

Effects of contact pressure, molecular weight, and supplier on the wear behavior and transfer film of polyetheretherketone (PEEK)

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Abstract

Polyetheretherketone (PEEK) is a designation given to materials of the polyaryletherketone family having a characteristic distribution of ether and ketone groups in the polymer backbone. PEEK materials have high strength and chemical resistance as well as very high melting points and glass transition temperatures. Because of this combination of properties, PEEK materials find use for wear application in extreme environments where they provide a light-weight and corrosion resistant bearing material that often does not require lubrication. This study focused on determining the effects of supplier and molecular weight on the wear of particular PEEK materials, in addition to the effect of contact pressure. Multidirectional wear testing was performed on four PEEK materials. The materials were obtained from two different suppliers, and two molecular weights were chosen for each supplier. Extensive analysis of transfer films produced during wear testing was performed using optical microscopy. White light profilometry was used to measure transfer film thickness in order to calculate a mean film thickness for given experimental conditions. Dynamic mechanical analysis, as well as gel permeation chromatography and differential scanning calorimetry were used to characterize each material's viscoelastic behavior, molecular weight, and crystallinity, respectively. It was found that the wear of PEEK materials was significantly affected by both contact pressure and molecular weight, but not by supplier. However, an interaction was observed that showed the low molecular weight material from one of the suppliers was more vulnerable to wear at high contact pressures than the other three materials. Results of transfer film analysis showed that film thickness was greatest in locations where pin sliding direction was perpendicular to the counterface roughness direction, but that mean transfer film thickness did not correlate to wear amounts. This work is significant because it highlights the fact that tribologically relevant polymers, such as PEEK materials, vary greatly in terms of their polymer morphology and processing history, and this variation must be recognized by investigators when reporting wear data.

Keywords: tribology, wear, polymers, transfer film, polyetheretherketone

1. Introduction

Polyetheretherketone (PEEK) is type of aromatic-backbone polymer belonging to the polyaryletherketone (PAEK) family of thermoplastics. PEEK materials demonstrate high strength, stiffness, and toughness along with excellent chemical resistance in many applications. These materials find widespread use in high temperature applications because of their very high glass transition temperatures, often in excess of 140°C [1]. Crystallinity, which has a significant impact on PEEK's mechanical properties and chemical resistance, is typically from approximately 30 to over 40 percent, based on polymer morphology and processing methods [2]. There are many variants within the family of PAEK materials, and they are named according to the presence of ether and ketone units in the backbone. Polyetherketone (PEK) and polyetherketoneketone (PEKK) are examples. Though often overlooked, there are wide variations in the molecular morphology among polymers classified as PEEKs, because of the varied approaches to polymerization chemistry and processing. Various grades of PEEK in their neat form have been shown to have good tribological and wear properties, possibly due to high strength, modulus, and toughness.

