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Case study

Tribological results of PEEK nanocomposites in dry sliding against 440C in various gas environments

N.L. McCook, M.A. Hamilton, D.L. Burris, W.G. Sawyer *

Department of Mechanical and Aerospace Engineering, University of Florida, Gainesville, FL 32611, United States

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Abstract

This case study reports on a recent survey of filled polyetheretherketone composites. A wide variety of available nanoparticles and microparticles were used during this exploratory activity. The friction coefficients ranged from below $\mu = 0.05$ to over $\mu = 0.60$; the wear rates varied from 10^{-4} to 10^{-7} $mm^3/(N\,m)$. The tests were run against 440C stainless steel counterfaces in open laboratory air with approximately 45% RH, a dry nitrogen environment with less than 0.5% RH, and unfilled PEEK was cycled between ambient air and high vacuum (10^{-6} Torr). The open (with edge sites) and closed (without edge sites) structures of the solid lubricant particles did not demonstrate a clear environmental dependence, while the PEEK showed a very low friction coefficient and wear rate in dry and vacuum environments.

Keywords: Solid lubrication; Space; Nanocomposites; Polymer tribology

1. Introduction

There is significant enthusiasm for the use of polymeric nanocomposites in application areas that involve environments where traditional fluid lubricants cannot be used. One of the often cited potential advantages of nanofillers over traditional hard fillers is that they can be less abrasive. It is thought that nanocomposites can provide novel functionality in the area of bulk polymeric components by either responding to environmental changes [1–5], or by being inert. For example, molybdenum disulphide (MoS_2) and coatings containing MoS_2 are well known materials for space applications, but oxygen poisoning and deterioration that occurs during ground testing can cause unacceptably high friction coefficients and wear rates. In contrast, graphitic materials operate best in humid environments. With the recent advent of closed shell nanoparticles, traditional solid lubricants are now available without the edge sites that are thought to drive these environmental sensitivities.

* Corresponding author. Tel.: +1 352 392 8488.
E-mail address: wgsawyer@ufl.edu (W.G. Sawyer).

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This case study surveys a wide range of filled PEEK composites, and provides the average friction coefficient data and wear rate data for these samples. This breadth of data is offered to the community to support of future composite developments, and to provide a comparative suite that modelers and experimentalists can use to complete their models and understanding. This data set may also help to further complete a fairly exhaustive set of experiments conducted on ceramic nanoparticle filled PEEK [6–11] conducted in open air environments.

2. Materials and experimental conditions

PEEK was chosen as matrix material because of its high mechanical strength, demonstrated ability to work as a nanocomposite matrix in tribological applications [6–11], and compatibility for vacuum operation (namely, low outgassing in vacuum). Fig. 1 shows TEM micrographs of the materials that were used in this study. A cryoground PEEK with a characteristic particle size of 10 μm was used [12] as the matrix. The filler materials were microcrystalline graphite (∼2 μm), carbon nano-onions [13], single-walled carbon nanotubes, C60 carbon

