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Kian Bashandeh

Texas A&M University

Pixiang Lan

ATSP Innovations

Andreas A. Polycarpou (✉ apolycarpou@tamu.edu)

Texas A&M University College Station

Research Article

Keywords: PEEK, ATSP, Polyimide, Transfer film, High-temperature tribology

Posted Date: March 22nd, 2021

DOI: <https://doi.org/10.21203/rs.3.rs-327230/v1>

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Tribology of high-performance PEEK-, PI-, and ATSP-based self-lubricating polymers up to 300 °C

Kian Bashandeh¹, Pixiang Lan², Andreas A. Polycarpou^{1*}

¹Department of Mechanical Engineering, Texas A&M University, College Station, Texas, USA

²ATSP Innovations, Houston, TX, USA

*Corresponding author: apolycarpou@tamu.edu; 1-9798455337 (T); 1-9798453081 (F)

ORCID:

Kian Bashandeh: 0000-0002-5720-5379

Andreas A. Polycarpou: 0000-0002-8178-3988

Abstract

High-performance polymers (HPPs) with self-lubricating properties are promising materials for bearing and tribological components that demand low friction and low wear in the absence of liquid lubrication. This study reports on the tribological performance of three advanced HPPs, namely ATSP-, PEEK-, and PI-based polymer composites. The experiments were performed using pin-on-disk configuration under dry sliding conditions and different environmental temperatures from 25 (room temperature) to 300 °C. The role of temperature on the formation of polymer transfer films on the steel counterpart was investigated using microscopy and profilometric measurements, and correlations were made to their tribological performance. From the three tested composites, ATSP-based composite exhibited the best overall performance with low friction and low wear.

Keywords:

PEEK, ATSP, Polyimide, Transfer film, High-temperature tribology

1. Introduction

High-performance polymers (HPPs) and their composites are increasingly used in different industrial machinery components, such as in automotive and aerospace industries, particularly for rubbing parts that demand reliable and durable operation at extreme sliding conditions, such as cryogenic and elevated temperature environments, where the use of conventional lubricants is not feasible. The broad use of HPPs stems from their satisfactory properties such as high load bearing capacity, lightweight, low cost, self-lubricity, and good friction and wear properties [1–3]. Since unfilled polymers suffer from poor tribological performance, they are typically blended with solid lubricants such as polytetrafluoroethylene (PTFE) and graphite flakes, and reinforcements such as carbon/glass fibers to improve their tribological and mechanical performance, respectively [4].

HPPs based on polyether ether ketone (PEEK), Polyimide (PI), and aromatic thermosetting copolyester (ATSP) have been shown to provide self-lubrication and enhanced tribological performance by blending them with solid lubricants [5]. As solid lubricant, PTFE and graphite are shown to enhance the self-lubricity due to their capability to form transfer films on the harder metallic counterface [6]. The development of transfer film is shown to play an important role in the reduction of friction and wear at the sliding interface, which makes polymers an attractive selection for dry sliding components [5]. The transfer film formation depends on parameters such as sliding speed, normal load, temperature, surface roughness, polymer structure, and the type of filler in the polymer matrix [7, 8].

PEEK is a thermoplastic polymer with a glass transition temperature (T_g) of 143 °C whose composites are widely used in many tribological applications [5]. For elevated service temperatures, a maximum operating temperature of 250 °C was reported [9]. PI-based composites are categorized among high operating temperature polymers with excellent friction and wear resistance under unlubricated conditions, particularly at elevated temperatures [10, 11]. ATSP is part of a newer family of HPPs (called Vitrimers; which is a thermoset that processes like a thermoplastic) that was invented in the mid-1990s [12]. Several studies conducted by Polycarpou et al. demonstrated the superior tribological performance of ATSP, compared to PTFE and PEEK polymer coatings for a wide range of temperatures from -196 to 300 °C [13–18]. However, tribological studies in bulk form are scarce, and a few studies investigated the performance of

ATSP/PTFE composites under R-134A refrigerant environment and temperature of 60 °C for air-conditioning compressor applications [8, 19, 20].

