



Transfer film evolution and its role in promoting ultra-low wear of a PTFE nanocomposite

J. Ye, H.S. Khare, D.L. Burris*

Department of Mechanical Engineering, University of Delaware, Newark, DE, United States

ARTICLE INFO

Article history:

Received 23 July 2012

Received in revised form

23 November 2012

Accepted 6 December 2012

Available online 19 December 2012

Keywords:

Solid lubricant

Polymer nanocomposite

Transfer film

Low wear

PTFE

ABSTRACT

Polytetrafluoroethylene (PTFE) is an important solid lubricant with an unusually high wear rate. For a half-century, fillers have been used to reduce PTFE wear by $> 100\times$ with $> 10\%$ loading through hypothesized mechanisms involving mechanical load support, crack arresting, and transfer film adhesion. More recently it was discovered that specific nanoparticles provide a unique nanoscale reinforcement mechanism enabling unprecedented wear reductions of $10,000\times$ with as little as 0.1% nano-fillers. Although the mechanisms responsible for this dramatic improvement remain unclear, there is substantial evidence that the transfer film plays a critical role. This paper uses interrupted microscopy measurements to investigate the evolution of transfer film development for an ultra-low wear PTFE nanocomposite. The run-in wear rates were similar to those of more traditional PTFE composites and transfer films consisted of large plate-like debris. Although the run-in wear rate and debris size decreased monotonically with distance, the run-in transfer films were removed each cycle. Detectable debris vanished and wear rates approached zero at an abrupt transition. During this ultra-low wear transition period, nanoscale and oxidized fragments of PTFE were transferred to the counterface. Most of these fragments persisted for the duration of the test and initiated the transfer film by progressively scavenging trace material from the bulk, growing into small islands, and merging with neighboring islands. The results of this study reflect a complex interplay involving elements of transfer film adhesion, chemistry, debris morphology, and mechanics.

© 2012 Elsevier B.V. All rights reserved.

1. Introduction

Polytetrafluoroethylene (PTFE) is an important solid lubricant with a unique combination of beneficial frictional, thermal, and chemical properties. At sliding speeds well below 5 mm/s , PTFE friction coefficients are low ($\mu \sim 0.05$) and wear rates are moderate ($k \sim 10^{-5}\text{ mm}^3/\text{N m}$) [1–3]. The low friction of PTFE is often associated with low adhesion, but Makinson and Tabor [2] rejected this hypothesis based on the observation of molecularly thin, highly oriented, and strongly adhered transfer films following low friction sliding. They concluded that low friction results from easy shear of PTFE lamellae at low shear rates. At more typical speeds above 50 mm/s , friction coefficients are moderate ($\mu = 0.15\text{--}0.3$), wear rates are high ($k \sim 10^{-3}\text{ mm}^3/\text{N m}$), and transfer films consist of large platelets of poorly adhered debris fragments [2,4,5].

The addition of $20\text{--}50\text{ wt\%}$ microscale fillers (e.g. glass fiber, carbon fiber, bronze particles) can reduce PTFE wear rates by $> 100\times$ at higher speeds [6–11]. However, large hard fillers can

abrade protective transfer films which limits wear resistance. Nano-fillers have been studied for their potential to reduce wear without abrading the beneficial transfer film. Although an initial study by Tanaka and Kawakami indicated that nano-fillers were ineffective fillers because they were too small to disrupt the generation of large-scale debris [10], a series of studies from 2001 to 2003 showed that nanoscale ZnO, SWCNT's, and Al_2O_3 reduced wear rates by $> 100\times$ at a fraction of the typical filler loading ($5\text{--}10\text{ wt\%}$) [12–14]. In 2006, Burris and Sawyer [15] discovered a unique PTFE nanocomposite which demonstrated $> 1000\times$ reductions in wear rates with $< 1\%$ filler loading. This change in wear rate relative to the filler loading remains unprecedented in the field of tribological polymer nanocomposites today [16]. As a result, this particular material has been the subject of numerous follow-up studies of nanoscale polymer reinforcement mechanisms [17–23].

The role of the filler in preventing wear of PTFE and other polymer composites remains an important topic of debate. Blanchet and Kennedy [24] describe two general wear rate determining factors for PTFE composites: (1) the prevention of initial removal of material from the composite; (2) the prevention of the secondary removal of material from the transfer film. Lancaster proposed that fillers reduce primary wear of the matrix

* Corresponding author. Tel.: +1 302 831 2006.

E-mail address: dlburris@udel.edu (D.L. Burris).

by preferentially supporting the normal force [25]. Briscoe [26] proposed that fillers reduce secondary removal by inducing polymer degradation which increases adhesion between the transfer film and the counterface. Ricklin [27], Tanaka et al. [10], and Bahadur and Tabor [28] suggested similar mechanisms of primary wear reduction involving the prevention of large-scale debris generation. Blanchet and Kennedy supported this hypothesis and determined that the specific mechanism of debris size reduction involved the interruption of subsurface crack propagation [29]. Bahadur and Gong [30] made two overarching observations in reviewing the literature: (1) PTFE composite wear rate and filler hardness do not correlate as would be expected for the load support hypothesis; (2) wear rate is strongly dependent on filler chemistry which conflicts with the purely mechanical debris size regulation hypotheses. They supported Briscoe's hypothesis but concluded that filler decomposition rather than polymer degradation increases bonding to the counterface [30]. Gong et al. [31] and Blanchet et al. [24] showed that fillers had no effect on the chemistry at the counterface and independently concluded that the wear behavior is independent of counterface adhesion. Gong et al. [32] suggested that fillers served to improve transfer film cohesion and therefore reduce secondary wear by arresting failure within the transfer film. Blanchet et al. [24] note that severe wear of PTFE only occurs at high speeds and argues that the primary role of the filler must be most closely related to the prevention of primary removal [4,27,28]; this hypothesis is supported by two independent observations that pre-deposited low wear transfer films do not reduce the wear rate of high wear PTFE-based materials [28,33].

