Effect of temperature and atmosphere on the tribological behavior of a polyether ether ketone composite

Felipe Darabas RZATKI, Diego Vinicius Dalevedo BARBOZA, Renan Muller SCHROEDER, Guilherme Mariz de Oliveira BARRA, Cristiano BINDER, Aloisio Nelmo KLEIN, José Daniel Biasoli DE MELLO

1 Federal University of Santa Catarina—UFSC, Mechanical Engineering Department, Materials Laboratory, Florianópolis, SC, Brazil
2 Whirlpool/Embraco, Joinville, SC, Brazil

Received: 29 June 2015 / Revised: 26 August 2015 / Accepted: 17 September 2015
© The author(s) 2015. This article is published with open access at Springerlink.com

Abstract: Polyether ether ketone (PEEK) is a high-performance thermoplastic, which is often selected for high-temperature tribological applications under chemically aggressive environments. The present work investigates the tribological behavior of a high-performance PEEK composite under conditions that are often found inside hermetic compressors. Therefore, an AMTI tribometer equipped with a hermetic chamber and a heating system was used to conduct sliding tests of PEEK cylinders on AISI 304 stainless steel polished discs ($S_q < 10$ nm) with reciprocating movement and a normal force of 175 N. The tribological behavior of the PEEK/AISI 304 stainless steel system was investigated as a function of ambient temperature (30 °C and 80 °C) and atmosphere (atmospheric air and tetrafluoroethane). Wear and surface roughness analyses were performed with white light interferometry and optical microscopy. Raman spectroscopy was used to investigate transfer films on the counter body surface. Temperature was observed to have a strong influence on the tribological behavior of the samples tested under atmospheric air, with a 25% decrease in the friction coefficient associated with a 100% increase in the wear rate. However, the friction measured from the samples tested under a tetrafluoroethane atmosphere showed no significant temperature dependence.

Keywords: solid lubricant; thermoplastics; environment; temperature

1 Introduction

Over the recent decades, the refrigerant industry has introduced several different refrigerant fluids—a change that is generally driven by environmental issues [1–7]. More recently, the refrigerant industry has shown interest in oil-less compressors [8, 9]. This new generation of products, in addition to having environmental benefits, attains new levels of efficiency and allows for the development of innovative refrigerators [10]. In this context, solid lubricant polymeric materials are a promising alternative to reduce the friction and wear in these lubricant-free systems. However, the harsh operating conditions of hermetic compressors require the use of high-performance polymers. Compounds based on polyether ether ketone (PEEK) are certainly some of the most promising materials in current polymer tribology [11].

Few studies regarding the tribological behavior of polymeric materials under dry conditions and refrigerant atmospheres are available [2–7]. Cannaday and Polycarpou [3] found that a tetrafluoroethane atmosphere resulted in tribological properties that were slightly superior to those reported in atmospheric air for several polymeric materials and composites. Moreover, McCook et al. [12] reported that neat PEEK showed a lower friction coefficient and less wear under dry and vacuum environments. However, the effect of the ambient temperature on these systems remains unclear.
The main goal of the present work is to investigate the impact of the ambient temperature and refrigerant atmospheres on the tribological behavior of a wear-resistant, solid-lubricated PEEK composite. Thus, a harsh cylinder-on-disc configuration and reciprocating movement were chosen as test conditions. White light interferometry, optical imaging, and Raman spectroscopy provide further insights into possible interactions between polymer composites and their test environments.

2 Materials and methods

A commercially available 10% PTFE, 10% graphite, and 10% carbon fiber (CF)-filled PEEK composite was selected for its superior self-lubrication and wear characteristics [11]. The material was provided as an 11-mm-thick injection-molded plate, which was machined into 8 mm rods and then sliced into cylinders of 4 mm height. Discs (30 mm in diameter) of AISI 304 stainless steel were chosen as the counter body material. Table 1 presents the nominal mechanical properties of the selected materials.

The tribological behavior of the PEEK/AISI 304 stainless steel pair was investigated as a function of ambient temperature (30 °C and 80 °C) and atmosphere (atmospheric air and tetrafluoroethane).