The focus of the present study is to investigate the role of environmental temperature (RT to 300 °C) on the tribological performance of commercially available high-performance polymer-based composites, namely PEEK bearing grade (HPV), a polyimide (PI)-based composite known as Vespel SP-21, and an ATSP-based polymer composite, which are shown to stand in the top of the polymer pyramid among the HPPs [1, 5]. Specific attention was given to the role of temperature on the development of transfer films and its subsequent effect on the tribological performance.

2. Experimental

2.1 Materials and sample preparation

Three different polymer composite pins, namely ATSP-based, PI-based (Vespel SP-21), and PEEK bearing grade were used for tribological testing. ATSP resins were synthesized using crosslinkable aromatic copolyester oligomeric systems. The oligomers CB with carboxylic acid functional end group and AB with acetoxyl functional end group were synthesized in a batch melt polymerization in a 2 L reactor at 270 °C under Argon atmosphere and then ground and sieved to a maximum particle size of 90 µm. Detailed description of the synthesis procedure can be found elsewhere [21]. To produce the ATSP-based bulk composites, the oligomers (CB and AB) were mixed with the desired graphite (Microfyne, Asbury Graphite Mills, Inc.) and PTFE (7A X, The Chemours Company FC, LLC) additives, with the weight ratios listed in **Table 1**. The mixture was then cured at 360 °C for 2 hours and ground into smaller size particles that pass through 125 µm sieve. The ground compound was then compressed in compression molding at a temperature of 360 °C under 5000 psi pressure for 2 hours, and then naturally cooled down to RT to obtain the final condensed ATSP bulk composite plate.

Thereafter, the sample was machined into cylindrical pins with a diameter of 6.35 mm for tribological testing. The Vespel and PEEK polymer composites were commercially available and were purchased from the vendors in stock shapes and machined into 6.35 mm diameter pins. Vespel SP-21 is a PI-based polymer that is blended with graphite to enhance its friction and wear properties. PEEK bearing grade is blended with graphite and PTFE to improve the tribological properties, and carbon fiber to enhance the dimensional stability [22]. 416 stainless steel (416SS)

disks were used as the counterpart with a diameter of 50.8 mm and thickness of 6.35 mm. The surface was ground to obtain root mean square surface roughness (R_q) of 0.215 μm .

Table 1. Description of blended polymer materials

Materials	Composition	Vendor
ATSP	CBAB + 30%Graphite + 10% PTFE	ATSP Innovations
Vespel SP-21 (PI)	Vespel [®] (polyimide) + 15% Graphite	DuPont
PEEK bearing grade (HPV)	PEEK + Carbon fiber + graphite + PTFE	Boedeker plastics

2.3 Experimental procedure

The tribological experiments were conducted using a flat-pin-on-disk configuration under dry sliding conditions. The experiments were performed at different environmental temperatures from RT to 300 °C, as summarized in **Table 2**. PEEK was tested only at 25 and 150 °C as the maximum operating temperature is below 300 °C. For each test, a constant normal load of 130 N equivalent to a nominal contact pressure of 4 MPa was applied from the pin side. The nominal contact pressure was calculated by dividing the normal force by the pin nominal contact area. The disk was unidirectionally rotating against the pin at a sliding speed of 1 m/s (530 rpm) for 1-hour duration equivalent to a sliding distance of 3600 m. The in-situ friction and normal forces were recorded by a two-axis transducer to calculate the in-situ coefficient of friction (COF) during the experiment. The experiments were repeated at least three times to ensure repeatability. Before each experiment, the polymer pin and steel disk were immersed in isopropyl alcohol and acetone, respectively, placed in an ultrasonic cleaner for 10 min, and subsequently rinsed with isopropyl alcohol, and dried using warm air. The same procedure was followed after each test and the tested coupons were used for characterization.

Table 2. Details of blended polymers and experimental conditions

Polymer composites	Temperature (°C)	Contact pressure, MPa (load, N)	Linear speed, m/s* (Speed, rpm)
ATSP	25 150, 300	4 (130)	1 (530)
PI			
PEEK	25, 150		

*The linear speeds were calculated based on 18 mm average diameter for the wear tracks

The mass of the polymer samples was measured before and after each test using a scale with a precision of 0.01 mg to calculate the wear rate using the following equation:

$$k = \frac{\Delta m}{F_N d}$$

Where k is the wear rate (mg/Nm), Δm is the polymer mass loss (mg), F_N is the applied normal load (N), and d is the total sliding distance (m). The worn and unworn surfaces of the polymers were examined using scanning electron microscopy (SEM) to visually depict the surface changes and associated wear mechanisms by the tribological testing. Optical microscopy, energy-dispersive X-ray spectroscopy (EDS), and profilometric scans were performed on the tested surfaces of the steel disks to study the formation and variation of polymer transfer films with temperature.

