



Tribological Sensitivity of PTFE/Alumina Nanocomposites to a Range of Traditional Surface Finishes

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Wear tests were performed with polytetrafluoroethylene (PTFE) + Al₂O₃ nanocomposites on various manufactured surfaces to determine whether or not the wear resistance of these nanocomposites is a strong function of surface preparation. Four different surface finishes of grade 304 stainless steel counterfaces were used: electropolished (R_q = 88 nm), lapped (R_q = 161 nm), wet-sanded (R_q = 390 nm), and dry-sanded (R_q = 578 nm). PTFE + Al₂O₃ nanocomposites made from powders of roughly 2-20 μm PTFE (matrix) and ~44 nm Al₂O₃ (filler) were prepared at filler weight percentages of 0, 1, 5, and 10% and tested on each surface finish. Additionally, 5 wt% 44-nm nanocomposites were compared to identically prepared 5 wt% 80- and 500-nm Al₂O₃ filled PTFE composites on each surface. Friction coefficients were between 0.12 and 0.19 and wear rates decreased from K = 810 × 10⁻⁶ mm³/(Nm) for the 5 wt% 500-nm alumina-filled PTFE on the dry-sanded surface to K = 0.8 × 10⁻⁶ mm³/(Nm) for the 5 wt% 80-nm filled composite on the lapped surface. It was found that the minimum wear rate occurred on the lapped counterface for every composite, and the wear rate is a strong function of the transfer film thickness and morphology.

KEY WORDS

Solid Lubricants; Nanocomposites; Friction and Wear; Surface Roughness

INTRODUCTION

Polytetrafluoroethylene (PTFE) exhibits many desirable tribological characteristics, including low friction, high melting temperature, and chemical inertness; it receives much attention for use as a solid lubricant. Although it offers a low friction coefficient in most conditions, PTFE wears rapidly compared to other polymers, precluding its use as a bearing material in many cases.

Significant amounts of research have been done to improve the wear resistance of PTFE without sacrificing its low friction capability. Just about everything put into PTFE generally improves the wear resistance at the expense of friction coefficient, including nanoparticles. Although there are many theories for the role of filler particles and fibers in tribological composites, there was a general speculation that nanofillers could not prevent large-scale destruction of the banded structure of PTFE and would be transferred with the PTFE onto the counterface (Tanaka and Kawakami (1)). The qualitative models suggest that the asperity peaks would dominate when their heights are larger than the filler particles. During this study, both the filler particle size and the surface roughness will be varied without changing material composition.

Within the tribology literature of polymer nanocomposites, wear rate and friction coefficient are generally reduced with the addition of nanoscopic filler particles for all polymer matrices. Xue, et al. explored the benefits of using nanometer-sized particles instead of micrometer-sized particles in a PEEK-SiC composite, which resulted in reduced friction coefficient and wear rate over composites made with filler particles of the same composition with characteristic sizes of micrometers (Xue and Wang (2)). The micrometer-sized SiC abraded both the polymer transfer film and the metallic counterface, resulting in increased friction and wear. Wang et al. investigated the tribology of nanometer SiO₂-filled PEEK (Wang, et al. (3)), as well as ZrO₂-filled PEEK (Wang, et al. (4)) on carbon steel, finding the lowest wear rates at 7.5 filler wt% for both composites. Furthermore, friction coefficients dropped with increasing filler percentages for the range tested (0-15%).

In the area of PTFE-based nanocomposites, Chen, et al. (5) created a single-walled carbon-nanotube filled PTFE composite that improved wear resistance by more than two orders of magnitude over unfilled PTFE and had limited reductions in friction coefficient. The carbon nanotubes were thought to strengthen the matrix, reducing wear of the PTFE. They hypothesized that the carbon nanotubes provided additional self-lubrication of the composites by breaking off, becoming trapped in the contact, and separating the surfaces. Li, et al. (6) filled PTFE with nanometer-sized ZnO and found 15 wt% (weight percent of fillers) to be the optimal filler content for reducing wear rate while retaining a low friction coefficient. Sawyer, et al. (7) made nanocomposites of PTFE with

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38-nm Al_2O_3 and found a 600-fold reduction in wear with 20 wt% filler concentration. Wear reduced as filler concentration increased to 20 wt%. It was hypothesized that the PTFE particles were decorated by the nanoscopic alumina during a powder blending process that preceded compression molding. The resulting structure after molding is cellular with thin regions of highly concentrated PTFE-alumina material surrounding micrometer-sized domains of PTFE. These concentrated regions act as barriers to crack propagation and reduce the delamination wear of PTFE. Furthermore, it was offered that, with increasing filler concentration, the number, size, and possibly strength of the compartmentalizing regions increased.