The surface of the AISI 304 stainless steel discs was prepared by sanding with 600- and 1000-mesh abrasive sand paper, followed by polishing with a 1 μm-diameter diamond abrasive. After this surface finishing step, the discs were subjected to ultrasonic cleaning in ethanol for 15 min. A white-light interferometer (Zygo New View 7200) was used to evaluate the resulting topography. A Gaussian filter (800 μm) was applied during surface roughness analysis to remove waviness from the sample surfaces.

Dry tribological tests were conducted in a servo hydraulic AMTI tribometer equipped with a hermetic chamber, a heating system, a 2-channel load cell, and closed-loop actuator controls (load and displacement). This apparatus was configured in a cylinder-on-plate mode (Fig. 1) with linear reciprocating movement under a constant normal load of 175 N. Each test was performed for 2 h at a frequency of 2 Hz and a stroke of 10 mm. Before the initiation of each tribological test under the tetrafluoroethane atmosphere, the refrigerant gas was purged three times using a mechanical vacuum pump to eliminate atmospheric contaminants.

The polymer wear rates were calculated using geometric wear volume measurements obtained from white-light interferometry. The results were constructed from an average of at least three tests under each condition. The wear tracks were analyzed using white-light interferometry, optical microscopy (Olympus BX60), and Raman spectroscopy (Renishaw 2000, equipped with a 514 nm argon laser) to obtain further information regarding wear mechanisms and tribo-layer formation.

3 Results and discussion

Figure 2 presents typical axonometric projections from virgin and worn polymeric cylinders. The geometric data show that the fiber-reinforced composites did not undergo long-range plastic deformation during the sliding test. Therefore, the enlargement of the apparent contact area in the worn cylinders (Fig. 2(b)) is attributed exclusively to material removal, i.e., volumetric wear. Moreover, because of the contact area enlargement, the initial maximum Hertzian pressure of 210 MPa dropped to nominal pressures of approximately 40 MPa.
The maximum Hertzian pressure ($p_{\text{max}}$) of the cylinder’s on-plane contacts is given by the expression:

$$p_{\text{max}} = \left( \frac{E'F}{\pi l R} \right)^{1/2}$$

(1)

$$\frac{1}{E'} = \frac{1 - v_1^2}{E_1} + \frac{1 - v_2^2}{E_2}$$

(2)

where $F$ is the applied load, and $l$ and $R$ are the cylinder length and radius, respectively. $E$ and $v$ represent the Young’s Modulus and the Poisson coefficient, whereas the indices 1 and 2 represent the polymeric cylinder and the metallic counter body, respectively. The nominal pressure is given by the applied load divided by the apparent contact area (worn area in Fig. 2(b)).

The resulting average wear rates experienced by the polymeric composites are summarized in Fig. 3. Atmospheric air and tetrafluoroethane showed no representative effect on the polymer wear rates. Cannaday and Polycarpou [3] reported that PEEK and PEEK composites showed a slight wear rate reduction when tested in a tetrafluoroethane atmosphere. However, the temperature drove the increase in wear rates from $1.7 \times 10^{-6} \text{mm}^3/(\text{N}\cdot\text{m})$ at 30 °C to approximately $3.5 \times 10^{-6} \text{mm}^3/(\text{N}\cdot\text{m})$ at 80 °C. At high temperatures, the cohesive properties of the polymeric matrix were significantly reduced, thus enhancing the polymer abrasive wear [13–15].

Figure 4 presents typical axonometric projections from virgin and as-worn counter body surfaces. Grooves aligned in the sliding direction on the metallic surfaces were formed during the tribological tests.

The resulting average root mean square roughness ($R_a$) values measured on the virgin and worn counter body surfaces are summarized in Fig. 5. Sliding tests increased the counter body surface roughness by approximately 600%. It is clear that the tribological tests drastically changed the topography of the counter bodies. However, the resulting roughness after the tribological tests seemed to be independent of evaluated temperatures and atmospheres.