Polymers such as PEEK, polyphenylenesulfide (PPS), polytetrafluorethylene (PTFE), and ultra high molecular weight polyethylene (UHMWPE), find use in extreme environments where the use of traditional metal bearing materials and fluid lubricants are impractical [3-5]. These polymers typically exhibit an acceptably low coefficient of friction (COF), and they often deposit a transfer film on a mating metallic counterface. It is hypothesized that the film protects the bulk polymer from direct abrasive wear by the harder counterface. The mechanical, thermal, and chemical causes for this film deposition in PEEK and its effect on wear have been studied by several researchers. Briscoe et al. [6] conceptualize the wear polymer surface during sliding to be two separate regions. An interface region with a depth of 100 nm has been proposed that is responsible for adhesive friction forces and a cohesive region that dissipates the frictional work through viscoelastic response. It is thought that the mechanical and thermal interaction of these two regions plays a primary role in wear debris generation and film formation. In thermoplastic polymers, such as PEEKs, the frictional heating that arises at the interface is enough to cause a significant drop in the storage modulus. Analysis of PEEK wear debris by Zhang et al. has shown a drop in crystallinity and interfacial temperatures above melting that may promote plastic flow [7]. Studies of amorphous PEEK films, however, have shown that temperatures above the crystallization temperature are reached and the storage modulus may increase from friction induced crystallization [8]. When the counterface has sufficient surface roughness, material can be removed from the polymer bulk through cutting and abrasion by the asperities. Counterface roughness is likely one of the dominant wear factors in both filled and unfilled polymers and has been studied by Blanchet et al. [9], among others. Hollander et al. [10] developed an average asperity radius model to explain the relation between roughness and wear. Their proposed model states that transfer film formation is aided by entrapment of wear debris between asperity contacts. There appears to be a transition point in surface roughness above which the transfer film is unable to fully protect the bulk polymer from the abrasive action of the counterface, resulting in increased wear [11]. Additionally, material removal during wear does not necessarily mean that a film will form since there must also be adhesion to the counterface. There is evidence to suggest that film adhesion occurs because of entrapment of particles between asperities, localized melting, and chemical bonding. Polymer wear performance is often discussed in terms of the wear in response to product of pressure and velocity, or PV. Because pressure and velocity can act synergistically, bearing materials are often assigned a PV limit above which loads cannot be sustained due to excessive wear and frictional heating. Zhang and Schlarb showed that at calculated normal pressures above 4 MPa, the cutting wear mechanism vanishes and there is evidence of plastic flow at the wear interface of PEEK [12]. Additionally, excessive velocity will strongly influence heating at the surface and cause debris aggregation that may disrupt film deposition [13, 14]. Though surface roughness and thermal factors strongly affect wear, polymers have been shown to form thick adherent transfer films when these factors are absent. In early work by Jain and Bahadur, the sliding wear between two different polymer surfaces indicated that the cohesive energy density of the two surfaces contributed to material transfer. They found that in sliding friction, material is transferred from the polymer surface of lower cohesive energy density to the one with greater cohesive energy density [15].

Despite numerous studies of polymer wear and transfer film formation, no model currently exists to precisely predict wear a priori. This is due to the complexity of the wear phenomenon and its combination of activity of many parameters. The viscoelastic nature of polymers deems that mechanical properties will change as a function of time and temperature. Couple this fact with the inexact knowledge of the temperature and stress distribution at the wear interface and it indicates that the problem is still not well understood. Furthermore, there is no definitive metric for what constitutes a good transfer film. Historically, PEEK films have been described as thick, uniform, and continuous relative to other polymers. Because of these subjective and qualitative descriptions, it has been challenging to understand the relationship between transfer film characteristics and wear. Very little conclusive work regarding transfer film height as it relates to wear volume has been done.

The purpose of this study was to explore the amount that wear and transfer film formation of various PEEKs varies with respect to different polymer morphologies and sliding conditions. In contrast to much of the earlier work in the area, PEEK is not assumed to be a unified homogeneous material, but rather a family of materials that have differences in molecular weight and dynamic mechanical properties with respect to the polymerization process used by different suppliers. Furthermore, the effect of normal load with respect to polymer morphology was examined. The investigation included wear testing conducted using a novel multi-directional pin-on-plane tribometer. Transfer film height was measured using white light interferometry to determine if a direct relationship exists between wear and transfer film height in PEEK materials. The significance of this work is not only the tribological data, but also the fact that extensive analyses were performed to fully understand the morphology of the various PEEK grades that were used in the work in order to understand how they affected wear.

2. Materials and Methods

2.1 Materials

Four neat PEEK materials from two different suppliers, identified as ‘1’ and ‘2’, were used in this work. The identities of the suppliers are not reported due to confidentiality requirements, however morphological data is reported in order for the reader to evaluate the reported results. All samples were injection molded under controlled and constant conditions and finish machined into pins for wear testing and thin bars for dynamic mechanical analysis. Two different grades of PEEK from each supplier were used based on measured molecular weight. The grades were selected so that the ‘low’ and ‘high’ values of M_w were comparable across the two suppliers. In this paper the high molecular weight grades are referred to as ‘1H’ and ‘2H’ and low molecular weight grades as ‘1L’ and ‘2L’, respectively. Gel permeation chromatography (GPC) was used to characterize the weight average molecular weight (M_w) and polydispersity index (PDI) of each of the four materials and the results are shown in Table 1. Differential scanning calorimetry (DSC) was used to characterize the degree of crystallinity of each of the materials, and the data are also reported in the table.

Table 1. Compilation of morphological (weight average molecular weight, M_w , and polydispersity index, PDI, and crystallinity) and viscoelastic data (storage and loss moduli) for the four PEEK grades used in the study.