Past work with polymers and micron-sized composites shows that changes in counterface surface roughness can trigger dramatic changes in the wear rates. Franklin and de Kraker (8) investigated the effects of countersurface topography on a commer-

cial POM/20% PTFE composite and found that increased roughness led to increased wear only when the lay was perpendicular to the direction of sliding, and when the roughness was greater than $\sim 1 \mu\text{m}$. Wieleba (9) studied the effect different counterface roughness parameters had on the wear and friction of carbon- and graphite-filled PTFE composites and found that the shape of the asperities had the greatest influence on friction, whereas the size of the asperities had the greatest effect on wear. Bahadur and Tabor (10) hypothesized that asperity slope and radius of curvature govern the wear rate of PTFE.

In this study, four surfaces were finished using standard techniques and tribological experiments were conducted with PTFE + Al_2O_3 nanocomposites similar to those reported earlier (Sawyer, et al. (7)) to explore the effect of surface finish on the tribological behavior of these composites and to gain further insight into PTFE composite wear mechanisms.

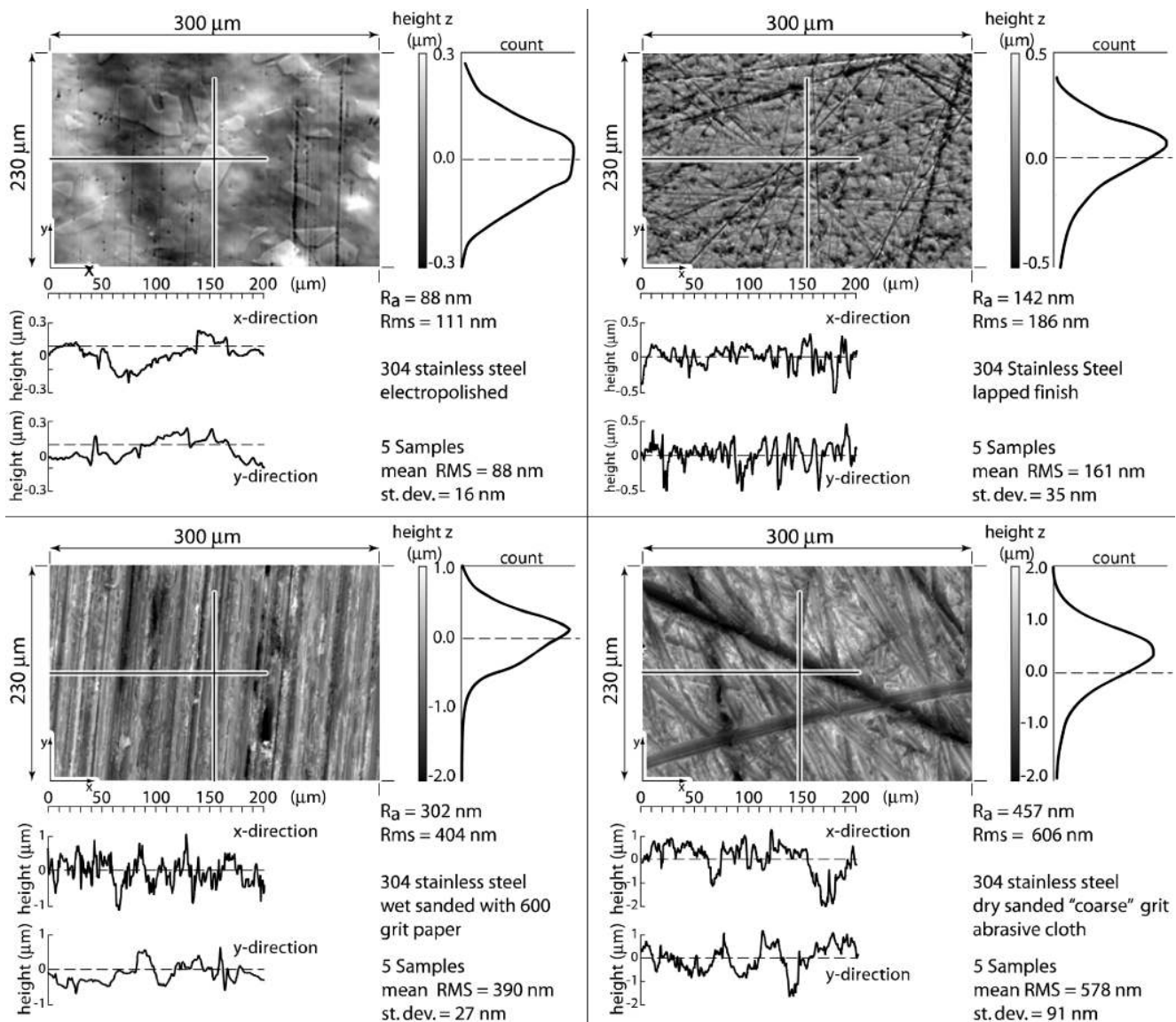


Fig. 1—Surface profilometry data from a scanning white-light interferometer with a $20\times$ objective, clockwise from top left: electropolished, lapped, dry-sanded, and wet-sanded surfaces.