Moreover, profile analyses on wear tracks were performed to evaluate the nature of the grooves displayed in Fig. 4. As exemplified in Fig. 6, peaks and valleys were defined from the mean height of the virgin region, and their areas were calculated. Six profiles were evaluated for each tested condition. From the results, no statistically relevant difference was observed between the total area of the peaks and valleys. In other words, representative volumetric changes did not occur on the metallic surfaces. Therefore, it is possible to attribute groove formation to a microploughing abrasive mechanism [16]. Because successive plowing leads to microfatigue wear mechanisms [16], one can consider these grooves to have been mainly produced in the earlier stages of tribological contact, when the nominal contact pressure was close to 210 MPa, which is about the yield stress of AISI 304 stainless steel (215 MPa). However, previous work [17] has revealed that even

![Fig. 2](image1.png)  
**Fig. 2** Geometry of PEEK cylinder before (a) and after (b) wear test at 30 °C in a tetrafluoroethane atmosphere. Red arrow indicates the sliding direction.

![Fig. 3](image2.png)  
**Fig. 3** Wear rate of PEEK cylinders during sliding tests against AISI 304 stainless steel discs.
under nominal contact pressures below the yield stress of the metallic material, the polymeric portion is still able to plow metallic polished surfaces.

Figure 7 presents typical results from image analyses of worn samples. Micrographs of PEEK wear regions revealed grooves aligned in the direction of sliding, typical of microploughing wear mechanisms, and shear-induced plastic flow regions on the boundary of the worn surfaces, attributed to smearing [11, 14, 16]. Additionally, as the cylinders wear out, one could observe carbon fibers protruding from the active surface. Contact between these spots and the metallic surface produces elevated pressures and temperatures, which could plow the counter body surface and result in the grooves shown in Fig. 4 [17–19]. Subsequently, abraded metallic surfaces filled with sharp asperities were able to plow the polymer surface and wear out the carbon fiber edges. However, although the topographical analyses did not show evidence of significant material deposition on the wear tracks, micrographs from the counter body surface revealed the formation of a tribo-layer. According to Ref. [17], the grooves plowed into polished counter bodies enhance the establishment of a tribo-layer. Note that free carbon fibers were not observed on the counter body surface.
Raman spectroscopy analyses (Fig. 8) performed on the observed tribo-layers revealed \( D \sim 1,360 \text{ cm}^{-1} \) and \( G \sim 1,580 \text{ cm}^{-1} \) bands, which are common to graphite-based structures [20, 21]. The low band intensities obtained from the samples, tested at 30 °C, indicate that the tribo-layer formed under these test conditions is thinner than the one formed at higher temperatures. This behavior is in agreement with Sheiretov et al. [2] who reported that higher temperatures enhance the formation of uniform tribo-layers. Furthermore, the higher intensity ratio between the D and G bands \( (I_D/I_G) \) from samples tested under atmospheric air at 80 °C (0.97 vs. 0.70 from tetrafluoroethane at 80 °C) indicates greater disorder in its graphite structure [20, 21]. The origin of these graphite-based tribo-layers remains unclear because it can be attributed to different carbon sources, such as (i) graphite fillers from polymer compositions; (ii) degraded polymers (PEEK and PTFE); and (iii) degraded carbon fibers.

Figure 9(a) shows the evolution of the friction coefficient during the sliding tests, whereas Fig. 9(b) shows their average steady-state values. Three tests were performed for each condition, and an average friction coefficient was calculated from each average value within the steady-state regime.

During the running-in regime, samples tested in atmospheric air exhibited friction coefficients 25% higher than those of samples tested in the tetrafluoroethane atmosphere. However, once the steady-state regime was established, the samples tested in atmospheric air and at high temperatures exhibited the lowest friction coefficients, with values of approximately 0.34. At low temperatures, the friction coefficients slightly changed from running-in to steady-state regimes, and the samples tested under the tetrafluoroethane atmosphere showed no significant temperature dependence, with steady state friction coefficients varying around 0.38.