Sample	M_w	PDI	% crystallinity	Storage Modulus (GPa)	Loss Modulus (MPa)
1L	76306	2.76	42	3.36	71.5
2L	84709	2.64	36	3.89	121
1H	143645	3.72	35	3.07	60.9
2H	118548	3.52	32	3.72	135

2.2 Wear Testing

Wear pins of the four PEEK materials were molded and machined to a diameter of 6.4 mm and length of 19.1 mm. Before testing, the wear surface of each pin was finished to a surface roughness of approximately 0.2 μm Ra. Samples were ultrasonically cleaned, dried, and weighed before and after each test on a precision balance with a resolution of 0.01mg. For this work, a two-axis tribometer was employed which incorporates two programmable linear stages (Aerotech). The tribometer allows for sliding of pin specimens against planar counterfaces under an applied normal load along a specified wear path and sliding velocity. Typical pin-on-disk methods of wear testing are only capable of subjecting the polymer specimen to unidirectional sliding. It has been shown by Schwartz et al. and others that unidirectional wear testing of polymers often underestimates the wear observed in actual bearing applications by orders of magnitude [16]. It is theorized that polymer chains on the wear surface orient themselves in the direction of sliding and thus exhibit directional strengthening and subsequent increased

wear resistance. Wear pins were loaded against tool steel counterfaces using pneumatically controlled actuators and the tribometer was programmed to slide the pins in a circular path with a diameter of 20 mm for 80,000 cycles (approx. 5km total sliding distance) at a constant sliding velocity of 0.1 m/s. The test duration was chosen to be sufficiently long to attain steady state wear. Testing was conducted using applied loads of 60 N (low) and 125 N (high), which resulted in Hertzian calculated pressures of 1.8 MPa and 3.9 MPa, respectively. Hardened D2 tool steel counterface plates (HRC 56.6 measured) were ground so that roughness was oriented in one direction with an average roughness Ra of 0.5 μm . Since the wear path was circular, the angle between the direction of the pin motion and the counterface roughness direction was constantly changing. The change in mass was recorded and the reported density of the respective PEEK materials was used to calculate volumetric wear. Wear testing followed a full factorial experimental plan with factors including normal load, polymer molecular weight, and polymer supplier. A total of four pins of each material were tested in randomized order to account for statistical variation.

2.3 Transfer Film Measurement

Transfer films have traditionally been assessed from a qualitative standpoint and therefore objective metrics related to the bonding, thickness, and uniformity of films has not been well reported. A goal of this work was to develop a quantitative measurement technique to compare transfer films formed by different PEEK grades. Optical microscopy was used to give information about film uniformity. A white light interferometry based profilometer (Zygo) with analysis software was used to measure the film thickness at locations along the wear path. The circular sliding path was indexed at 45° angular increments and film thickness was measured at these locations. The profilometer produced a topographic map for both the film and underlying counterface for more detailed examination of film continuity where required.

2.4 Dynamic Mechanical Analysis

Dynamic Mechanical Analysis (DMA) allows for the characterization of a material's viscoelastic behavior by measurement of the storage and loss moduli of the material across a range of temperatures and loading frequencies. Thermoplastic polymers undergo transitions from glassy to rubbery at a temperature known as the glass transition temperature, T_g . This transition was found with DMA for the materials by locating a drop in storage modulus and a peak in the trigonometric tangent of the phase lag between the applied stress and material response, referred to as 'tan δ '. Storage modulus indicates the elastic stiffness component of a material when subjected to a load, while the loss modulus is related to the capability of the material to damp the oscillating mechanical energy input into the sample. A simple model of the sliding wear process suggests that wear resistant materials must be able to support applied load without extensive deformation (storage modulus) but also absorb the impact produced from sliding over asperities (loss modulus). DMA (TA Instruments, RSA-G2) was performed to characterize each of the four materials and determine if any relationship existed between the wear performance of different PEEK grades and their viscoelastic properties. Testing was performed in three-point bending mode. Injection molded samples were machined to dimensions of 40 x 11.5 x 3.3 mm in accordance with ASTM D790 [17]. A temperature sweep was performed with a ramp rate of 3°C/minute between 25 and 300°C. During the test, a 0.02% strain was applied with a loading frequency of 1 Hz.