TABLE 1—TRIBOLOGICAL RESULTS OF AN EXPERIMENTAL MATRIX THAT USES A NEW COUNTERFACE AND INDIVIDUALLY MADE COMPOSITE SAMPLES WITH AN ALUMINA FILLER PARTICLE SIZE OF 44 NM

Counterface preparation	Weight Percent of 44-nm alumina filler			
	0%	1%	5%	10%
Electropolished	$K = 695$	$K = 174$	$K = 52$	$K = 75$
	$\mu = 0.165$	$\mu = 0.175$	$\mu = 0.191$	$\mu = 0.178$
Lapped	$\langle K \rangle_6 = 586$	$K = 89$	$\langle K \rangle_4 = 66$	$K = 38$
	$\langle \mu \rangle_6 = 0.136$	$\mu = 0.174$	$\langle \mu \rangle_4 = 0.173$	$\mu = 0.184$
Wet-sanded	$K = 770$	$K = 85$	$K = 99$	$K = 50$
	$\mu = 0.135$	$\mu = 0.172$	$\mu = 0.162$	$\mu = 0.163$
Dry-sanded	$K = 634$	$K = 293$	$K = 294$	$K = 97$
	$\mu = 0.142$	$\mu = 0.159$	$\mu = 0.145$	$\mu = 0.183$

The $\langle \rangle_n$ represents mean values over n experiments; otherwise only a single experiment was run. The units on wear rate K are $\times 10^{-6} \text{ mm}^3/(\text{Nm})$.

TABLE 2—TRIBOLOGICAL RESULTS OF AN EXPERIMENTAL MATRIX THAT USES A NEW COUNTERFACE AND INDIVIDUALLY MADE COMPOSITE SAMPLES WITH AN ALUMINA FILLER PARTICLE NOTED

Counterface preparation	Size of PTFE filler in 5 wt% composites			
	Unfilled	44 nm	80 nm	500 nm
Electropolished	$K = 695$	$K = 52$	$K = 0.80$	$K = 70.3$
	$\mu = 0.165$	$\mu = 0.191$	$\mu = 0.191$	$\mu = 0.152$
Lapped	$\langle K \rangle_6 = 586$	$\langle K \rangle_4 = 66$	$K = 0.84$	$K = 47.4$
	$\langle \mu \rangle_6 = 0.136$	$\langle \mu \rangle_4 = 0.173$	$\mu = 0.158$	$\mu = 0.174$
Wet-sanded	$K = 770$	$K = 99$	$\langle K \rangle_2 = 8.74$	$K = 64.9$
	$\mu = 0.135$	$\mu = 0.162$	$\langle \mu \rangle_2 = 0.151$	$\mu = 0.168$
Dry-sanded	$K = 634$	$K = 294$	$K = 0.95$	$\langle K \rangle_2 = 664$
	$\mu = 0.142$	$\mu = 0.145$	$\mu = 0.141$	$\langle \mu \rangle_2 = 0.145$

Each composite is 5 wt% filler, balance PTFE. The $\langle \rangle_n$ represents mean values over n experiments; otherwise only a single experiment was run. The units on wear rate K are $\times 10^{-6} \text{ mm}^3/(\text{Nm})$.

