

In Vacuo Tribological Behavior of Polytetrafluoroethylene (PTFE) and Alumina Nanocomposites: The Importance of Water for Ultralow Wear

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Abstract Polytetrafluoroethylene (PTFE) is widely regarded as an excellent candidate for solid lubrication in vacuum. However, it is often precluded from many practical applications due to its intrinsically high wear rate. Over the past decade, it has been discovered that small loading fractions of alumina nanofillers can increase the wear resistance of PTFE by three to four orders of magnitude. This dramatic increase in wear resistance has in turn prompted numerous tribological studies to examine the robustness of this performance. In this study, the wear and friction behavior of unfilled PTFE and PTFE and alumina nanocomposites were evaluated under a broad range of vacuum environments from 760 to 4×10^{-6} Torr. The nanocomposites of PTFE/alumina showed a dramatic increase in wear of over two orders of magnitude at the highest vacuum conditions. There appears to be an optimal vacuum environment around 1–10 Torr, in which these samples achieved the lowest wear rates of approximately $2.5 \times 10^{-7} \text{ mm}^3/(\text{Nm})$.

Keywords Polytetrafluoroethylene · Alumina · Nanocomposite · Tribochemistry · Wear · Friction · Vacuum

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1 Introduction

Polytetrafluoroethylene (PTFE) is an excellent candidate material for solid lubricant applications due to its characteristic vacuum compatibility, low friction, and high melting temperature. However, its utility as an engineering polymer is impaired by its high wear rate [$\sim 7 \times 10^{-4} \text{ mm}^3/(\text{Nm})$] [1–5]. In 2006, it was reported that the wear of PTFE could be reduced by three to four orders of magnitude when filled with a small amount ($\sim 5 \text{ wt\%}$ or less) of an alumina nanofiller [6], resulting in an ultralow wearing polymer nanocomposite [6–16]. If the advantageous properties of PTFE could be preserved along with this remarkable wear rate, numerous applications in space and extraterrestrial atmospheres would benefit. While it is important to evaluate candidate materials in the environment in which they will be used [9], the relative ease of performing vacuum studies in the laboratory is an ideal starting point.

The detailed mechanisms that underlie the ultralow wear behavior of these nanocomposites remain a mystery. At such low loadings of fine alumina particles, the wear reduction cannot be a simple rule of mixtures and must involve other mechanisms including tribofilms generated on the countersample (transfer film) and wear surface of the polymer (running film) [13, 17, 18]. These particular alumina-containing nanocomposites have been repeatedly used as samples to interrogate the origins of ultralow wear behavior [6–15]. Tribological studies in various humidity and oxygen environments show that the ultralow wear behavior of this nanocomposite depends in part on the presence of moisture and oxygen in the surrounding environment [8]. This finding suggests tribochemical mechanisms participating in the formation of ultralow wearing interfaces. Furthermore, X-ray photoelectron spectroscopy (XPS) of the tribofilms generated by the PTFE and alumina nanocomposites under atmospheric conditions revealed

partially degraded PTFE, a new chemical species that was not present in the unfilled PTFE sample [8, 19, 20].

In this investigation, we report on the role of vacuum environments on the wear of unfilled PTFE samples and nanocomposites of PTFE and alumina. Based on the prior experience and finding of an environmental effect on the wear of the nanocomposites, it was hypothesized that the wear behavior of these composites would be sensitive to the vacuum environment. In contrast, previous studies with unfilled PTFE have found no clear dependence of wear on vacuum conditions, though the coefficient of friction of this material shows some dependence at elevated sliding speeds [4, 21–24].

2 Materials and Sample Preparation

The nanocomposites were prepared by using a granulated PTFE of approximately 35- μm -sized particles (DuPont Teflon[®] PTFE 7C resin). The alpha-phased alumina was distributed by Nanostructured & Amorphous Materials Inc. (Stock#: 1015WW) and specified by the distributor as having a diameter range of 27–43 nm [16]. Though the particle size of this filler was not independently verified in this investigation, the authors are aware of studies of similar particles which exhibit broad particle size distributions [25]. A powder collection of 5 wt% alumina was prepared prior to jet milling and processed as described by Burriss et al. [12]—it is of interest to note that some of the fine particles are lost during the jet milling process and the actual weight percent loadings are less than the powder mixture.

The unfilled PTFE control and the jet-milled composite powders were molded with identical procedures. Approximately 10 g of powder was packed into a 440C stainless steel cylindrical mold (76.2 mm in length, 50.8 mm in diameter) with a 12.7-mm-diameter hole through the center. Two stainless steel plugs (12.7 mm in diameter) sealed each open end of the mold; the powder was consolidated at a pressure of 100 MPa using a hydraulic press prior to sintering treatment. The mold was installed in a pneumatic press and 20 MPa was initially applied while the mold was heated from ambient temperature to 365 °C at a rate of 2 °C per minute. The polymer sample was held at a constant 365 °C for 1 hour before it was cooled to ambient temperature at a rate of 2 °C per minute.

After compression molding, the samples were machined into 6.35 mm \times 6.35 mm \times 12.7 mm pins for tribological experimentation. One 6.35 mm \times 6.35 mm end of each pin was polished using 800 grit silicon carbide sanding sheets to an average surface roughness, $R_a \sim 100$ nm; this is the face that was used for sliding experiments, and these faces were carefully cleaned, ultrasonicated in methanol for 30 minutes, and allowed to dry in laboratory air for at least 3 hours prior to experimentation.