3. Results

3.1 Wear

The results from the multidirectional wear testing of the four PEEK grades are plotted in Figures 1 and 2 for the calculated contact pressures of 1.8 and 3.9 MPa, respectively. Figure 1 shows that at the lower pressure, wear is reduced as molecular weight is increased. At high M_w , the wear of the grade from supplier 2 is roughly half of that from supplier 1. Interestingly, the variance of performance in the '2L' samples was greater than for the others. At the higher pressure, the same trend was observed with a reduction in wear resulting from an increase in molecular weight. The approximately 2:1 ratio of the wear of supplier 2 versus supplier 1 still holds at the high molecular weight. The most notable result is the large

increase in the wear of the low molecular weight grade from supplier 1 compared to that of the other supplier. The results show a drastic increase in wear for grade ‘1L’ as pressure is increased. In this latter case, variance of the wear amounts is again very large.

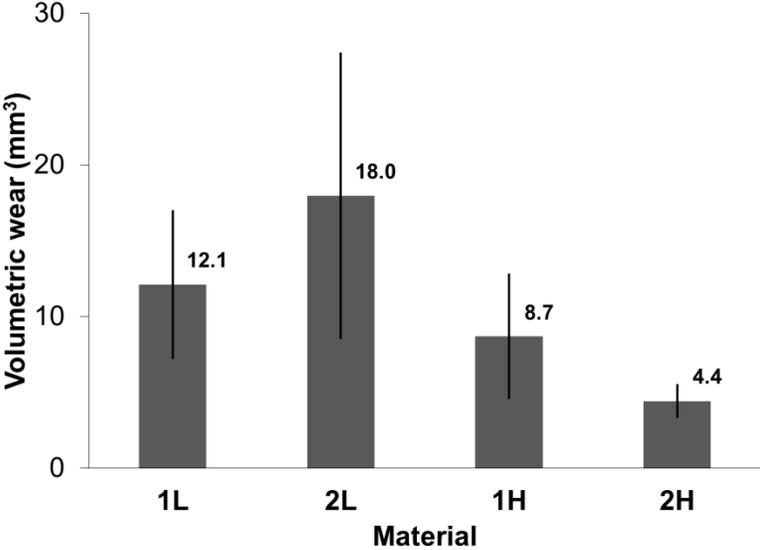


Figure 1. Wear results for the various PEEK materials after 5 km sliding distance under calculated contact pressure of 1.8 MPa. The bars indicate the size of standard error of the means (n=4).

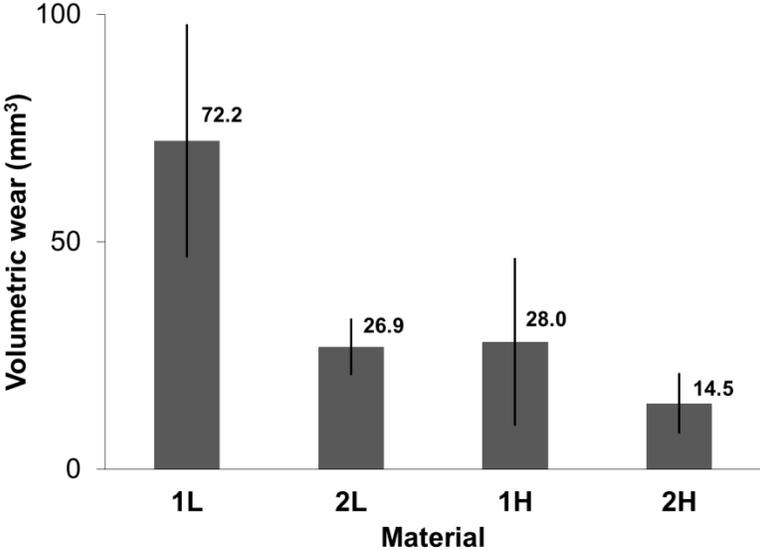


Figure 2. Wear of the various grades after 5 km sliding distance under contact pressure of 3.9 MPa.