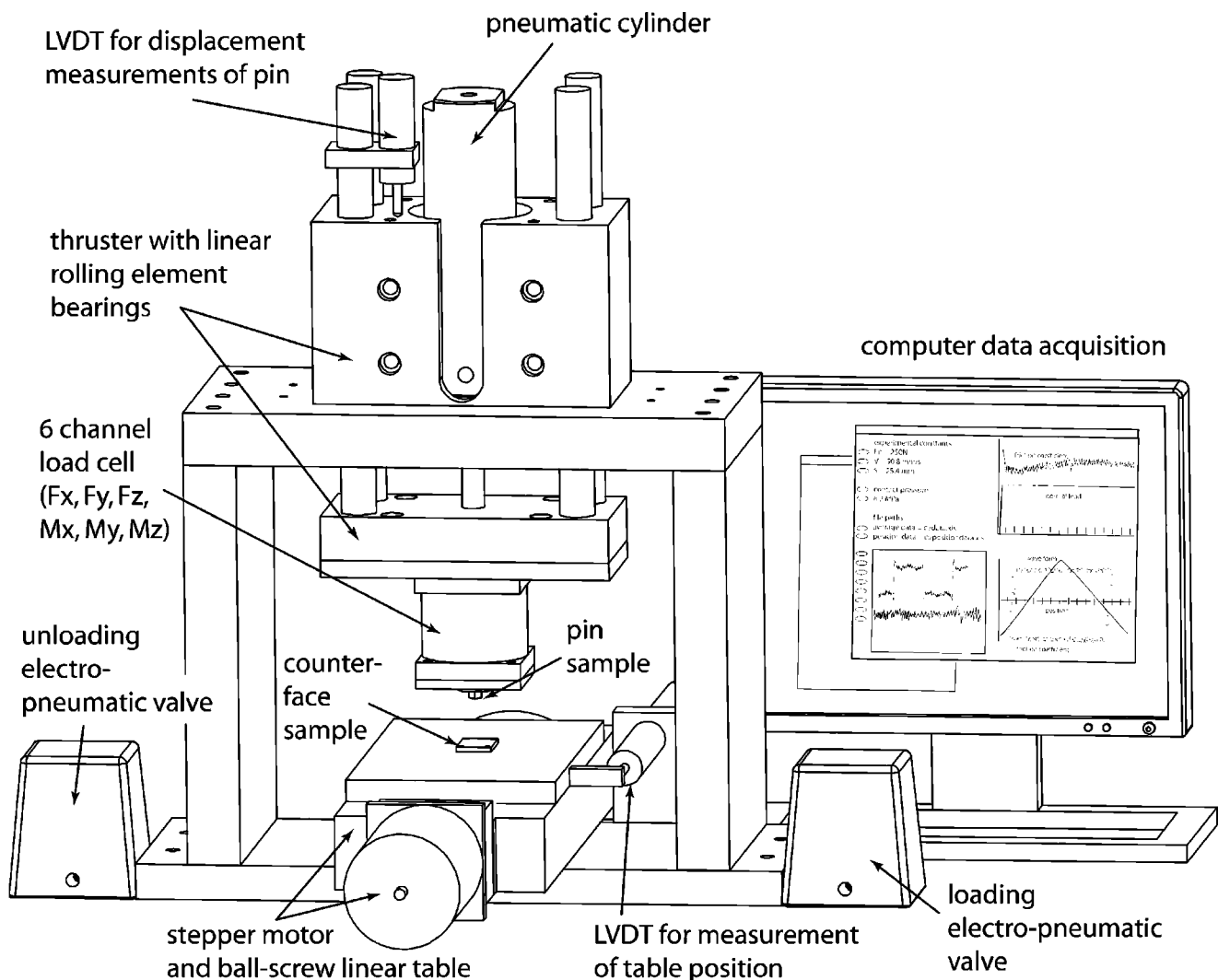


Fig. 2—Tribometer used for friction and wear testing.

EXPERIMENTAL DETAILS

Wear and friction tests were performed on PTFE composites against counterfaces of varying surface finishes. Grade 304 stainless steel counterfaces, measuring 38 mm × 25.4 mm × 3.4 mm were used in these wear tests. This material has a measured Rockwell B hardness of 87 kg/mm². The surfaces were prepared using four different traditional finishing processes: electropolishing, lapping, wet-sanding, and dry-sanding. The electropolished samples were prepared by wet-sanding with 600-grit silicon carbide paper, followed by lapping, and finished by electropolishing. Similarly, the lapped samples were initially wet-sanded with the 600-grit silicon carbide paper and then lapped. The wet-sanded samples were only exposed to the 600-grit silicon carbide paper. The dry-sanded samples were initially wet-sanded and then roughened with 80-grit "coarse" silicon carbide paper. The samples were examined under a scanning white-light interferometer with a 20× objective. Areas of 230 μm × 300 μm were measured on five different samples from each batch. A grayscale contour plot with accompanying line scans, amplitude parameters R_a (average roughness) and R_q (root mean squared roughness), and histograms of the surfaces are shown in Fig. 1. For these surfaces, the average R_q and standard deviation among five samples (σ) was $R_q = 88$ nm, $\sigma = 16$ nm; $R_q = 161$ nm, $\sigma = 35$ nm; $R_q = 390$ nm, $\sigma = 27$ nm; and $R_q = 578$ nm, $\sigma = 91$ nm for the electropolished, lapped, wet-sanded, and dry-sanded surfaces, respectively.

Four PTFE-based nanocomposites, of composition 0, 1, 5, and 10 wt% 44-nm Al₂O₃, were processed following the same approach as reported by Sawyer, et al. (7). Briefly, the procedure involves blending the appropriate masses of constituent powders using a jet-milling apparatus. The mixtures were then compression molded, machined, measured, and weighed; density of the sample was calculated from these measurements. The sample sizes are 6.35 mm × 6.35 mm × 12.7 mm and tests are run against the square face with an average contact pressure of 6.2 MPa. Results and organization of the experiments that vary surface roughness and filler loading, holding particle size constant, are shown in Table 1. The experimental design consists of four counterface conditions and four particle loadings with repeat tests of unfilled PTFE and 5 wt% filled PTFE on the lapped counterface; this provides some indication of scatter in the data from processing variations. Experimental variations and uncertainties are discussed in detail by Schmitz, et al. ((11), (12)).