The metal countersamples used in this investigation were lapped 304L stainless steel rectangular flat coupons (38 mm \times 25 mm \times 3.7 mm) with an average surface roughness $R_a \sim 150$ nm. These samples and their surface finish are more fully described in prior publications [6–8]. The metal samples were washed with soap and water, wiped and rinsed with methanol, and allowed to dry prior to each experiment.

3 Experimental Methods

3.1 Tribometer

A linear reciprocating tribometer housed in a custom vacuum chamber was used to perform wear and friction experiments [26] and is shown schematically in Fig. 1. The polymer sample was mounted through a loading flexure to a multi-axis force transducer (AMTI MC2.5D-100) and loaded against the metal countersample. The tribometer was located inside of a vacuum chamber connected to an oil-free dry-scroll pump (Varian Scroll Vacuum Pump) and a cryogenic vacuum pump (Cryo-Torr[®] 8 Cryopump). The vacuum pressure inside the chamber was measured by a pressure sensor (Ionivac ITR 90).

3.2 Experiments

Wear and friction experiments were conducted in a wide range of vacuum environments from ambient laboratory air to high vacuum. Four chamber pressures were targeted: ambient air at 760, $\sim 5, 1 \times 10^{-2}$, and 4×10^{-6} Torr. For ambient air experiments, the chamber was closed and sealed but not

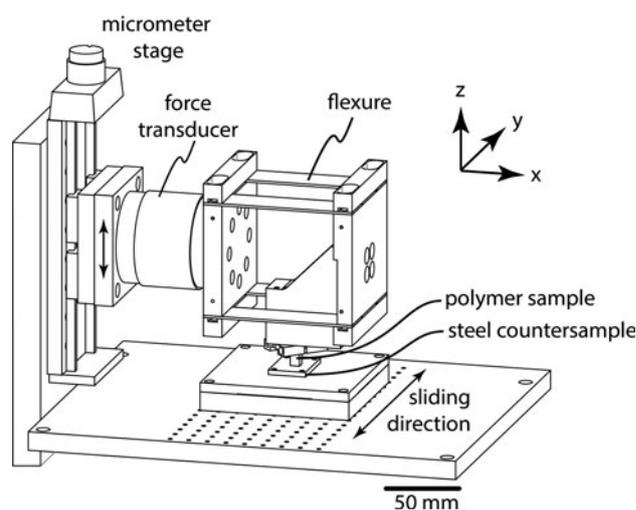


Fig. 1 The linear tribometer is housed inside of a vacuum chamber. The normal load is applied using a micrometer stage, and the sample is mounted to a flexure

pumped down. To achieve chamber pressures for the experiments targeted at ~ 5 Torr, the scroll pump was used until the chamber reached 0.1 Torr, after which the pump was turned off and the chamber was allowed to equalize at the desired pressure of 5 Torr. The scroll pump was also used to reach $\sim 1 \times 10^{-2}$ Torr. The cryopump was used to achieve pressures in the 10^{-6} Torr range. Each vacuum environment attained a relatively steady-state chamber pressure prior to tribological experimentation; maximum and minimum pressures were recorded and are represented on the graphs by uncertainty intervals.

Experiments were performed with a sliding velocity of 50.8 mm/s, a reciprocating length of 30.8 mm, and a prescribed normal force of 250 N, which yielded a nominal contact pressure of 6.25 MPa. As the polymer sample wore and deformed during sliding, the normal force gradually decreased as the load was applied through an elastic flexural spring. The normal force was continuously monitored and recorded during experiments. The greatest variation in normal load occurred with the highest wearing sample (unfilled PTFE) and decreased from 250 N to approximately 175 N. Because of the significant differences in the wear rates of these materials, the number of sliding cycles were significantly shorter for the PTFE control samples, which experienced only 10,000 cycles of sliding. The ultralow wearing PTFE and alumina composite samples had negligible deviations in applied normal load due to very small amounts of wear, but required much longer sliding experiments of 100,000 cycles to achieve low uncertainty measurements of wear.

3.3 Measurements

Wear rate, K [in $\text{mm}^3/(\text{Nm})$] is calculated as the volume of material removed during wear, V_{loss} (mm^3), divided by the product of the applied normal load, F_n (N), and the total sliding distance, d (m) (Eq. 1) [27]. For polymeric materials that can undergo shape changes not associated with wear, it is recommended to measure wear by monitoring changes in the mass of the samples. This change in mass due to wear, Δm , and the sample density, ρ , can be used to calculate the wear rate (Eq. 2).

$$K = \frac{V_{\text{loss}}}{F_n d} \quad (1)$$

$$K = \frac{\Delta m}{\rho F_n d} \quad (2)$$

For these experiments, polymer samples were weighed before and after sliding using a Mettler-Toledo balance (10 μg resolution). This preserved the integrity of each vacuum environment though limited wear rate measurements