Analysis of Variance (ANOVA) was performed on the factorial wear data to determine which, if any, of the three factors (molecular weight, contact pressure, or supplier) had a significant effect on wear behavior. The analysis showed that both molecular weight and contact pressure did indeed affect the resulting wear of the PEEK materials, with p-values of 0.009 and 0.045, respectively. Within the resolution of this experiment, the material supplier did not have a significant effect on wear amounts (p=0.113). However, the interaction between contact pressure and supplier gave indications of potentially

being significant ($p=0.096$), as shown by the drastic effect of increased pressure on the wear of the low molecular weight sample from supplier 1.

3.2 Transfer films

Figure 3 shows selected images of transfer films from supplier 1 at both low and high contact pressures. The results for supplier 2 were very comparable and are thus not shown here. Two things are of note in the images. The first is that thicker regions of film tend to be deposited at the top and bottom of the image, where the sliding direction of the wear pin was perpendicular to the counterface roughness direction. Films become much thinner at the locations where sliding was parallel to roughness. Secondly, there is a qualitative increase in the continuity and what looks to be thickness of the transfer films as both molecular weight and contact pressure are increased. This is most evident when comparing the low weight, low pressure film to that of the high weight, high pressure film in Figure 3(a) and (d), respectively. Another observation of note is that in Figure 3(a), the film looks to be more of a discoloration of the counterface than a substantial deposition of polymeric material. The intermediate films, Figure 3 (b) and (c), show a film that appears to be a collection of discrete lumps of material as opposed to a continuous film. In fact, these images suggest that a more precise definition of the term ‘transfer film’ is required, because there are different morphological characteristics apparent.

In order to determine if there was a strong relationship between average transfer film thickness and wear, data from each of the wear tests was plotted against thickness. Average film thickness was calculated using the thickness measurements from 45° increments around the circumference of each film. The resulting plot is shown in Figure 4. Two pieces of information are apparent from the data. The first is that all films had a mean thickness of at least $2\ \mu\text{m}$. It is not clear if this is a limit that is dependent on the specific properties of the PEEK materials used, or if it depends on the sliding conditions used in the study. Secondly, there does not appear to be any direct relationship between film thickness and the resulting amount of wear. This is a somewhat surprising result in light of a paradigm in the polymer tribology literature that suggests that ‘thin’ films lead to low wear rates. These results show that films with mean thicknesses below approximately $4\ \mu\text{m}$ produce low wear, but low wear is also found in some cases with much thicker films. Another aspect of the relationship that could not be determined from this study is whether the variation in film thickness around the wear path had an effect on the wear amounts. ANOVA was performed using the thickness data for the three experimental factors and it was found that none of the individual factors had a significant effect.

4. Discussion

The overriding conclusion from this study is that there are very clear differences in wear behavior in PEEK with respect to sliding conditions and polymer morphology. The former aspect is routinely expected, but the latter aspect is what is most crucial for the practical uses of these materials. This study represents a well controlled experimental investigation of various PEEK materials in a consistent manner and with very substantial knowledge of the actual polymer morphology of the samples themselves. This approach is vital to producing useful results, because the properties of polymers, including PEEKs, are highly influenced by their processing conditions.

