small particles of PEEK and the jet-mill processing, but characterization efforts for this PEEK and PTFE composite with various 2-D microscopy and etching techniques were unsuccessful.

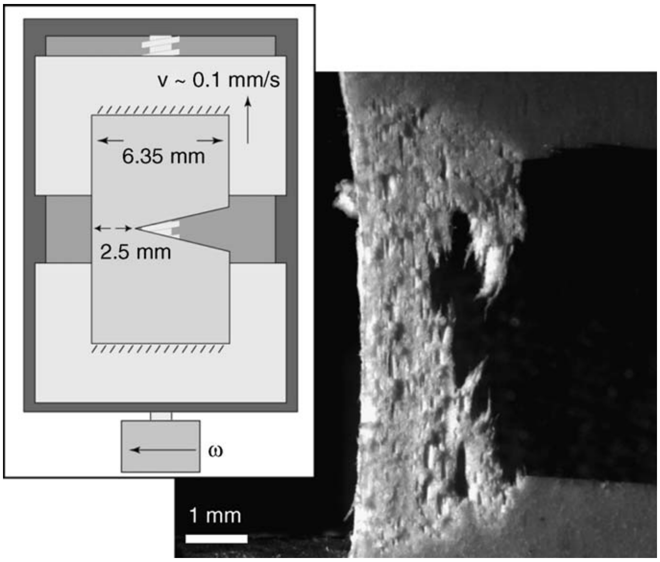

Fig. 12. Experimental schematic and resulting optical image of a notched fracture experiment for a 30 wt.% specimen under quasi-static extension at room temperature. The stress raiser does not induce a fracture response; instead, the material fibrillates in the direction of the stress and elongates an estimated 250–500% prior to breaking.

In an attempt to gain insight into the microstructure by other means, a series of mechanical tests were conducted. The first of these was a creep test performed on neat PTFE, neat PEEK and a 30 wt.% composite. Tests were performed at room temperature in a dry N_2 environment with a uniaxial compressive stress of 10 MPa. The displacement was measured as a function of time. Two limiting composite structures, shown schematically in Fig. 11, are used to confine the expected response of the composite. The first structure has a predominant orientation perpendicular to the load. Because each constituent in the composite supports the same load, this model is referred to as the equal pressure model, and the mechanical response follows a simple linear rule of mixtures. Many structural composites have reinforcing fibers aligned with the load for superior strength and stiffness. Because the elongation of filler and matrix are equal, the composite behaves more like the reinforcing phase, and will be referred to as the equal strain model.

The results of this creep testing are shown in Fig. 11. It is clear that the mechanical response of the composite did not follow a linear rule of mixtures. The effective elastic modulus of the composite is approximately equal to that of unfilled PEEK with a creep rate that was approximately 5% greater than for the equal strain composite. One would not expect equal strain behavior in a composite without intentional orientation, unless a continuous network of PEEK was present, or the mechanical properties of the matrix were somehow changed by incorporation of the filler.

Other interesting mechanical properties to study from a tribological perspective are fracture toughness and strain to failure. These properties may correlate with wear resistance because they determine the ability of a material to absorb energy without liberating material from the bulk. To interrogate these properties, quasi-static extension of a notched 30 wt.% specimen was performed and compared to unfilled PTFE and a commercially available reinforced PEEK composite that exhibited excellent wear resistance ($k \sim 1 \times 10^{-7}$ mm^3/Nm). The samples were cut with a razor blade to create a large stress concentration, mounted on a linear stage under a microscope, and quasi-statically extended until fracture. A schematic of the test and an optical micrograph of the failed composite sample (the PEEK and PTFE controls are not shown for brevity) are shown in Fig. 12. The composite sample was extended out of the field of view of the microscope without fracture. A rough estimate of strain to failure can be obtained by using the length of the discolored envelope of strained material as the effective original length of an equivalent tensile specimen located at the border of failed material. Using this approach an approximate strain at failure of 250–500% is obtained. The high resistance to fracture of this sample is in stark contrast to the results of identical tests with the neat PTFE and PEEK composite samples. The PEEK composite is strong but fractures easily with the presence of a defect. The PTFE gives little resistance to the opening and blunting of the crack, and fractures after incurring a large (but much less than the composite) amount of deformation. The compression creep test illustrates the stiffness of the composite, while the fracture experiment depicts the resistance of the composites to material removal.

The highly strained material from Fig. 12 was next investigated with energy dispersive spectroscopic fluorine dot mapping and scanning electron microscopy (SEM) with the goals of identifying the fibrillating phase and characterizing the expanded 3-D microstructure. The sample was removed from the extension fixture and carbon coated before SEM observation. Fig. 13 shows an area originally near the crack tip, in a portion of material that did not fail. The fluorine map shows that the fibrillating phase is PTFE, while the dark particle phase is PEEK. It is interesting to note that the majority of the PEEK particles in the composite are on the order of 100 μm while the PEEK powder has an average particle size of about 5 μm. This further suggests that coalescence of the PEEK is occurring during melt.