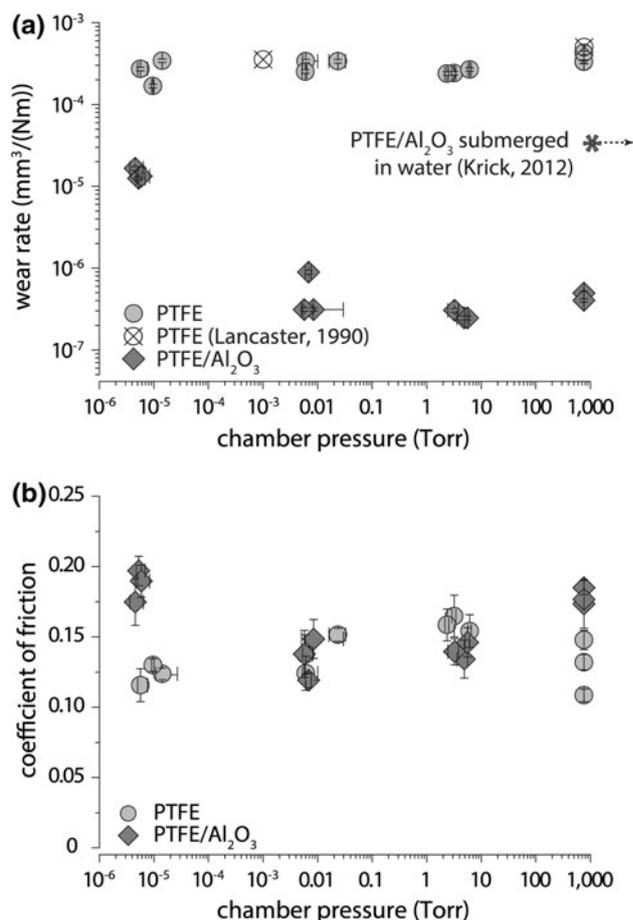
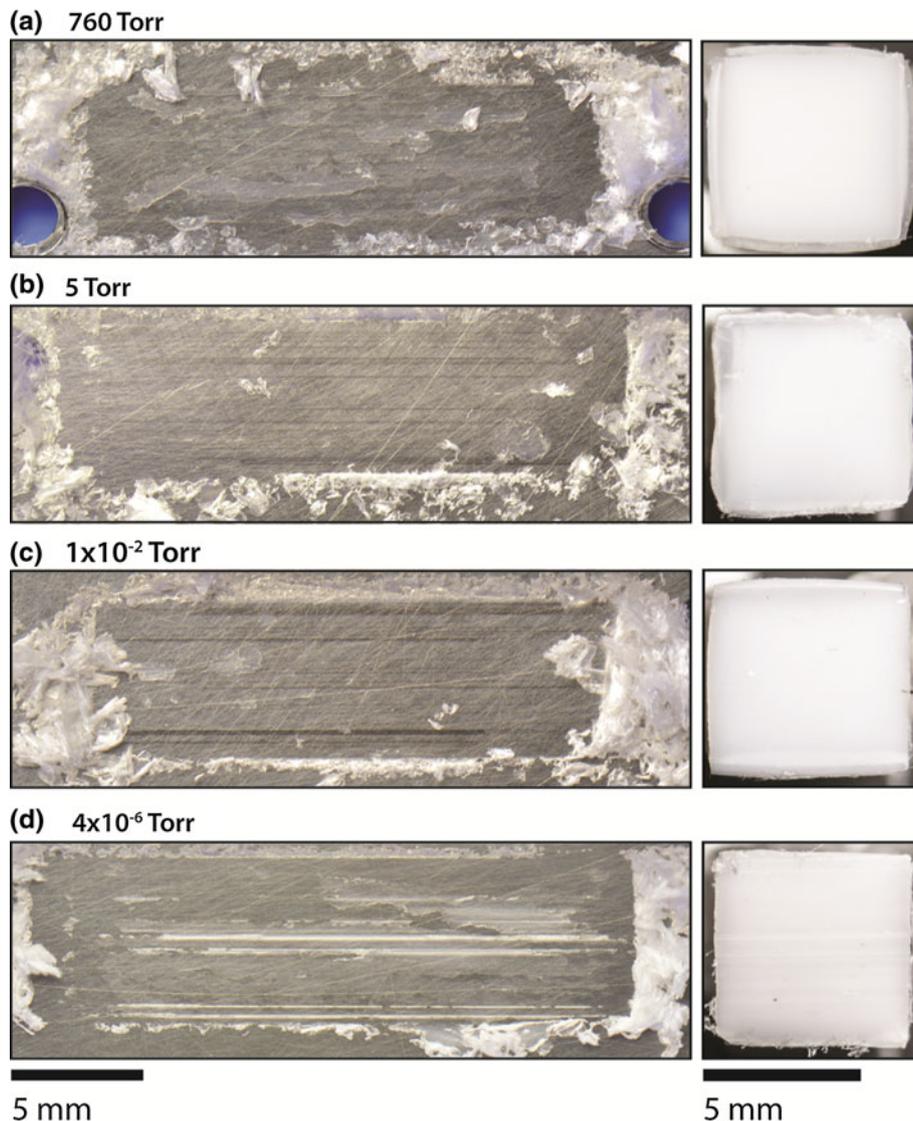


Fig. 2 a Wear rate and b coefficient of friction of PTFE control and PTFE filled with 5 wt% alumina as functions of vacuum environment, as well as wear rates of PTFE and PTFE alumina composites from previous studies [8, 32]. The unfilled PTFE samples were slid for a total of 10,000 cycles (308 m), and the PTFE composites were slid for a total of 100,000 cycles (3,080 m) under 250 N of applied normal load and 50.8 mm/s sliding speeds. The error bars in the chamber pressure correspond to the maximum and minimum vacuum pressures recorded during each experiment

to single-point wear rates. Single-point wear rates do not account for the initial transient wear behavior, or “run-in”, and thus are generally higher than those calculated using multiple mass or volume measurements that reveal a steady state; methods to compute the uncertainty in measurements follow references [28, 29].