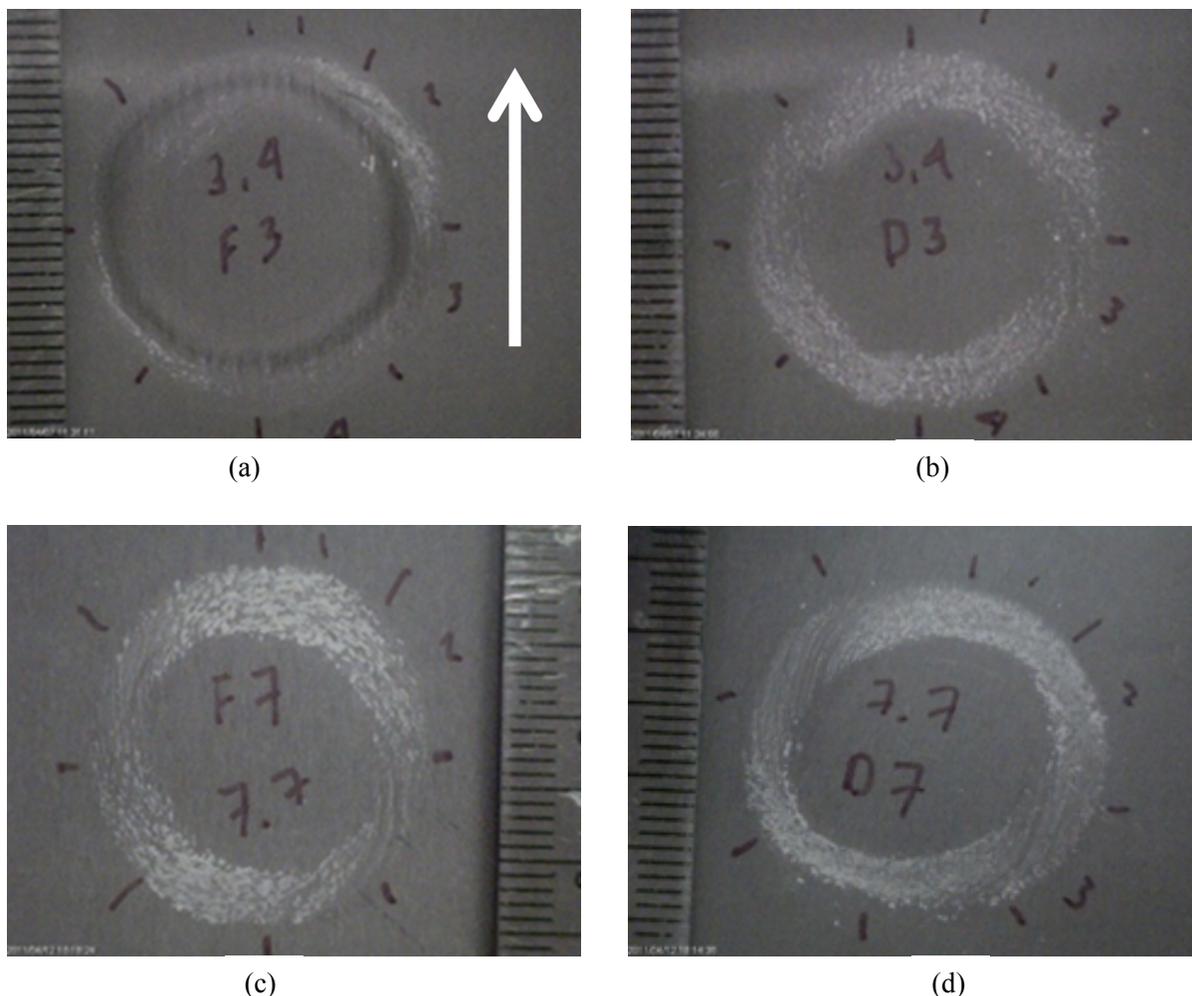


Figure 3. Optical micrograph images of transfer films for PEEK grades from supplier 1 resulting from the following sliding conditions: a) low molecular weight, 1.8 MPa; b) high molecular weight, 1.8 MPa; c) low, 3.9 MPa; and d) high, 3.9 MPa. The tick marks on the scale in this images are separated by 1 mm. Arrow indicates the orientation direction of the counterface surface roughness for all images.

The first issue to consider in analyzing the wear data is why is there a very significant effect of molecular weight at low contact pressure, but less of an effect at the higher pressure. One potential explanation for this behavior may be that the primary difference between the low and high molecular weight materials for a given supplier, was the assumed entanglement density of the polymer chains. Characterizing this was beyond the scope of this study, but it could account for the results observed. Typically, when entanglements are increased in a polymer, mechanical properties such as tensile strength and toughness are increased. Dynamic mechanical properties are not necessarily increased, as evidenced by the results reported in Table 1. It is surmised that with a high number of entanglements, a polymer exhibits greater tenacity when attempts are made to separate fragments from the bulk polymer. In this way, entanglement density might explain the reduced wear behavior of the high molecular weight polymers in two ways. Firstly, it becomes more difficult for the counterface asperities to remove debris from the bulk. Secondly, once material has been removed and is adhered to the counterface, the transfer film has a high mechanical integrity thus maintaining a persistent film. At the high contact pressure, ignoring the low molecular weight material from supplier 1, wear results become more comparable at the different molecular weights. Higher contact pressure may likely produce higher temperatures at the wear interface, and thus the effect of the greater number of entanglements in the high molecular weight materials might be overcome by the