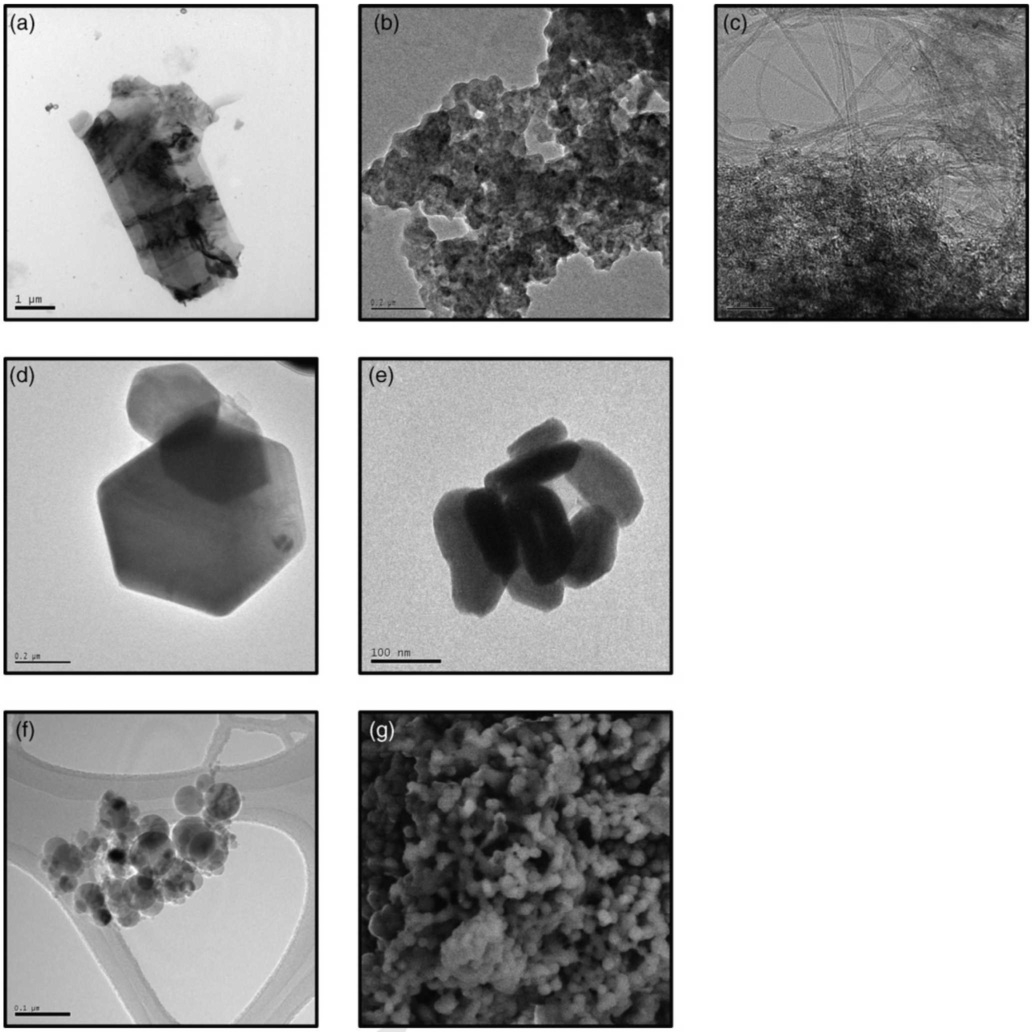

Fig. 1. Materials used as fillers in PEEK composites: (a) graphite powder, (b) carbon nano-onions, (c) single-walled carbon nanotubes, (d) tungsten disulphide powder, (e) tungsten disulphide fullerenes, (f) alumina nanoparticles and (g) PTFE nanopowder. No images of the C60 were collected.

fullerenes, microcrystalline WS_2 (∼600 nm), WS_2 fullerenes [14–21], $\Delta{:}\Gamma$ 40 nm alumina nanoparticles [22], and nanoparticles of PTFE [23].

The composites were prepared by jet-milling powder ensembles of 2, 5, and 10 wt.% as described in a previous publication [24], and compression molding at 360 °C for 10 min with a 2 °C/min ramp up and down in temperature. Samples were machined from the interior of the molded sample using a computer numerical control milling machine. The final samples were 6.35 mm × 6.35 mm × 12.7 mm. The 440C stainless steel counterfaces had a hardness of approximately 3.2 GPa and were polished to better than R_q = 30 nm.

For the majority of the studies a linear reciprocating pin-on-disk tribometer was used. This apparatus and the experimental uncertainties associated with friction coefficient measurements [25] and wear rate measurements [26] were reported earlier. For this study the normal load was maintained at 250 N, the sliding speed was 50 mm/s, and the stroke length was 25 mm. All tests were run for 70,000 cycles (∼3.5 km of sliding) and wear rates were calculated from single point observations at the end of the experiment. In general, tribological transients constituted less than 10% of the test. The tribometer was located in either an open air clean room environment at 45% RH, or in a temperature and environmentally controlled glove box that was backfilled with nitrogen gas (measured at better than 0.5% RH). A vacuum study at 10^{-6} Torr was performed on a custom pin-on-disk tribometer that uses a vacuum compatible six-channel load cell with similar design and uncertainties to the reciprocating tribometer described earlier [25,26]; the only sample evaluated on this tribometer was the unfilled PEEK sample.