Friction coefficients were calculated by dividing the average friction force by the average normal force, addressed in detail in previous studies [30, 31]. Uncertainty analysis associated with these measurements using a similar tribometer is delineated in Schmitz et al. [29, 30]. Accounting for friction coefficient bias due to force transducer and countersample misalignments is addressed in Burris et al. [31].

Fig. 3 Images of transfer films (*left*) and polymer wear surfaces (*right*) produced after unfilled PTFE samples were slid against stainless steel for 10,000 cycles (308 m) in the following vacuum environments: **a** 760 Torr, **b** 5 Torr, **c** 1×10^{-2} Torr, and **d** 4×10^{-6} Torr. Note that all transfer films and wear surfaces are to their respective scales



4 Results

4.1 Wear and Friction Results

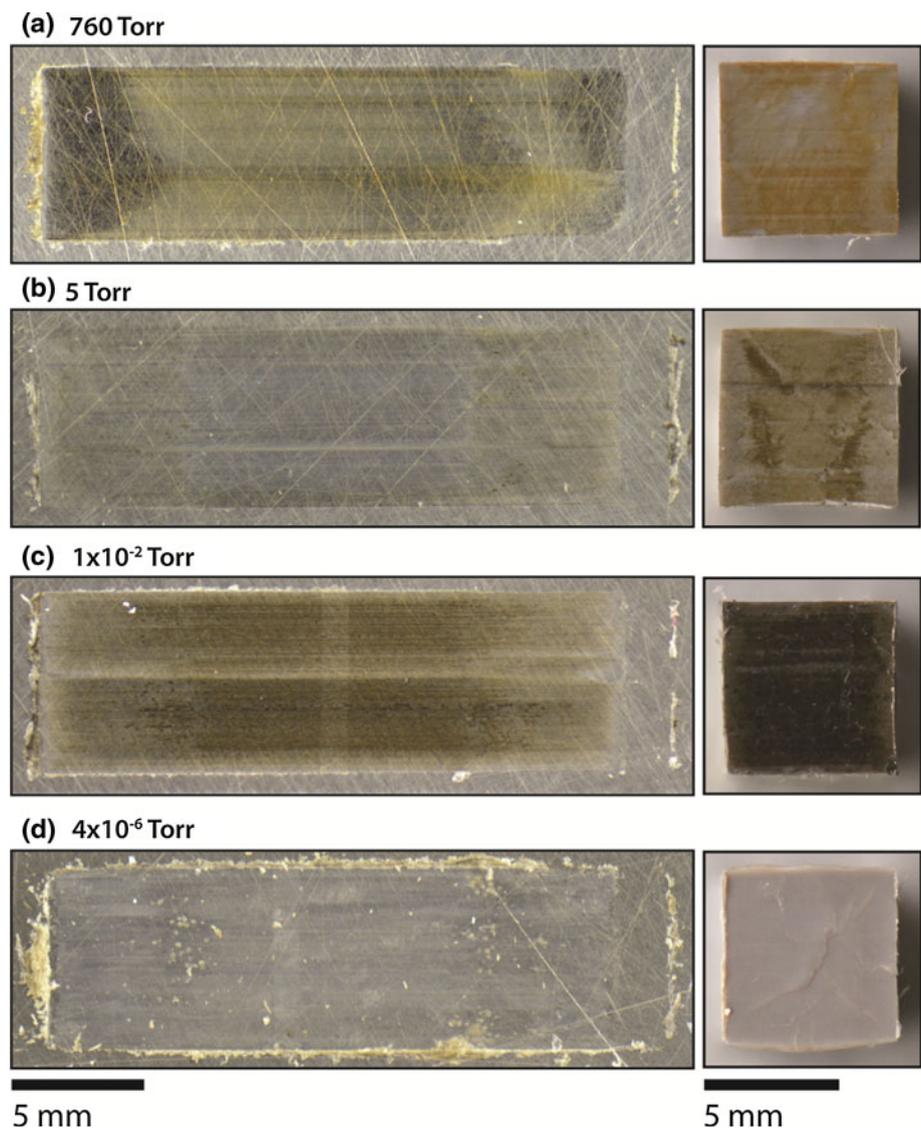
Figure 2a shows the wear rates of PTFE and PTFE/alumina composite samples sliding against stainless steel countersamples in four different nominal vacuum environments: 760, ~ 5 , $\sim 1 \times 10^{-2}$, and $\sim 4 \times 10^{-6}$ Torr. The wear of the nanocomposites show a clear dependence on the vacuum level. The highest vacuum conditions yielded the highest wear rates [$1.5 \times 10^{-5} \text{ mm}^3/(\text{Nm})$], while ambient conditions resulted in wear rates nearly two orders of magnitude lower [$\sim 4.5 \times 10^{-7} \text{ mm}^3/(\text{Nm})$], and the low and medium vacuum conditions yielded even lower wear rates [$2.5 \times 10^{-7} \text{ mm}^3/(\text{Nm})$ and $3.05 \times 10^{-7} \text{ mm}^3/(\text{Nm})$, respectively]. As previously reported, there does not appear to be a significant

relationship between wear and chamber pressure for PTFE samples although wear may be slightly higher in ambient pressures. Also shown in this figure are wear rates of PTFE in vacuum (1×10^{-2} Torr) and in ambient conditions from previous studies [32]. The effects of varying the vacuum levels on the coefficients of friction of PTFE and PTFE and alumina composite samples are illustrated in Fig. 2b.