3. Results and discussion

3.1 Friction and wear

Figure 1(a) shows the evolution of unfiltered in-situ COF as a function of sliding distance for the pin-on-disk experiments of blended HPPs at temperatures of 25, 150, and 300 °C. For all polymers, the in-situ COF started with an initial running-in behavior and eventually reached a steady-state period. For RT experiments, both PEEK and ATSP showed the most stable friction after the running-in period. Unlike PEEK and ATSP, PI showed a transient period between two steady-state stages where instabilities in the COF occurred. These variations and instabilities in the COF led to vibration and squeaking noise at the interface, which is undesirable in industrial applications. This behavior could be attributed to the inability of the material to form a transfer film on the counterpart, and therefore the pin continued to wear off due to the plowing of the asperities from the steel disk surface. When the temperature increased to 150 °C, ATSP and PI showed the most stable friction with a decreasing trend, while the COF of PEEK followed an increasing trend and needed the longest time to reach steady-state behavior.

Figure 1. (a) In-situ COF vs. time for all polymer composites, (b) variation of average COF at steady-state, and (c) average calculated wear. Error bars designate ± 1 standard deviation.

Figure 1(b) summarizes the variation of the average COF with temperature. The COF is calculated for the steady-state period, and the error bars in the figure designate the $\pm$ one standard deviation of the averaged calculated COFs. Increasing the testing temperature from RT to 150 °C reduced ATSP and PI COF by 36 and 58%, respectively, while it had an adverse effect on the PEEK COF by increasing it by 71%. For ATSP and PI, the COF followed a decreasing trend with temperature and compared with RT, the COF reduced by 53 and 70% at 300 °C, respectively. Among all tested samples, the highest COF was obtained for PI (0.31) for experiments at RT followed by PEEK (0.29) at 150 °C, and the lowest COF occurred at 300 °C for both ATSP (0.099) and PI (0.092) polymer composites. The high friction of PI at RT could be attributed to its inability to form a uniform transfer layer on the counterpart, as it will be discussed later.

The variations in COF with temperature could be attributed to several different reasons, such as the changes in real contact area (due to changes in polymer elastic modulus), changes in the interfacial shear strength, and the extent and uniformity of transferred polymer to the counterpart. Although the increase of temperature facilitates easier sliding at the interface due to lower shear strength of the softened polymer and reduces friction, lower elastic modulus on the other hand could increase the real contact area and therefore increases the COF. Thus, different trends for friction could be obtained depending on which factor is more dominant. For example, as shown in **Figure 1(b)**, it can be postulated that the decrease of interfacial shear strength was the dominant factor for PI and ATSP, while the increase in real contact area could be the dominant factor for PEEK. This can be explained by the difference in glass transition temperature (T_g) of the polymers. PEEK is reported to have a T_g value around 143 °C [23], while the T_g for the ATSP with oligomers CBAB is 307°C (provided by the manufacturer), and the SP grades of Vespel have no T_g . Therefore, for the experiments at 150 °C, PEEK was exposed to temperatures in the vicinity of its T_g and softened more than the other two polymers. Similar behavior was observed in [24] where during temperature ramping from RT to 240 °C, a significant increase in COF was observed due to reduction in storage modulus at temperatures above T_g up to 180 °C.

Figure 1(c) summarizes the variation of average wear rate of the tested polymers at different temperatures. A similar trend for all tested polymers is the increase of wear from room to elevated temperatures. However, the rate of increase in wear is lower for ATSP, compared with PEEK and PI. Compared with RT, the wear of ATSP and PI at 300 °C increased by 57 and 85%, respectively, and for PEEK the wear increased by 71% at 150 °C. In general, PEEK showed the lowest wear for RT experiments, while ATSP showed minimal wear at elevated temperatures. Overall, ATSP-based composite could be considered as the best performing polymer considering the tribological performance (friction and wear) for this wide range of operating temperatures from 25 to 300 °C, making it an attractive solution for oil-less engineering applications.