Fig. 14 shows a sequence of images at increasing magnification at a region bordering the failed material on the fracture sample. The 3-D nature of this sample gives a unique perspective into the microstructure that was not available on the two dimensional surfaces obtained by standard sectioning techniques. These images reveal a microstructure that is not networked in the originally hypothesized sense; particles of PEEK appear to be imbedded within a PTFE matrix. As a 'discrete' particle is examined under higher magnification, the nano-scale networking of the PEEK becomes evident. The PEEK particle has many holes and irregularities, and appears as though it

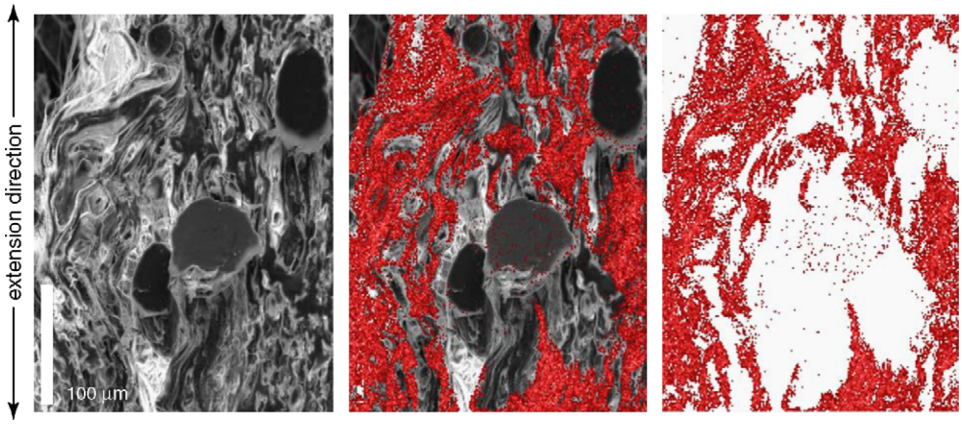

Fig. 13. Energy dispersive spectroscopic fluorine dot mapping of the expanded specimen after extension and removal from the test fixture. From left to right: SEM image of the mapped area; the fluorine dot map overlaying the image; the fluorine dot map. The PTFE is clearly identifiable as the fibrillating phase.

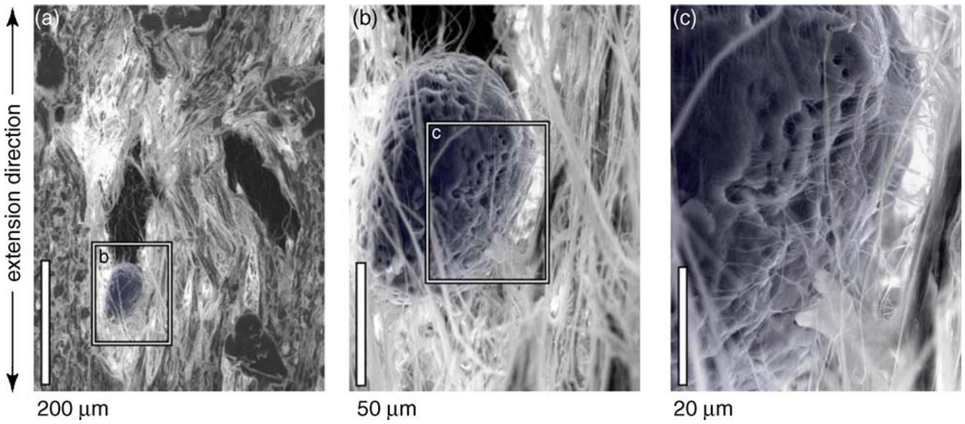

Fig. 14. Scanning electron images of the 30 wt.% fracture sample. This sample was extended, removed from the test fixture and carbon coated. From left to right: low magnification image revealing an undamaged PEEK particle; increased magnification image of the particle showing highly fibrillated microscale PTFE encapsulating the particle; high magnification image showing a networked structure of nanoscale PTFE fibrils trapped within the coalesced PEEK particle.

coalesced around a fibrillated PTFE structure during melt. Fibrils of PTFE are thickest away from the particle and become thinner as they approach the PEEK. In the highest magnification image, fibrils of PTFE with diameters of around 100 nm can be observed connecting the PEEK to the PTFE. The fibrils seem to enter the PEEK through the holes and irregularities as if trapped during the coalescence process.

The large amount of energy used in jet-milling is thought to fibrillate the PTFE. Upon melt, the PTFE is much more viscous than the PEEK, giving more resistance to motion. The PEEK particles are forced to coalesce around the PTFE fibrils setting up the observed nanoscale networking of the PEEK and PTFE. This interfacial connection is significant since PTFE and PEEK have negligible interfacial shear strength; without mechanical interlocking of both phases, PEEK particles can simply fall out of the matrix. This is likely the source of the exceptional wear resistance observed in wear tests of this composite.

6. Conclusions

1. This composite material has a friction coefficient lower than unfilled PTFE and PEEK for every sample tested. The lowest average friction coefficient of $\bar{\mu} = 0.111$ was obtained for three samples having a PEEK wt.% of 50. The friction coefficient of PTFE was found to be $\bar{\mu} = 0.132$.
2. The composite has a wear rate lower than unfilled PTFE and PEEK for every sample tested. The lowest wear rate of $K = 2 \times 10^{-9}$ mm^3/(Nm) was obtained for a 32 wt.% PEEK filled sample. This sample was 900 times as wear resistant as the unfilled PEEK and 260,000 times as wear resistant as the unfilled PTFE.
3. Samples having a PEEK content greater than 32 wt.% had no wear transients. The wear rates were observed to increase with increasing PEEK content approaching that of unfilled PEEK.
4. Both PEEK networking and nanoscale penetration of PTFE through PEEK particles were found in this composite system.

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