3. Results and discussion

The results from these experiments are given in Table 1 and are summarized by the bar graphs in Fig. 2, and the scatter plot in Fig. 3. The only notable trend was that the composites tended to operate better in the inert gas environment. This is most clear in Fig. 3, where both wear rate and friction coefficient were reduced

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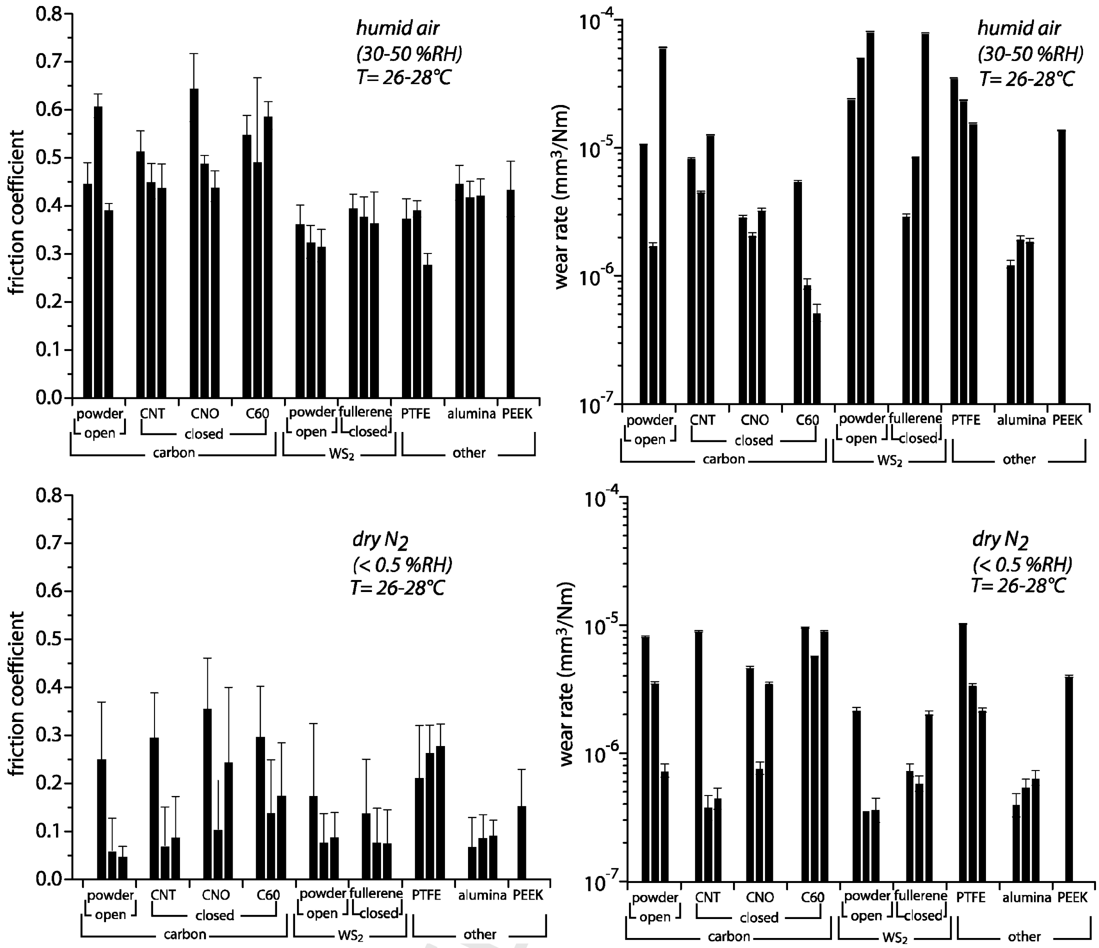

Fig. 2. Tribological results for various PEEK composites against 440C counterfaces in laboratory air and dry N_2 environmental testing. Top left: friction coefficient for laboratory air testing, top right: wear rate for laboratory air testing, bottom left: friction coefficient for dry N_2 testing, and bottom right: wear rate for dry N_2 testing. The words "open" and "closed" denote structure, with or without edge sites, respectively.

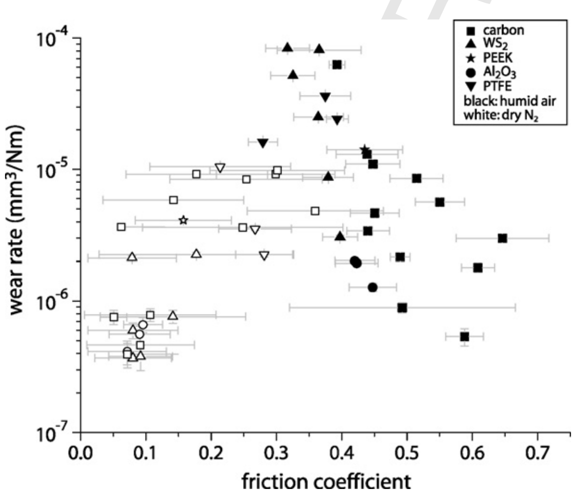

Fig. 3. Wear rates and uncertainties plotted vs. friction coefficients and the experimental variations in friction coefficient for composites tested in laboratory air (black) and dry N_2 environments (white).

in the dry nitrogen environment. Surprisingly, this was ubiquitous even from samples such as the microcrystalline graphite (which we assumed would perform best in open air), and alumina (which we assumed would be insensitive).