Despite opposing viewpoints about the specific wear reducing roles of filler decomposition, polymer degradation, transfer film adhesion, and transfer film cohesion, there is a broad agreement that transfer films play a very important role in the tribology of polymers. Thin and uniform transfer films always accompany low wear sliding of polymers and transfer film adhesion arguments persist today as a causative explanation for low wear rates [34–36]. The purpose of the present study is to elucidate the relationships between wear rate, debris size, and transfer film morphology via direct *in situ* observations of debris generation and transfer film formation. A well-documented PTFE-alumina nanocomposite was chosen as a model of effective reinforcement due to its exceptional wear reduction with low filler loading [17–20].

2. Materials and methods

2.1. Materials

The PTFE nanocomposite materials used in this study replicate, as closely as possible, the preparation conditions from existing literature [15,17–20,37]. The polymer resin is Teflon™ 7C from DuPont (~30 µm diameter particles). The alpha phase aluminum oxide nanoparticles had a reported diameter range of 27–43 nm and were acquired from Nanostructured & Amorphous Materials Inc.¹

2.2. Sample preparation

Aluminum oxide nanoparticles and polytetrafluoroethylene resin were weighed to 0.5 g and 9.5 g, respectively, combined in

a PET container of 10 times the batch volume and pre-mixed by hand shaking for 60 s. The powder ensemble was suspended in 80 mL of anhydrous ethanol which wets PTFE reasonable well [38]. An ultrasonic horn (Sonic Ruptor 400, OMNI International) was pulsed with a 50% duty cycle of 400 W for 5 min. Immediately after ultrasonication, the suspension was transferred to a petri dish placed within a heated vacuum desiccator. Two hours of rough vacuum drying at 110 °C removed the ethanol and prepared the dispersed nanocomposite resin for subsequent compression molding. The dry powder was cold pressed into a 25 mm long, 12.5 mm diameter cylinder using a cylindrical die. Each preform was held at a pressure of 170 MPa for 20 min to eliminate porosity. Following cold compaction, samples were held at 6 MPa pressure and heated to 365 °C at 120 °C per hour, held for 3 h, and cooled at the same rate.

2.3. Wear testing

Prior to testing, each cylindrical specimen was machined into a pin of 6.4 × 6.4 mm testing cross-section and 12 mm height. Grade 304 stainless steel plates (38 × 25 mm) with an average surface roughness of 20 nm (R_a) were used as counterfaces. Wear tests were conducted on the linear reciprocating pin-on-flat tribometer shown in Fig. 1; the tribometer is nominally identical to those reported in previous studies [15,37]. Prior to testing, the sample was preconditioned with 100 mm of sliding at 0.7 MPa of pressure against 600 grit SiC paper to create a uniform pressure distribution for testing. During the test, the applied normal force was 250 N, the contact pressure was 6.4 MPa, the reciprocating length (one direction) was 25.4 mm, and the sliding speed was 50 mm/s. Initial length and mass measurements were used to determine sample density. Mass measurements were then used to quantify mass loss; the volume loss is the ratio of mass loss and density. Friction coefficients, wear rates, and uncertainties were determined using previously described methods [39–41].

Tests were interrupted periodically for analysis. Following interruption, the sample was removed from the mounting fixture and weighed on a balance with a resolution of 0.01 mg. The counterface was removed and located on a kinematic mount beneath a Nikon microscope with a digital camera. Images were captured at a single location in the center of the counterface to follow the morphological evolution of the transfer film. The primary test was interrupted every few cycles to capture the details of the run-in process; interruption intervals increased as the distance between distinct events at the interface increased. Repeat tests with three independent samples were conducted with less frequent interruptions to determine repeatability and the effects of test interruption interval on the results.

3. Results

3.1. Wear behavior of α -aluminum oxide PTFE nanocomposites

This PTFE nanocomposite material is known to exhibit a transient period of moderate wear followed by a transition to a low steady state wear rate [15]. A mass loss measurement taken after 0.2 m revealed an initial wear rate of $k = 4 \times 10^{-4} \text{ mm}^3/\text{N m}$. This high initial wear rate suggests that the direct mechanical reinforcement effect of the nanoparticles (e.g. preferential load support, crazing, crack arresting) is initially limited. Wear volume is plotted as a function of sliding distance for the first 300 m of the primary test in Fig. 2a. There is an obvious run-in period during the first 20 m of sliding where the wear rate decreases monotonically with distance. The wear rate at the end of the run-in period is typical of other PTFE composites and nanocomposites

¹ It is important to note that only particular alpha phase alumina particles activate the ultra-low wear response of interest in this paper. The initiating mechanism likely involves surface-chemistry (and history) dependent interactions between particle and polymer. Prior studies used 27–43 nm particle from Alfa Aesar which are no longer available. Efforts to locate a suitable surrogate demonstrated wide variability in wear rates between suppliers. The particles in this study exhibited the same wear response and have the same reported size range.

Download English Version:

<https://daneshyari.com/en/article/7005150>

Download Persian Version:

<https://daneshyari.com/article/7005150>

[Daneshyari.com](https://daneshyari.com)