effects of thermal softening of the materials during wear. Interestingly, at high molecular weights, the wear of materials from supplier 2 is approximately half of that from supplier 1. This may indicate a fundamental difference in polymer morphology in the materials beyond molecular weight. In fact, the table indicates that the molecular weight of 2H is somewhat lower than 1H. Other morphological factors that may play a role include differences in entanglement density, crosslinking, or branching. These factors were not characterized in this study but provide clues to future steps to be taken in this investigation. The only substantial difference observed between these two high molecular weight materials is that the loss modulus of 2H is approximately double that of 1H. This further suggests a morphological difference as suggested above, and very significant differences in the viscoelastic response of the materials under loading at the elevated temperatures of the wear interface.

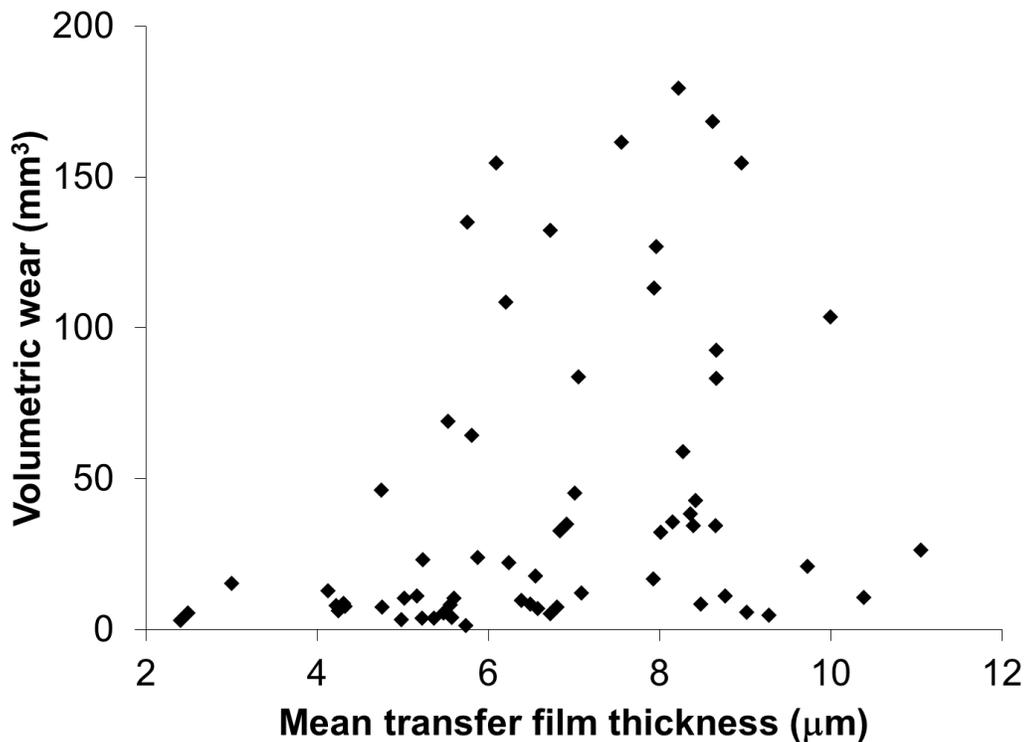


Figure 4. Plot of volumetric wear versus the mean transfer film thickness measured for the individual tests. No strong correlation exists between the parameters.

Another aspect of the wear data that bears examination is the substantial increase in the wear of material 1L when transitioning from low to high contact pressure. At the low pressure, the material has a wear amount comparable to the low weight material from the other supplier. However, the mean wear rate of 1L increases nearly six-fold as the pressure is increased. This behavior is very challenging to explain in light of any differences in entanglements or dynamic mechanical properties, as discussed above. Material 1L has the highest crystallinity of all four materials at 42%. One possible explanation for the wear behavior observed is that at low loads, the increased crystallinity has the effect of imparting wear resistance that is not present in the other three materials. At the high contact pressure, it may be that interface temperatures are high enough to anneal the surface layer of the materials, thus rendering the initial crystallinity irrelevant. Predicting temperatures at the wear interface accurately is an imposing challenge, however thermal modeling in future investigations may shed light on the presence of crystalline morphology during sliding.