The observed friction behavior results from dissipated energy, attributed to the adhesive interfacial forces and plowing process on the polymeric and counter body surfaces. The adhesive interfacial forces are often affected by the formation of tribo-layers, the establishment of which has long been recognized as the reason for the gradual transition from the running-in to steady-state regime [13, 14, 17].

According to Yen et al. [22], “the presence of vapors, such as water, is required for graphite to lubricate.” Thus, the water vapor present in atmospheric air reduces the bonding energy between the hexagonal planes of the graphite structures present in the formed graphite-based tribo-layer. This behavior agrees with
the lower steady-state friction coefficient observed in the samples tested in atmospheric air at 80 °C. Moreover, according to McCook et al. [12], the relative humidity present in atmospheric air increases the friction coefficient of the PEEK matrix running against the metallic surfaces. Therefore, it is reasonable to assume that during running-in and steady-state regimes at low temperatures, the adhesive interfacial forces are governed by interactions between the PEEK matrix and the metallic surface, rather than the tribo-layer. In other words, the tribo-layers during running-in regimes and steady-state regimes at low temperatures were not thick enough to be effective.

4 Conclusions

1. PEEK composite wear rates were drastically affected by the test temperature, but no significant atmospheric dependence was detected.
2. Sliding tests wore AISI 304 stainless steel counter bodies under all testing conditions, resulting in an increased surface roughness ($R_q$) but no volumetric losses.
3. Graphite-based tribo-layers were observed on counter body surfaces after the sliding tests.
4. Higher test temperatures enhanced the formation of tribo-layers, and the presence of humid atmospheric air resulted in graphite structures with greater disorder, and consequently lower friction coefficients.
5. Without an effective tribo-layer, the presence of humidity in atmospheric air enhanced the system’s friction coefficient.
6. The friction of samples tested in a dry tetrafluoroethane atmosphere showed no significant temperature dependence.

Acknowledgements

The authors wish to thank CNPq, FAPESC and Capes/Proex for financial support.

Open Access: This article is distributed under the terms of the Creative Commons Attribution Noncommercial License which permits any noncommercial use, distribution, and reproduction in any medium, provided the original author(s) and source are credited.

References

[1] Lorentzen G. The use of natural refrigerants—A complete solution to the CFC/HCFC predicament. *Int J Refrig* **18**(3): 190–197 (1995)
[2] Sheiretov T, Vanglabbeek W, Cusano C. Evaluation of the tribological properties of polyimide and poly(amide-imide) polymers in a refrigerant environment. *Tribol Trans* **38**(4): 914–922 (1995)
[3] Cannaday M L, Polycarpou A A. Tribology of unfilled and filled polymeric surfaces in refrigerant environment for compressor applications. *Tribol Lett* **19**(4): 249–262 (2005)
[4] Demas N G, Polycarpou A A. Tribological performance of PTFE-based coatings for air-conditioning compressors. *Surf Coat Tech* **203**(3–4): 307–316 (2008)
[5] Dascalescu D, Polychronopoulou K, Polycarpou A A. The significance of tribochemistry on the performance of PTFE-based coatings in CO$_2$ refrigerant environment. *Surf Coat Tech* **204**(3): 319–329 (2009)
[6] Nunez E E, Yeo S M, Polychronopoulou K, Polycarpou A A. Tribological study of high bearing blended polymer-based coatings for air-conditioning and refrigeration compressors. *Surf Coat Tech* **205**(8–9): 2994–3005 (2011)
[7] Yeo S M, Polycarpou A A. Tribological performance of PTFE- and PEEK-based coatings under oil-less compressor conditions. *Wear* **296**(1–2): 638–647 (2012)
[8] Solzak T A, Polycarpou A A. Tribology of WC/C coatings for use in oil-less piston-type compressors. *Surf Coat Tech* **201**(7): 4260–4265 (2006)
[9] De Mello D B, Binder R, Demas N G, Polycarpou A A. Effect of the actual environment present in hermetic compressors on the tribological behaviour of a Si-rich multifunctional DLC coating. *Wear* **267**(5–8): 907–915 (2009)
[10] Oil-less linear compressor launched. Cooling spot. http://www.coolingpost.com/world-news/oil-less-linear-compressor-launched/, 2014.
[11] Schroeder R, Torres F W, Binder C, Klein A N, De Mello D B. Failure mode in sliding wear of PEEK based composites. *Wear* **301**(1–2): 717–726 (2013)
[12] McCook N L, Hamilton M A, Burris D L, Sawyer W G. Tribological results of PEEK nanocomposites in dry sliding against 440C in various gas environments. *Wear* **262**(11–12): 1511–1515 (2007)
[13] Stachowiak G W, Batchelor A W. *Engineering Tribology*. Oxford (UK): Butterworth-Heinemann, 2005.
[14] Briscoe B J, Sinha S K. Wear of polymers. *J Eng Tribol* **216**(6): 401–413 (2002)
[15] Hanchi J, Eiss N S. Dry sliding friction and wear of short carbon-fiber-reinforced polyetheretherketone (PEEK) at elevated temperatures. Wear 203–204(1): 380–386 (1997)