It was hypothesized that increases in the nondimensional roughness parameter R_q/D_f (the ratio of the root-mean-squared roughness to the characteristic filler diameter) would increase the wear rate. Although this parameter can be easily varied by changing only the surface roughness, another series of experiments described here varies the filler particle size as well. All of these composites were processed identically. The experimental design and results of varying filler particle size and roughness, holding filler loading constant, are given in Table 2.

A linear reciprocating tribometer was used and is shown schematically in Fig. 2. This tribometer is described in much more detail in the literature (Sawyer, et al. (7); Schmitz, et al. (11), (12)). Prior to testing, the counterfaces were washed with soap and water, cleaned with acetone, sonicated for ~15 min in methanol, and then dried. The nanocomposites were wiped down with methanol

but were not washed or sonicated. A normal force of 250 N was continuously monitored and controlled. The normal load, friction force, stroke, and pin displacement were continuously measured and recorded using a data acquisition system. The reciprocating length was 25.4 mm with an average sliding speed of 50.8 mm/s. The total sliding distance depended on the wear resistance of the sample but was generally on the order of 500 m. The entire apparatus is located inside a soft-walled cleanroom with conditioned laboratory air of relative humidity between 25 and 50%.

RESULTS AND DISCUSSION

Varying Filler Loading and Surface Finish at Constant Filler Diameter

The average friction coefficient data for these experiments are plotted in Fig. 3 versus filler weight percent. The error bar on a coefficient of friction data point is the standard deviation of the friction coefficient data collected during the entire test. It is encouraging to note that the friction coefficient is not greatly affected by changes in composition or surface roughness; it has the general trend of increasing slightly with increasing filler concentration and decreasing surface roughness. Friction is thought to increase with the addition of the ceramic particles because the filler and counterface make a higher friction pair than the PTFE and counterface.

The wear rates and uncertainties for these experiments were calculated using single-point measurement of mass loss at the conclusion of the test; numerous interrupted experiments were conducted to support the reasonableness of this method (i.e., the interrupted measurements showed a linear trend of volume lost with sliding distance). These data are plotted in Fig. 4 versus filler weight percent. The error bar on a wear rate data point is the

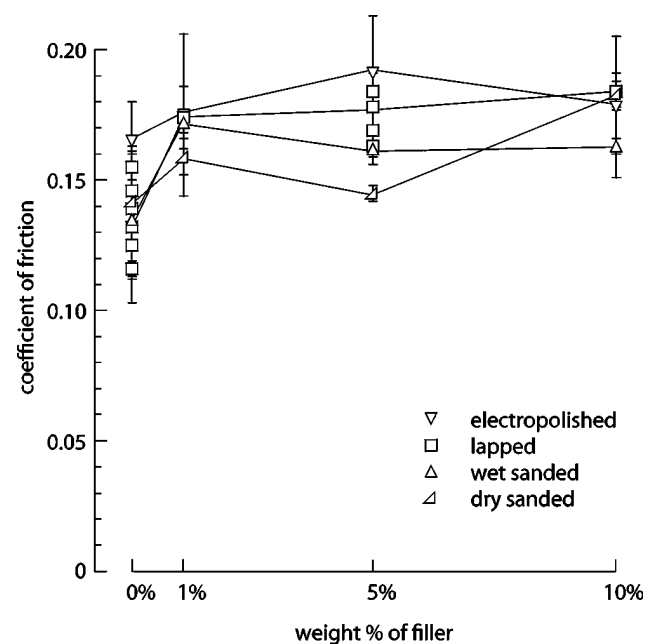


Fig. 3—Average friction coefficients of all 44-nm composites plotted versus weight percent of filler particles. The normal load was 250 N and the sliding speed was 50 mm/s (the sliding distances varied). The error bars represent the standard deviation of the friction coefficient during each test.

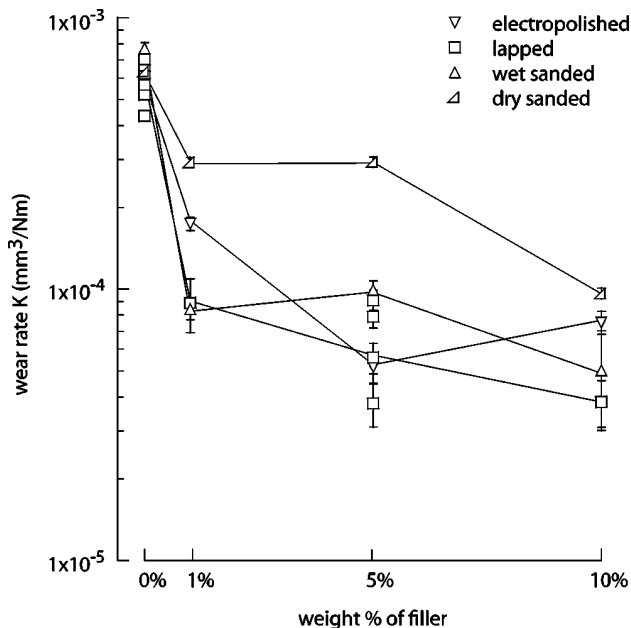


Fig. 4—Average wear rates of all 44-nm composites plotted versus weight percent of filler particles. The normal load was 250 N and the sliding speed was 50 mm/s (the sliding distances varied). The error bars represent the standard uncertainty of the measurement.