3.2 Transfer Film Analysis

Introducing solid lubricants such as PTFE and graphite into polymer matrices is an effective method for reducing friction and wear through the formation of transfer films on the counterface [5, 25, 26]. During sliding, when the interfacial adhesion between the asperities of the tribo-pairs exceeds the cohesive strength of the softer material (typically polymers), the material will begin

to transfer to the counterpart in the form of a thin transfer layer [27]. The formation of such transfer film is also observed in this study and is visually depicted in **Figure 2 (a-c)** using optical microscopy images of the wear tracks on the steel disks.

The optical images of the RT experiments indicate the formation of a uniform and continuous transfer film on the disk surface after testing of ATSP and PEEK, which enabled the protection of the softer polymer against harder asperities of the steel disk. However, the transfer film from PI showed a patchy and non-continuous characteristic and did not properly adhere to the counterface, which caused higher wear of PI compared to the other polymer composites, as shown in **Figure 1(c)**. This behavior could be attributed to the lack of PTFE in the composition of PI, as shown in **Table 1**. The effectiveness of graphite in formation of transfer film is shown to be for temperatures greater than 100 °C, and its wear rate is shown to increase with temperature [28], and thus, it formed a non-uniform film at RT. Increasing the temperature helped to develop a uniform and continuous transfer film after testing with ATSP and PI, which helped to reduce friction. As shown in **Figure 1(a)**, the experiments at elevated temperatures took a longer time to reach steady-state condition, compared with RT experiments, indicating longer time to form the uniform transfer film on the counterface. Therefore, the increased wear rate at elevated temperatures could be attributed to continuous high wearing of material by the hard asperities of the metallic counterpart until a stable and uniform film was formed.

Figure 2. Optical microscopy images of the steel disks after experiments at different temperatures with (a) ATSP-based, (b) PI-based, and (c) PEEK-based polymer composites (scale bar is 1 mm for all images).

Figures 3(a-c) show the SEM images and corresponding EDS mapping of the steel disk surfaces after testing with ATSP, PI, and PEEK at RT. The formation of transfer film from the polymer pins to the disk is clearly discernible from the SEM images, revealed by darker areas on the surface. However, the extent and uniformity of the transferred films are not the same among the polymers. **Figure 3(a)** shows the transfer material from ATSP pin to the steel surface showing a uniform and continuous transfer film. PI on the other hand formed irregular transfer film, and only small patches were developed on the asperity crevices at RT, as shown in **Figure 3(b)**. The obtained SEM image of the steel surface after testing with PEEK (shown in **Figure 3(d)**) indicates the evidence of continuous transfer film, but with lower area coverage compared with ATSP. The EDS analysis of

the developed film on the steel surface shows that the dark areas on the wear track of the SEM images correspond to carbon elements, which is the primary atom in the polymer structure.

Figure 3. SEM images and corresponding EDS analysis of the steel surface showing the formation of transfer film by sliding of (a) ATSP, (b) PI, and (c) PEEK at RT (scale bar is 25 μm for low magnification (left) and 10 μm for high magnification (middle and right) images).

Figures 4(a-c) show representative profilometric line scans across the wear track on the surface of the steel disks after testing with ATSP, PI, and PEEK, respectively. From **Figure 4(a)**, a continuous transfer of material from ATSP is evident for experiments at RT and 150 $^{\circ}\text{C}$, with transferred layer thickness of approximately 0.4 and 0.15 μm , respectively. For the experiment at 300 $^{\circ}\text{C}$, the thickness of the transferred film was less than the stylus resolution, and therefore it could not be measured. **Figure 4(b)** shows similar line scans for experiments with PI. All the scans show no change on the surface topography outside and inside the wear track due to the inability of the material to form a transfer layer at RT, and inability of the stylus to measure the extremely thin transferred film at the temperatures of 150 and 300 $^{\circ}\text{C}$. In the case of PEEK bearing grade, the

profile at RT (**Figure 4(c)**) shows a uniform and continuous transferred layer with thickness of 0.5 μm . However, the scan at 150 $^{\circ}\text{C}$ shows the development of partial and non-continuous film. The formation of such discontinuous film and deterioration of the mechanical properties, namely elastic modulus at 150 $^{\circ}\text{C}$, could be the reason for higher friction and wear for PEEK.