4.2 Wear Surface Description

Images of the transfer films formed by the PTFE sample sliding against the stainless steel countersamples in ambient air (Fig. 3a) and 4×10^{-6} Torr (Fig. 3d) conditions revealed large regions of patchy, plate-like deposited PTFE. The transfer films formed at 5 Torr (Fig. 3b) and 1×10^{-2} Torr (Fig. 3c) had only sparse regions of patchy, plate-like debris

Fig. 4 Images of transfer films (*left*) and polymer wear surfaces (*right*) produced after PTFE and alumina nanocomposite samples were slid against stainless steel for 100,000 cycles (3,080 m) in the following vacuum environments: **a** 760 Torr, **b** 5 Torr, **c** 1×10^{-2} Torr, and **d** 4×10^{-6} Torr. Note that all transfer films and wear surfaces are to their respective scales



of PTFE and there appear to be scratches in the steel countersamples aligned with the sliding direction. The wear surfaces of all PTFE samples appear to be white in color with abrasions aligned in the direction of the sliding.

The transfer films formed by the PTFE and alumina nanocomposites appear brown in color in ambient air (Fig. 4a) and slightly bronze and exceptionally thin at 5 Torr (Fig. 4b). The transfer films appeared darker brown at 1×10^{-2} Torr (Fig. 4c). However, at 4×10^{-6} Torr (Fig. 4d), the transfer films appeared to be hazy white in color with light bronze clusters of debris near the reversal regions of the wear track. Tribofilms developed on the wear surface of the PTFE and alumina composites were light brown for experiments conducted in ambient conditions (Fig. 4a), slightly darker brown at 5 Torr (Fig. 4b), very dark brown at 1×10^{-2} Torr (Fig. 4c), and virtually indistinguishable from the color of the bulk polymer at 4×10^{-6} Torr (Fig. 4d).

4.3 Wear Surface Characterization

4.3.1 Stylus Profilometry

The transfer films generated by the unfilled PTFE and PTFE and alumina composites were analyzed with a Veeco Dektak 8 surface profiler using a stylus with a radius of curvature of $2 \mu\text{m}$, an applied normal load of 5 mg, and line spacing of $2.5 \mu\text{m}$. A representative region of $1 \text{ mm} \times 10 \text{ mm}$ in the center of the wear track was chosen for each scan.

For unfilled PTFE samples slid in ambient conditions (Fig. 5a), the transfer film was patchy and approximately $10 \mu\text{m}$ thick in most regions. The transfer film developed at 5 Torr was so thin that it was difficult to measure by profilometry (Fig. 5b), and at 1×10^{-2} Torr, it appeared similar to the transfer film generated in ambient conditions (Fig. 5c). At

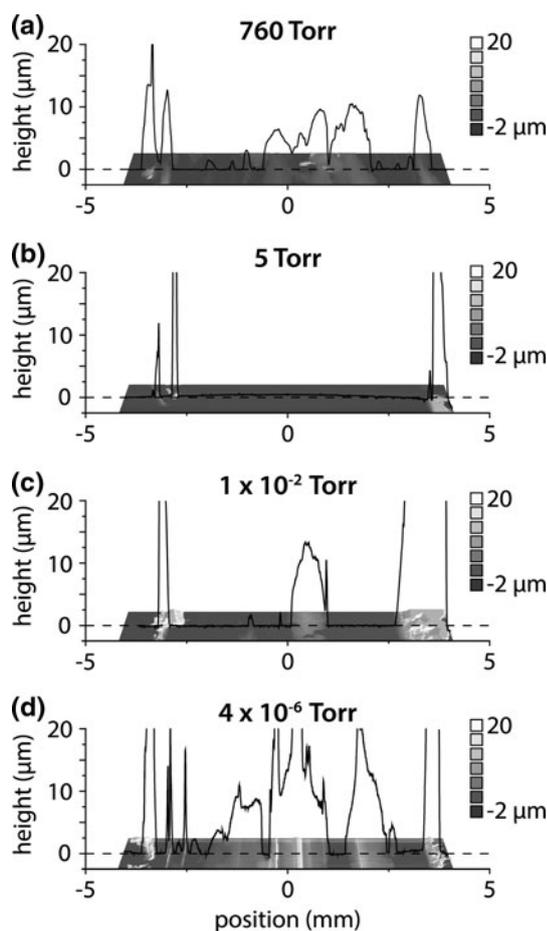


Fig. 5 Profilometry scans of the transfer films on stainless steel countersamples generated by PTFE samples. A single 2D line scan plotted over the 3D representation of the transfer film is shown for vacuum chamber pressures of **a** 760 Torr, **b** 5 Torr, **c** 1×10^{-2} Torr, and **d** 4×10^{-6} Torr

4×10^{-6} Torr (Fig. 5d), the transfer film was very thick and patchy, and exceeded $10 \mu\text{m}$ in thickness in some regions.