standard uncertainty of this measurement (Schmitz, et al. (12)). The wear rate decreased monotonically with increasing filler content. Figure 5 is a graph of the wear rate data plotted versus dimensionless roughness: R_q/D_f . There is no relationship between wear rate and normalized counterface roughness, but the wear rate

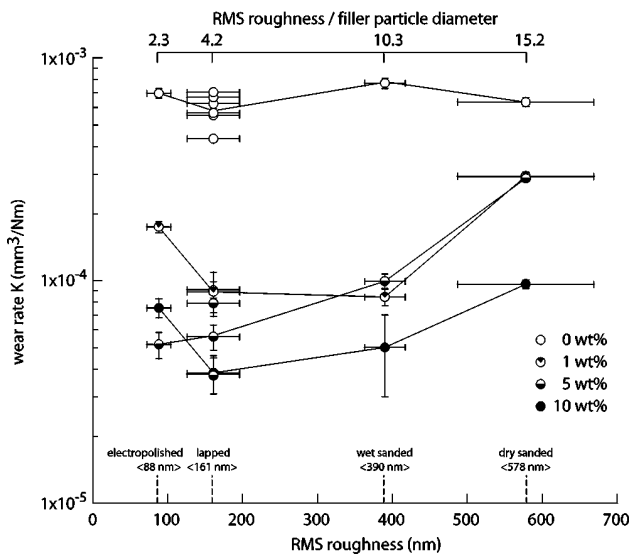


Fig. 5—Average wear rates of the 44-nm nanocomposites plotted versus the roof-mean-square (RMS) roughness of the counterfaces and R_q/D_f . The normal load was 250 N and the sliding speed was 50 mm/s (the sliding distances varied). The vertical error bars represent the standard uncertainty of the measurement and the horizontal error bars represent the standard deviation of the RMS roughness over five samples.

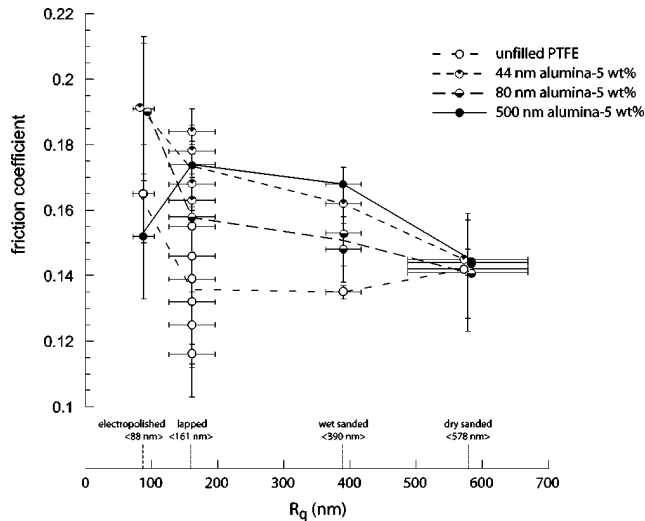


Fig. 6—Average friction coefficients of the composites plotted versus the RMS roughness of the counterfaces. The normal load was 250 N and the sliding speed was 50 mm/s (the sliding distances varied). The vertical error bars represent the standard deviation of the measurements and the horizontal error bars represent the standard deviation of the RMS roughness over five samples.

appears to be minimized on the lapped counterface ($R_q/D_f \sim 4$). This apparent optimum may be due to filler particles mechanically engaging the valleys of the negatively skewed lapped counterface. Scanning white-light interferometric examination of the counterfaces revealed a trend of decreasing debris size and thinner, more uniform transfer films with increasing filler loading and decreasing wear rates.

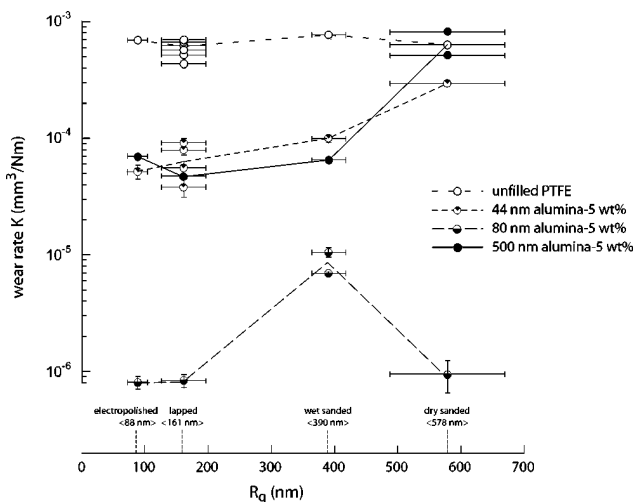


Fig. 7—Average wear rates of the composites plotted versus the RMS roughness of the counterfaces. The normal load was 250 N and the sliding speed was 50 mm/s (the sliding distances varied). The vertical error bars represent the standard uncertainty of the measurement and the horizontal error bars represent the standard deviation of the RMS roughness over five samples.