[16] Zum Gahr K H. Microstructure and Wear of Materials, Tribology Series 10. Amsterdam (NE): Elsevier, 1987.

[17] Rzatki F D, Barboza D V D, Schroeder R, Barra G M O, Binder C, Klein N A, De Mello D B. Effect of surface finishing, temperature and chemical ageing on the tribological behaviour of a polyether ether ketone composite/52100 pair. Wear 332–333(1): 844–854 (2015)

[18] Kalin M. Influence of flash temperatures on the tribological behavior in low-speed sliding: a review. Mater Sci Eng A 374(1–2): 390–397 (2004)

[19] Tzanakis I, Conte M, Hadfield M, Stolarski T A. Experimental and analytical thermal study of PTFE composite sliding against high carbon steel as a function of the surface roughness, sliding velocity and applied load. Wear 303(1–2): 154–168 (2013)

[20] Binder C. Desenvolvimento de novos tipos de aços sinterizados autolubrificantes a seco com elevada resistência mecânica aliada a baixo coeficiente de atrito via moldagem de pó por injeção. Ph.D. Thesis. Florianópolis (Brazil): Universidade Federal de Santa Catarina, 2009.

[21] Kaniyoor A, Ramaprabhu S. A Raman spectroscopic investigation of graphite oxide derived graphene. J Appl Phys 2(3): 032183 (2012)

[22] Yen B K, Schwickert B E, Toney M F. Origin of low-friction behavior in graphite investigated by surface x-ray diffraction. Appl Phys Lett 84(23): 4702–4704 (2004)

Felipe Darabas RZATKI. He received his Bachelor and M.S. degree in materials engineering from Federal University of Santa Catarina, Florianópolis, Brazil, in 2010 and 2012 respectively. Currently, he is a Ph.D. student in the Materials Laboratory at the same university. His research interests include fatigue and tribology of polymeric materials.

José Daniel Biasoli DE MELLO. He is an Emeritus Professor in the College of Mechanical Engineering at the Federal University of Uberlândia, Brazil and visiting professor at the Federal University of Santa Catarina, Florianópolis, Brazil. He is also a senior researcher (level 1) of the National Research Council (CNPq), Brazil. He received his B.S. degree in mechanical engineering from the Federal University of Uberlândia. In 1983, he received his Doc. Ing. degree in metallurgy from the Institut National Polytechnique de Grenoble, Grenoble, France. He acted as Professeur Associé in the École Nationale Supérieure de Mécanique et Microtechniques, Besançon, France (1990). In 1998—1999, he worked as a visiting scholar at the Department of Materials Science and Metallurgy, University of Cambridge, UK. In 2007 he worked as a Fulbright visiting professor at the University of Illinois at Urbana-Champaign, USA. He is the director of The Tribology Technical Division of The Brazilian Society for Metals, Materials and Mining and member of the editorial board of several journals including Friction. Professor De Mello has published more than 250 full papers in proceedings of national and internationals conferences, congresses and journals. Professor De Mello's current research are tribo-corrosion, abrasion-corrosion, surface durability of solid lubricants and tribological behaviour of sintered material.