Profilometry scans of PTFE and alumina composites revealed thin, uniform transfer films formed in ambient air (Fig. 6a) and 5 Torr (Fig. 6b), and slightly thicker transfer films under 1×10^{-2} Torr (Fig. 6c). At 4×10^{-6} Torr, the transfer film of PTFE and alumina composites appeared patchy (Fig. 6d), had a maximum transfer film thickness of approximately $3 \mu\text{m}$, and was accompanied by an increase in wear rate of two orders of magnitude.

4.3.2 Scanning Electron Microscopy

Backscattered electron micrographs reveal differences in transfer film characteristics and coverage. Despite the low variation in wear rate with vacuum chamber pressure, there are still noticeable differences between the tribofilms formed by unfilled PTFE. Unfilled PTFE in ambient

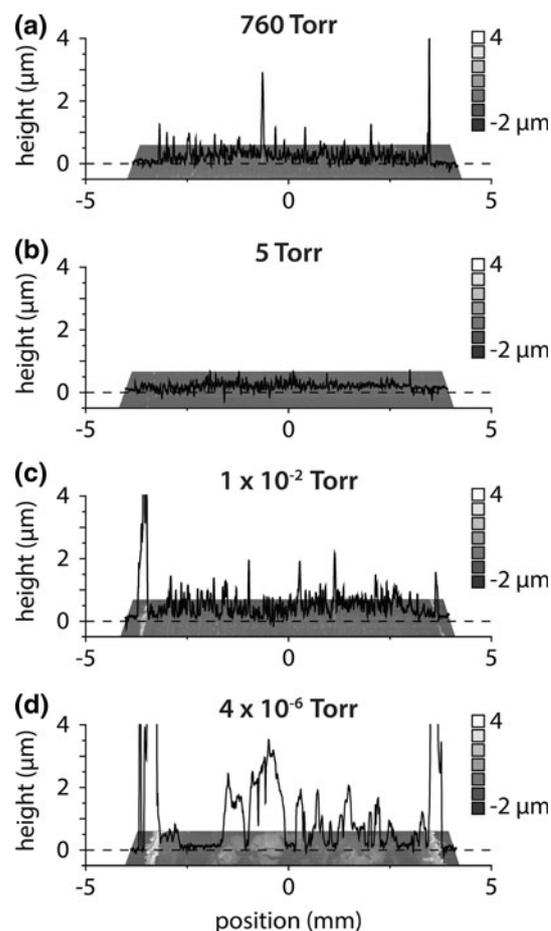


Fig. 6 Profilometry scans of the transfer films on stainless steel countersamples generated by PTFE and alumina samples. A single 2D line scan plotted over the 3D representation of the transfer film is shown for vacuum chamber pressures of **a** 760 Torr, **b** 5 Torr, **c** 1×10^{-2} Torr, and **d** 4×10^{-6} Torr

conditions (Fig. 7a, e) and in high vacuum (Fig. 7d, h) formed thick, patchy transfer films and experienced lower friction than in intermediate conditions (Fig. 7b–g). Interestingly, the nanocomposite experienced higher friction in ambient conditions (Fig. 7a, e) and high vacuum (Fig. 7d, h) than in intermediate conditions.

The PTFE and alumina nanocomposite formed smaller islands of transferred material than unfilled PTFE, and the transfer films are shown to be more uniform. At ~ 5 Torr, the vacuum chamber pressure at which the wear rate of the nanocomposite was lowest, the transfer film was thinnest and quite uniform. In high vacuum, 4×10^{-6} Torr, the wear rate of the nanocomposite was highest and the transfer film was thickest and most closely resembled that of the less wear-resistant unfilled PTFE. In all cases, it appeared that the polymers tended to fill in any polishing scratches on the stainless steel countersamples in addition to forming transfer films.