Filler accumulation at the sliding interface should result in a wear rate that decreases with increasing sliding distance. The wear volume of this particular nanocomposite is a linear function of the product of normal load and sliding distance, which suggests that filler accumulation at the interface is not the wear-reduction mechanism. The linear trend of this material suggests that the surface composition is near steady state at the onset of sliding.

Varying Filler Size and Surface Finish at Constant Filler Loading

The average friction coefficient data with error bars calculated as discussed previously are plotted versus counterface R_q in Fig. 6 and are given in Table 2. No conclusive trends in the friction coefficients of the 44-, 80-, and 500-nm composites were observed. Unlike the previous study, steady-state wear rates were calculated

from interrupted mass measurements because the 80-nm composites exhibited a transient region of substantially higher wear rate. This method of calculating wear rate and uncertainty is described in detail by Schmitz, et al. (12).

The results of these wear tests are plotted in Fig. 7 versus the counterface R_q . In all cases, wear rate was the lowest on the lapped counterface. This is encouraging because lapping is a common and inexpensive finishing technique. The wear of unfilled PTFE is relatively insensitive to counterface roughness, shown by the scatter to vary in the repeat experiments. The most prominent feature in this graph is the 100 \times reduction in wear rate for the 80-nm composite over the other composites. This may be the result of filler accumulation at the sliding interface by preferential removal of PTFE because this was also the only composite to have transient wear characteristics (Blanchet (13)). However, wear rate for this