Figure 4. Profilometric measurements on the surface of steel disks after experiments at different temperatures with (a) ATSP, (b) PI, and (c) PEEK bearing grade polymers. Red lines represent the wear scar.

3.3 SEM Analysis of the Worn Surfaces

The SEM images of the untested and worn surfaces of the polymeric samples were taken to study the wear mechanisms associated with the experiments at different temperatures. The samples were sputter-coated with 4 nm layer of Pt/Pd using a sputter coater machine to obtain the SEM images.

Figures 5(a-d) show the SEM images of the untested and tested surfaces of ATSP pins at different temperatures. The surface texture of the untested ATSP is depicted in **Figure 5(a)** showing a rough

topography after machining. The SEM images of the worn surfaces after experiments in **Figures 5(b-d)** indicate that the dominant wear mechanism at all temperatures is polishing and removal of top asperities and initial irregularities in the topography of the surface.

Figure 5. SEM images of ATSP composite pins (a) untested surface, and tested surfaces at temperatures of (b) 25 °C, (c) 150 °C, and (d) 300°C. The solid white arrow in (d) shows the sliding direction for all cases.

Figure 6(a) shows the SEM image of the untested PI composite surface. In general, the surface features a smoother topography compared to ATSP and contains directional shallow grooves from machining. As shown in **Figures 6 (b-d)**, the removal and polishing of the initial irregularities and grooves are characteristics of the worn surface of the PI composite pins after sliding at all temperatures causing the surface to become smoother than the as-received one. However, as shown in **Figure 6(a)**, sliding at RT caused severe micro-cuttings and scratches on the surface, implying harsh sliding conditions at the interface, which contributed to high friction and wear at RT.

Figure 6. SEM images of PI composite pins (a) untested surface, and tested surfaces at temperatures of (b) 25 °C, (c) 150 °C, and (d) 300°C. The white solid arrow in (d) shows the sliding direction for all cases.

The SEM image of the untested surface of PEEK composite is shown in **Figure 7(a)**. The surface features a rough topography and contains machining marks, tracks and scrapes from machining. However, similar to the other polymer composites, the surface became smoother upon sliding at both RT and 150 °C. The SEM image after the RT experiment in **Figure 7(b)** shows the exposed carbon fibers on the worn surface (shown with dashed arrows), which were embedded in the matrix in different orientations. Since PEEK is less wear resistant than carbon fibers, it can easily wear out, and therefore the majority of the load will be carried out by the fibers, which gives the composite a high strength and wear resistance. Due to different modulus between carbon fiber and PEEK matrix, the stress concentration can be generated at the matrix/fiber interface, which contributes to debonding at the interface or detachment of fiber from the matrix [29]. From **Figure 7(c)**, some regions show pitting marks that could be from detachment of fibers and interfacial cracking (marked with the dashed circle). Note that the detached fibers serve as third-body abrasive

particles and therefore generate abrasive marks on the surface, as shown with the white solid arrow in **Figure 7(b)**. Signs of plastic deformation and continuous grinding of carbon fibers are extra surface features once the polymer was slid at higher temperature (**Figure 7(d)**).

Figure 7. SEM images of PEEK composite pins (a) untested surface, and tested surfaces at temperatures of (b) 25 °C at low magnification, (c) 25 °C at high magnification, and (d) 150 °C. The solid arrows in (b) show the abrasive marks and dashed arrows show the embedded carbon fibers. The dashed circle in (c) show the interfacial cracking and pitting. The solid white arrow in (d) shows the sliding direction.

The comparison of wear mechanisms among the polymers revealed that ATSP was the least affected polymer when the temperature changed from RT to 300 °C and the surface experienced only mild burnishing at all temperatures resulting in a good friction and wear. The PI wear mechanism changed from severe micro-cutting and abrasive wear at RT, to burnishing effect at elevated temperatures resulting in lower friction at elevated temperatures. The PEEK wear mechanism on the other hand showed the plastic deformation of the surface at elevated temperatures, indicating the deterioration of mechanical properties at 150 °C that resulted in high COF, compared with RT.