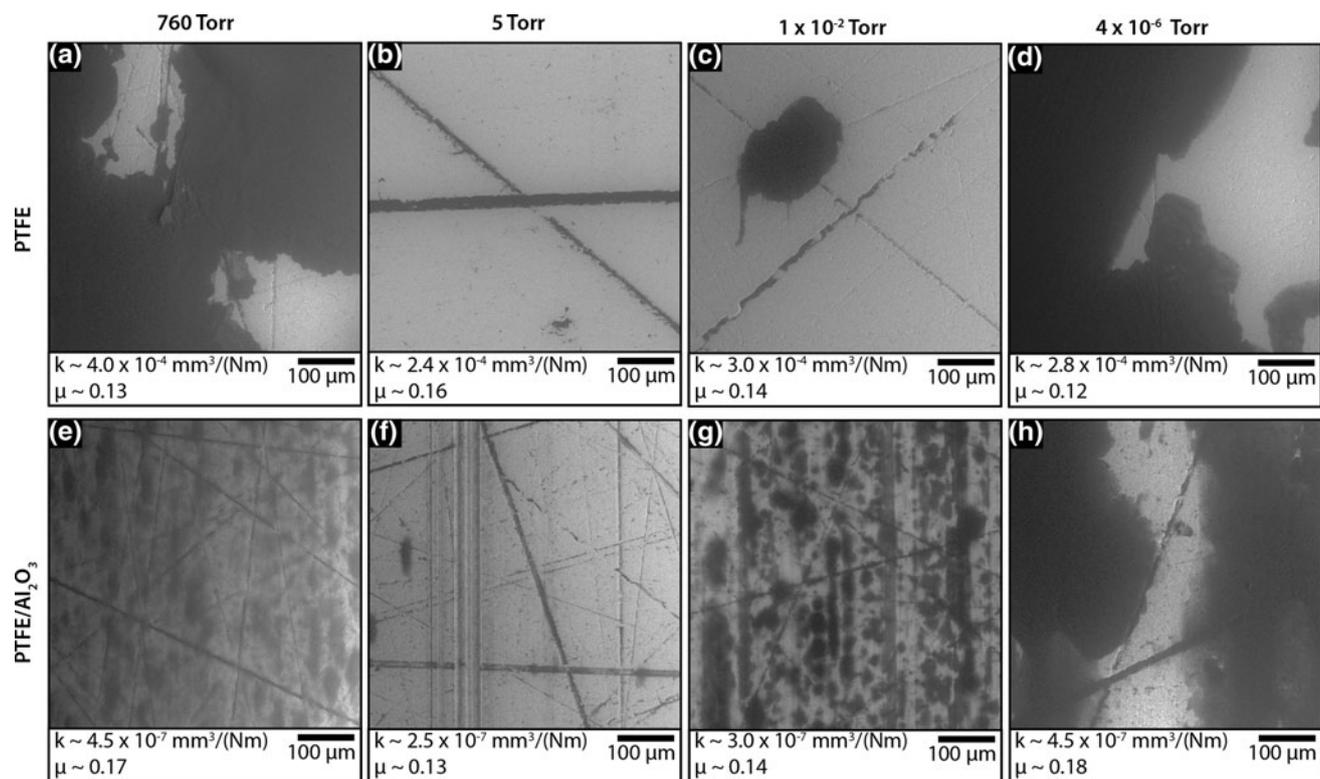


Fig. 7 Backscatter electron micrographs of transfer films formed on 304L stainless steel from **a–d** unfilled PTFE and **e–h** PTFE filled with 5 wt% alumina samples in the following vacuum conditions:

a, e 760 Torr, **b, f** 5 Torr, **c, g** 1×10^{-2} Torr, and **d, h** 4×10^{-6} Torr. Note that *dark regions* are polymeric material

5 Discussion

5.1 Tribofilms and Wear Performance of PTFE and Alumina Composites

In the atmospheric environment, a well-developed, brown, robust transfer film with very little wear debris is easily observed; this is consistent with many previous studies of this material [6–15]. In this case, as well as the cases for other favorable environments (5 and 1×10^{-2} Torr), a new sliding interface is generated in which PTFE and alumina no longer slides on steel. Instead, the interface is self-mated tribochemically degraded PTFE/alumina composite on composite transfer film. When comparing the wear rates of the same material in different vacuum environments, thinner transfer films correlate with lower wear rates (Fig. 8). It is hypothesized that the formation of this thin transfer film changes the sliding interface to be polymer composite sliding against polymer composite transfer film; this protects the composite and the metal counter-sample from each other. The formation of these thin, protective transfer films is dependent upon a variety of factors, including the chemical constituents of the sliding environment [8, 13, 15, 33].

5.2 Link Between Vacuum Environment and Wear of PTFE and Alumina Composites

There is a clear trend between wear rate and vacuum chamber pressure for PTFE and alumina nanocomposites. It is evident that the remarkable reductions in wear achieved by these composites are dependent upon a tribochemical mechanism involving constituents of the composite and the environment. PTFE and alumina composites slid against stainless steel displayed consistently ultralow wear behavior in ambient conditions [6–15, 19]; the present study suggests that in laboratory air, there is sufficient moisture at the sliding interface to assist the formation of a tribochemically generated, robust, protective transfer film [8].

At 4×10^{-6} Torr, there are substantially fewer environmental constituents to participate in tribochemical interactions, making conditions less favorable for tribofilm formation. This results in a significantly higher wear rate of the PTFE and alumina composite. It is estimated that the time to form a monolayer of water at 4×10^{-6} Torr is ~ 0.6 s [34–36], which is nearly identical to the duration of one half of a reciprocating stroke. This suggests that there is insufficient water present at the sliding interface at 4×10^{-6} Torr to promote the formation of tribofilms

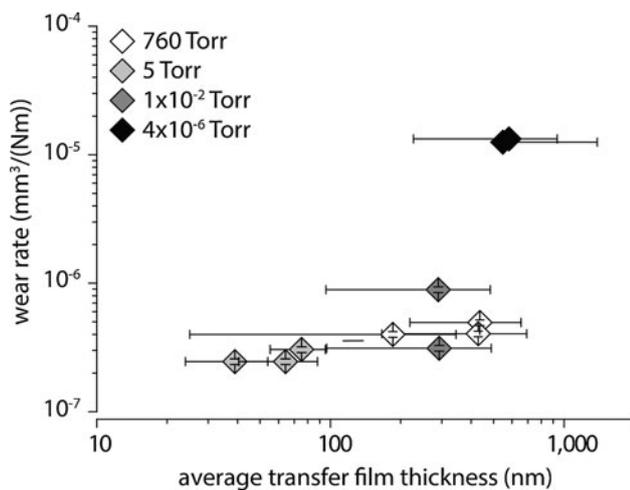


Fig. 8 Wear rate of PTFE and alumina nanocomposites as a function of resulting transfer film thickness over several vacuum chamber pressures. Standard deviation of transfer film height is represented by error bars. Note that one of the 4×10^{-6} Torr data points has a standard deviation greater than the limits of the log scale. PTFE and alumina samples were slid for a total of 100,000 cycles (3,080 m) under 250 N of applied normal load and 50.8 mm/s sliding speeds

observed in more favorable conditions, resulting in higher wear rates.