We speculated that the tribological behavior of the PEEK matrix was sensitive to the amount of water vapor in the environment. An experiment that varied the water vapor content in the environment chamber showed a monotonic increase in friction coefficient for unfilled PEEK going from $\mu = 0.1$ to $\mu = 0.55$ with relative humidity changes from 0.2 to 1.6%. Above 2% relative humidity, no further increases in friction coefficient were observed for unfilled PEEK. In high vacuum tests pin-on-disk tests at 8×10^{-7} Torr PEEK also showed a friction coefficient around $\mu = 0.1$. It is interesting that in dry environments the more wear resistant coatings also had lower friction coefficients, while in open air environments the more wear resistant coatings had higher friction coefficients.

The polymeric nanocomposites of PEEK all had similar environmental responses. We speculate that PEEK is not as environmentally insensitive to water vapor we had originally thought. The PEEK matrix may have swamped the potential effects of the nanoparticles that we were trying to interrogate.

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Table 1
Results of friction coefficient and wear tests for various PEEK composites against 440C counterfaces in (a) laboratory air (35–50% RH) and (b) dry N_2 (<0.5% RH) environments

	0 wt.%	2 wt.%	5 wt.%	10 wt.%
(a) Laboratory air				
Carbon				
Powder		$\mu = 0.448$, $k = 10.9$	$\mu = 0.608$, $k = 1.80$	$\mu = 0.393$, $k = 62.6$
CNT		$\mu = 0.515$, $k = 8.52$	$\mu = 0.451$, $k = 4.65$	$\mu = 0.439$, $k = 13.0$
CNO		$\mu = 0.646$, $k = 2.99$	$\mu = 0.490$, $k = 2.16$	$\mu = 0.440$, $k = 3.39$
C60	$\mu = 0.435$, $k = 14.1$	$\mu = 0.550$, $k = 5.65$	$\mu = 0.493$, $k = 0.894$	$\mu = 0.588$, $k = 0.537$
WS_2				
Powder		$\mu = 0.364$, $k = 24.9$	$\mu = 0.325$, $k = 51.6$	$\mu = 0.317$, $k = 83.6$
Fullerence		$\mu = 0.397$, $k = 3.05$	$\mu = 0.379$, $k = 8.69$	$\mu = 0.365$, $k = 81.3$
Other				
PTFE		$\mu = 0.375$, $k = 36.1$	$\mu = 0.393$, $k = 24.3$	$\mu = 0.279$, $k = 16.2$
Alumina		$\mu = 0.448$, $k = 1.28$	$\mu = 0.420$, $k = 2.02$	$\mu = 0.423$, $k = 1.94$
(b) Dry N_2				
Carbon				
Powder		$\mu = 0.254$, $k = 8.38$	$\mu = 0.062$, $k = 3.65$	$\mu = 0.050$, $k = 0.755$
CNT		$\mu = 0.299$, $k = 9.18$	$\mu = 0.072$, $k = 0.394$	$\mu = 0.091$, $k = 0.464$
CNO		$\mu = 0.359$, $k = 4.83$	$\mu = 0.107$, $k = 0.787$	$\mu = 0.248$, $k = 3.61$
C60	$\mu = 0.157$, $k = 4.09$	$\mu = 0.301$, $k = 9.82$	$\mu = 0.142$, $k = 5.82$	$\mu = 0.178$, $k = 9.19$
WS_2				
Powder		$\mu = 0.177$, $k = 2.25$	$\mu = 0.080$, $k = 0.368$	$\mu = 0.092$, $k = 0.378$
Fullerence		$\mu = 0.141$, $k = 0.761$	$\mu = 0.080$, $k = 0.60$	$\mu = 0.079$, $k = 2.12$
Other				
PTFE		$\mu = 0.214$, $k = 10.5$	$\mu = 0.267$, $k = 3.52$	$\mu = 0.281$, $k = 2.24$
Alumina		$\mu = 0.071$, $k = 0.413$	$\mu = 0.090$, $k = 0.562$	$\mu = 0.095$, $k = 0.663$

Wear rates are expressed with units of 10^{-6} $mm^3/(N\,m)$.

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