4. Conclusion

The tribological performance of three high-performance polymer-based composites, namely ATSP-based, PI-based, and PEEK-based polymer composites, were experimentally investigated under different environmental temperatures of 25, 150, and 300 °C under dry sliding conditions. The variation of COF and wear and evolution of transfer layer on the counterface with temperature were studied, and the following conclusions could be drawn:

- For ATSP and PI-based composites, the COF followed a decreasing trend with increase of temperature from 25 to 300 °C: It was reduced by 53 and 70% at 300°C, compared with RT. The COF of PEEK-based composite increased by 71% when the temperature increased to 150 °C due to formation of a discontinuous film and deterioration of mechanical properties.
- The transfer film was found to play significant role in the tribological performance at all temperatures. ATSP developed a uniform and continuous transfer film at all temperatures, whereas PI could not develop the transfer film at RT, resulting in high friction and wear. Increase in temperature helped to develop a uniform and continuous transfer film, leading to a decrease in the COF. PEEK could not maintain its uniform developed transfer film at RT, once the temperature was raised to 150 °C resulting in high COF and wear at elevated temperature.
- For all polymer composites, the wear followed an increasing trend with temperature due to the inability of the polymers to develop the transfer layer at the early stage of sliding period, and therefore the material was worn out faster until a stable film was formed on the counterface. The dominant wear mechanism for all polymers was polishing of the surface, as shown by SEM analysis. In addition, severe micro-cuttings and scratches formed on the PI surface at RT, which caused high friction and wear.
- Based on the overall tribological performance at all operating temperatures, the ATSP-based composite is recommended as the best performing polymer for use in oil-less engineering applications that demand reliable operation in a wide range of temperatures.

Declarations**Funding**

The authors did not receive support from any organization for the submitted work.

Conflicts of interest/Competing interests

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Andreas Polycarpou is a professor at Texas A&M and also a co-founder of the startup company ATSP Innovations and Pixiang Lan is a full-time research engineer at ASTP Innovations.

Availability of data and material

Data not available / Data will be made available on request

Code availability

Not applicable

Ethics approval

Not applicable

Consent to participate

Not applicable

Consent for publication

Not applicable

Acknowledgments

The authors acknowledge the use of the Texas A&M Materials Characterization Facility (MCF), where SEM/EDS experiments were performed.

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Figures

Figure 1

(a) In-situ COF vs. time for all polymer composites, (b) variation of average COF at steady-state, and (c) average calculated wear. Error bars designate ± 1 standard deviation.

Figure 2

Optical microscopy images of the steel disks after experiments at different temperatures with (a) ATSP-based, (b) PI-based, and (c) PEEK-based polymer composites (scale bar is 1 mm for all images).

Figure 3

SEM images and corresponding EDS analysis of the steel surface showing the formation of transfer film by sliding of (a) ATSP, (b) PI, and (c) PEEK at RT (scale bar is 25 μm for low magnification (left) and 10 μm for high magnification (middle and right) images).

Figure 4

Profilometric measurements on the surface of steel disks after experiments at different temperatures with (a) ATSP, (b) PI, and (c) PEEK bearing grade polymers. Red lines represent the wear scar.

Figure 5

SEM images of ATSP composite pins (a) untested surface, and tested surfaces at temperatures of (b) 25 °C, (c) 150 °C, and (d) 300°C. The solid white arrow in (d) shows the sliding direction for all cases.

Figure 6

SEM images of PI composite pins (a) untested surface, and tested surfaces at temperatures of (b) 25 °C, (c) 150 °C, and (d) 300°C. The white solid arrow in (d) shows the sliding direction for all cases.

Figure 7

SEM images of PEEK composite pins (a) untested surface, and tested surfaces at temperatures of (b) 25 °C at low magnification, (c) 25 °C at high magnification, and (d) 150 °C. The solid arrows in (b) show the abrasive marks and dashed arrows show the embedded carbon fibers. The dashed circle in (c) show the interfacial cracking and pitting. The solid white arrow in (d) shows the sliding direction.