It is intriguing that the wear of PTFE and alumina composites is at a minimum in environments between humid air and high vacuum. The average wear rate of PTFE and alumina composites was approximately $2.7 \times 10^{-7} \text{ mm}^3/(\text{Nm})$ at ~ 5 Torr and $4.3 \times 10^{-7} \text{ mm}^3/(\text{Nm})$ at atmospheric pressure with standard deviations in wear rate of 3.4×10^{-7} and $5.3 \times 10^{-7} \text{ mm}^3/(\text{Nm})$, respectively (standard deviation divided by average values was ~ 0.12 for both environment datasets); while the wear rate at 5 Torr was $\sim 40\%$ lower than wear in humid air, there may be a statistically significant difference. If so, it is possible that these environments of ~ 5 Torr are slightly more favorable to facilitate low wear behavior. In a recent paper, Krick et al. measured wear of PTFE and alumina nanocomposites in controlled gas environments while varying environmental humidity [8]. While this study did not provide definitive conclusions, experiments conducted in pure nitrogen environments resulted in lower wear rates than those in air for all humidity environments studied. This suggests that oxygen partial pressures may impact wear performance. At the vacuum levels used, trace oxygen and other gases may still be present. Furthermore, if water is the only environmental species required to allow the tribochemistry for ultralow wear PTFE composites, one might expect exceptional results for the wear when sliding in a submerged water environment; however, a prior study

showed that a PTFE/alumina composite sliding in liquid water resulted in a high wear rate of $3.6 \times 10^{-5} \text{ mm}^3/(\text{Nm})$. This wear rate is comparable to the wear performance of this composite in high vacuum [8]. This suggests that the presence of liquid water interrupts the tribochemical wear mechanisms and/or another species present in air required for the ultralow wear performance to be achieved, even if said species is in trace amounts.

This new understanding of competitive environmental species may explain wear at a $\sim 1 \times 10^{-2}$ Torr vacuum environment. At this vacuum pressure, it is postulated that water vapor constitutes at least 98 % of the total pressure [34]. A hypothesis on the importance of water has already been highlighted and is further supported by the ultralow wear behavior in this environment. Furthermore, one of three experiments conducted at $\sim 1 \times 10^{-2}$ Torr resulted in higher wear [$\sim 9 \times 10^{-7} \text{ mm}^3/(\text{Nm})$] than the other two [$\sim 3 \times 10^{-7} \text{ mm}^3/(\text{Nm})$] in approximately the same environment; this suggests that this vacuum pressure is perhaps on the cusp of favorable conditions for ultralow wear.

The lowest wear of this material appeared at 1–10 Torr. It is likely that nitrogen and oxygen are removed at more favorable rates than water when pumping to achieve a chamber pressure of 1–10 Torr. However, it is not until $\sim 1 \times 10^{-2}$ Torr that the environment is considered to be mostly water [34]. This minimum suggests that while oxygen may negatively compete in humid air [8], there may be an optimal concentration of oxygen for wear reduction. Current results show that optimum oxygen concentration may result in optimal transfer film formation, lower friction coefficients, and lower wear rates that persist through medium vacuum chamber pressures. While the mechanism associated with oxygen is still unclear, oxygen's possible role in transfer film formation is becoming evident. Oxidized transfer films have been linked with ultralow wear behavior of this material [8, 16], and vacuum experiments reveal minimum wear and friction at 5 and 1×10^{-2} Torr. Unfortunately, it is difficult to draw definitive conclusions as to the exact environmental constituents present at each vacuum chamber pressure without the use of advanced gas characterization.

The balance between H_2O and O_2 gas species and their effects on tribofilm formation and thus wear performance of the same material is clearly a part of the delicate mechanisms in which low concentrations of alumina can reduce the wear of PTFE. The complex nature of these interfacial interactions suggests that wear of these materials is system-dependent. This necessitates future wear studies to evaluate dependencies in wear of this material on sliding velocity, contact pressure, countersample material, sliding geometry, and other factors.

6 Conclusions

The wear of unfilled PTFE samples remained largely unaffected in various vacuum environments. The wear of the nanocomposites of PTFE and alumina was highly sensitive to the vacuum level: experiments at 1×10^{-2} , 1–10 Torr, and in ambient air resulted in ultralow wear performance. The wear rate was lowest [$2.5 \times 10^{-7} \text{ mm}^3/(\text{Nm})$] in the 1–10 Torr environment. The wear rate in the 4×10^{-6} Torr environment was orders of magnitude higher than other environments at $1.5 \times 10^{-5} \text{ mm}^3/(\text{Nm})$.

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