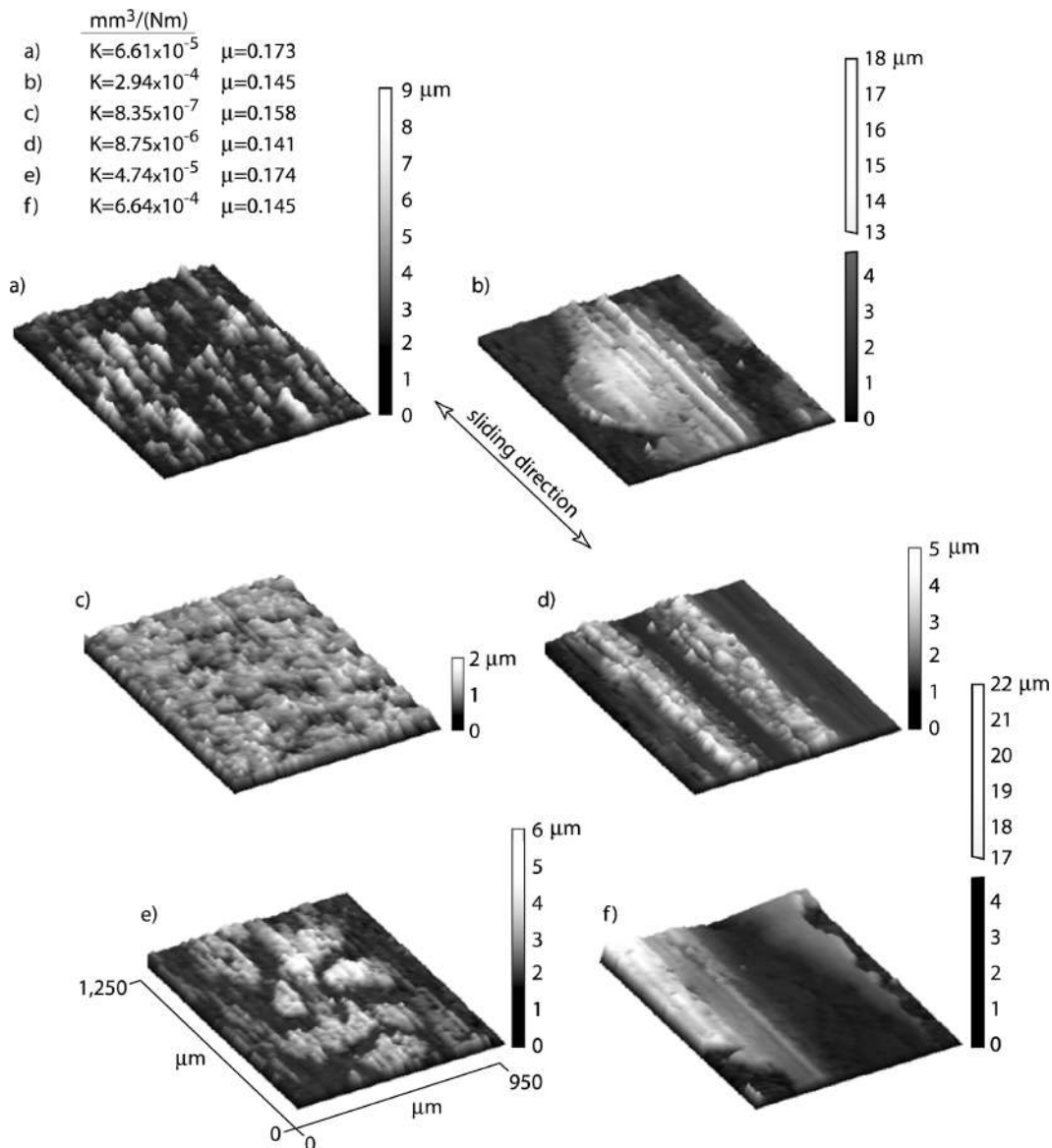


Fig. 8—Scanning white-light interferometry of representative transfer films: (a) 5 wt% 44-nm composite on the lapped counterface, (b) 5 wt% 44-nm composite on the dry-sanded counterface, (c) 5 wt% 80-nm composite on the lapped counterface, (d) 5 wt% 80-nm composite on the wet-sanded counterface, (e) 5 wt% 500-nm composite on the lapped counterface, (f) 5 wt% 500-nm composite on the dry-sanded counterface.

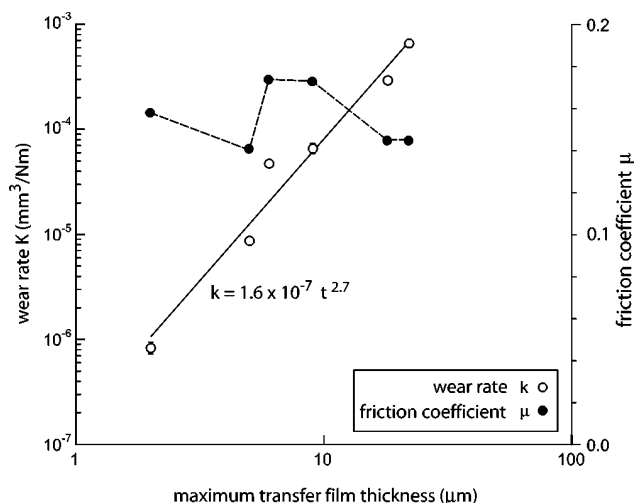


Fig. 9—Wear rate and coefficient of friction plotted versus transfer film thickness. There is a correlation between wear and transfer film thickness. The friction coefficient shows no correlation.

composite increased by an order of magnitude on the wet-sanded counterface. The 44- and 500-nm composites had increased wear rates on the dry-sanded counterface.

Transfer Film Examination

Various surface parameters have been shown to significantly affect tribological interactions (Franklin and de Kraker (8); Wieleba (9); Bahadur and Tabor (10)). Surfaces with negatively skewed histograms generally have good tribological properties. These surfaces are thought to perform well because the asperity peaks are broad and less abrasive while the valleys are deep and sharp, providing sites for transfer film engagement. Perhaps the most important result is that the composites did not show as strong a sensitivity to the counterface roughness as was postulated. In fact, these nanocomposites can be used quite effectively on engineering surfaces and their ability to provide reduced wear rates is not limited to operation on the traditionally highly finished surfaces used in most laboratory testing.

One constant in filled PTFE composites research is that low wear rates are accompanied by small wear debris. The nature of the transfer films, specifically the thickness and coverage, was quantitatively analyzed by using a scanning white-light interferometer. The worst- and best-performing transfer films for each composite are shown in Fig. 8. It is evident from these scans that there is a direct relationship between the thickness of the transfer film and the wear rate. Additionally, the thin transfer films appear more uniformly distributed across the counterface, whereas the thicker films are more banded in the direction of sliding for a given composite. Figure 9 shows wear rate and friction coefficient plotted versus maximum transfer film thickness. The wear rate data follow a power-law curve fit. The friction coefficient appears to be independent of transfer film thickness and morphology.

CONCLUSIONS

1. The addition of alumina particles into a PTFE matrix resulted in smaller wear debris, thinner transfer films, and lower wear rates.
2. The wear rates of the 44-nm composites, albeit a strong function of filler concentration, were a weaker function of surface roughness and correlated well with transfer film thickness.
3. The friction coefficient of the PTFE composites ranged from $\mu \sim 0.12$ to 0.19 and tended to increase with increasing filler concentration and decreasing surface roughness but was not influenced by the thickness of the transfer film.
4. Minimum wear rates were observed for every composite on the lapped surface, and no correlation was observed between wear rate and R_q/D_f .

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