Examination of the transfer films has shown that films attain their greatest thickness at the portions of the wear path where the pin was sliding perpendicular to the counterface roughness direction. On the other hand, some tests resulted in a complete lack of film in locations where sliding was parallel to roughness. The explanation for this behavior is most likely the mechanical abrasive action of the hard counterface asperities against the PEEK materials during sliding. Early in the wear process, the asperities are able to remove debris from the bulk polymer and entrap it mechanically into the grooves of the counterface when the roughness is transverse to sliding direction. However, sliding parallel to the roughness direction likely results primarily in plowing of the polymer material without significant debris removal. As entrapped debris accumulates at the perpendicular directions, the transfer film forms as sliding continues. At steady state wear conditions, the situation may change somewhat because the hard asperities at the perpendicular directions are now covered by a soft transfer film. Another aspect of the transfer films is that as contact pressure or molecular weight are increased, the films tend to have more a smeared characteristic especially at the locations on the wear path where sliding was parallel to the roughness direction. This is especially apparent in Figure 3 (c) and (d). It is not visible at the low contact pressure condition shown in Figure 3 (a). This behavior suggests that wear interface temperatures were sufficient to cause viscous flow of the polymer face and/or the top layer of the transfer film, such that softened polymer was smeared along the wear path. Such behavior has been shown in the deposition of transfer films on low-thermal-conductivity counterfaces, such as glass, and typically results in high wear rates [18].

The final issue of consideration regarding these results is the lack of a relationship observed between mean transfer film thickness and the resulting wear in these materials. There are a few potential explanations for this lack of correlation. Firstly, it may be a weakness in the use of the average film thickness over the full circumference of the wear path. The mean takes into account all of the thickness measurements taken at 45° angular intervals, but does not account for the distribution of thicknesses. Furthermore, it is not clear at this point how to analyze films that are not continuous but are collections of discrete lumps of transferred material. Future work will focus on developing quantitative metrics to further characterize the film in light of potential non-uniformity over the entire wear path. A second possible explanation for this result is that there is some mean thickness threshold beyond which wear is accelerated rather than reduced when a film is present. Examining the data, it is clear that film thicknesses less than approximately 4 μm coincided with the lowest wear amounts observed. Thicker films may undergo a continual process of debris removal, film deposition, and film removal. It is likely that such iterative behavior would lead to accelerated wear. A third explanation for this behavior might be that transfer films do not attain an asymptotic thickness at some steady state condition during sliding, but that their thickness may actually exhibit an oscillatory behavior as wear progresses. In the latter case, obtaining a static measure of film thickness does not reflect the true dynamic situation occurring during wear. If such behavior does happen with the PEEK materials studied here, it would be reasonable to characterize time-based measures such as a time constant or a frequency relationship to film height. These would be further parameters to consider in terms of their relationship to wear. Finally, the lack of correlation between film thickness and wear may be due to the fact that the PEEK transfer films were not smooth, continuous films but tended to be discrete patches of deposited material. It could be argued that a discontinuous film might lead to higher wear because of extreme variations at the wear interface. What remains to be investigated with these PEEK materials, is whether continuous films can be achieved under different sliding conditions, or if these materials are averse in some way to depositing uniform films.

5. Conclusions

The following conclusions were drawn based on the results of this study:

1. The family of PEEK materials varies widely in properties among different grades. It was shown that molecular weight played a very significant role in the wear of particular grades of PEEK.

2. The wear of these PEEK materials was significantly affected by the contact pressure during sliding, with higher pressures leading to higher wear. The change in pressure had a more drastic impact on the wear of the low molecular weight grade from supplier 1 than on the other three materials.

3. Transfer films showed their maximum thickness at the locations on the wear path where pin sliding was perpendicular to the counterface roughness orientation. Conversely, the thinnest portions of the transfer films were found when pin sliding was parallel to the roughness direction. This is likely due to a mechanical abrasion and debris anchoring mechanism.

4. Mean transfer film thickness varied from approximately 2.5 μm up to 11 μm . Mean thickness did not show a direct correlation to the wear of the PEEK materials, though it was observed that films with mean thickness of less than 4 μm correlated with materials having the lowest wear.

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