NASA Technical Memorandum 107299

Spectroscopic Analysis of Perfluoropolyether Lubricant Degradation During Boundary Lubrication

Pilar Herrera-Fierro
Ohio Aerospace Institute
Brook Park, Ohio

Bradley A. Shogrin
Case Western Reserve University
Cleveland, Ohio

William R. Jones, Jr.
Lewis Research Center
Cleveland, Ohio

National Aeronautics and
Space Administration

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Pilar Herrera-Fierro
Ohio Aerospace Institute
Brookpark, OH 44142

Bradley A. Shogrin
Case-Western Reserve University
Cleveland. OH 44106

William R. Jones, Jr.
NASA Lewis Research Center
Cleveland. OH 44135

ABSTRACT

The degradation of a branched perfluoropolyether (PFPE) under boundary lubrication conditions was studied using μ-FTIR and μ-Raman spectroscopies. Stainless steel (440C) discs coated with thin (600Å), uniform films of the PFPE were tested in a ball-on-disc apparatus until various levels of friction coefficient were attained. Discs were then examined using the above techniques. When the friction coefficient surpassed the value obtained with an unlubricated control, the lubricant film had either been physically displaced or partially transformed into a "friction polymer". Infrared analysis of this "friction polymer" indicated the presence of a polymeric fluorinated acid species (R_fCOOH). Raman spectroscopy indicated the presence of amorphous carbon in the wear track and in the friction polymer. Some reaction mechanisms are suggested to explain the results.

Keywords: perfluoropolyethers, boundary lubrication, degradation

INTRODUCTION

The class of liquid lubricants, known as the perfluoropolyethers (PFPE's), have been used extensively for space applications for many years (*1*) and more recently as lubricants for magnetic recording media (*2*). These fluids are also excellent candidates for advanced turbine engine applications (*3*). One member of this class based on the polymerization of hexafluoropropene oxide (HFPO) has been available for over 30 years (*4*).

Although these materials are normally quite stable compared to conventional lubricants, they do degrade at high temperatures in contact with catalytic surfaces and at room temperature in tribological contacts (*5*). In fact, these tribochemical degradation products allow unformulated PFPE fluids to survive in boundary lubricated contacts by forming low shear boundary films, such as FeF_3 (*1*), (*6*).

A number of investigators have studied the fate of fluorinated materials in boundary lubricated contacts using a variety of surface analytical techniques. In a series of papers, Sugimoto and Miyake (*7*), (*8*), and (*9*) studied the progression of degradation of sputtered films of polychlorotrifluoroethylene (PCTFE) and polytetrafluoroethylene (PTFE) sliding against 440C steel. They used a variety of techniques to study the surface chemistry including: X-ray photoelectron spectroscopy (XPS), auger electron spectroscopy (AES), micro-Fourier transform infrared spectroscopy (μ-FTIR) and laser Raman spectroscopy. They concluded that these fluorinated polymers are progressively degraded into a fluorine deficient material and finally into an amorphous carbon network and metallic carbides.

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Novotny et al (*10*) and Karis et al (*11*) have reported on the degradation of PFPE lubricants on magnetic media and in ball mill experiments, respectively. They concluded that PFPE lubricants are removed from the contact area by both physical displacement and loss due to degradation. The dominant degradation process was tribochemical scission resulting in lower molecular weight fragments that can desorb and carboxylic acid containing fragments that can physically adsorb on the surfaces.

Carré and Markovitz (*12*) reported the formation of fluorinated carboxylic acids in static experiments with a branched PFPE in the presence of Lewis acids at elevated temperatures. Eapen et al (*13*) performed similar experiments and arrived at the same conclusions. Infrared spectroscopy was used in both cases to identify the products. Xuan et at (*14*) also detected carbonyl and OH infrared absorption peaks.

The objective of this paper is to further investigate the progressive degradation of a branched PFPE during sliding experiments with a ball-on-disc apparatus. Micro-Raman and micro-FTIR were used to analyze the surfaces. Test conditions included: a sliding speed of 0.05 m/s, a 3N load, 440C steel specimens, ambient temperature (~25°C), and a dry nitrogen atmosphere with a relative humidity of < 2%.

EXPERIMENTAL

Infrared Spectroscopy

Infrared spectra were collected with an infrared microscope in the reflectance mode using a 32X grazing angle objective (GAO). A gold coated glass slide was used to record the background spectra. The acquisition time, spectral resolution and area of analysis were 400 seconds, 8 cm^{-1}, and 100 μm, respectively.

Raman Spectroscopy

Raman spectroscopy was obtained on an instrument equipped with a CCD (charge couple device) detector and confocal slits set at 5 microns. The excitation source is an argon (Ar) ion laser (5414.5 nm wavelength), with a maximum power of 20 mW. A 50X objective was used to analyze an area of 2-4 μm^2. The acquisition time was 50 seconds and the spectral range was 150 to 3200 cm^{-1}. Since some samples were highly fluorescent, the actual laser power used was 10 mW. This also reduced the possibility of damage to the sample. The instrument was aligned to the type 2A diamond line at 1331.2 and Si (111) at 520.8 cm^{-1}.

Materials

Discs and balls were made of fully hardened 440C stainless steel. The discs were 17.5 mm in diameter and 4.76 mm thick. Each disc had a mirror finish with an arithmetic roughness average (R_a) of ~0.01 μm. Bearing balls were Grade 10 with a diameter of 9.53 ± 0.001 mm. The lubricant was a commercially available branched PFPE fluid based on hexafluoropropene oxide (HFPO). Typical properties appear in Table 1.

Sample Preparation

The 440C discs were polished on microcloth with 0.3 μm alumina and tap water. Polishing was repeated twice, using a previously unused microcloth. After polishing, the discs were rinsed with tap water to remove residual alumina. The discs (with accompanying ball) were then ultrasonically cleaned in hexane, followed by acetone, and finally methanol (~ 10 minutes each). The pair was then blown dry with nitrogen and promptly covered to minimize dust contamination. Both specimens were then cleaned in a

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commercial UV-ozone apparatus (*15*) to remove adventitious carbon. Application of the

PFPE film followed within 5 minutes after UV-ozone cleaning.

Lubricant Application

Lubricant films were deposited onto the discs (balls were not lubricated) using a

solution of lubricant in a perfluorinated cyclobutane solvent (50 g/l concentration). The

disc was placed in an apparatus that allowed the withdrawal of the solution at a constant

flow rate (*16*). As the solvent evaporates, a thin, uniform film of the nonvolatile lubricant

remains on the surface.

Lubricant Film Thickness Measurements

The thickness of the lubricant on each disc was measured with an infrared

microscope in the reflectance mode using a Grazing Angle Objective (GAO). A spectrum

from a gold-coated glass slide was used as the background. Absorbance as a function of

wave number was determined for each film at three different radii on the disc's surface,

$\sim 120°$ apart. The absorbance of the 1313 cm^{-1} band was measured and compared to the

absorbance versus film thickness curve obtained theoretically (*17*). The films for this

study were 600 Å $\pm$ 120 Å thick.

Tribological Testing

Tribological testing of each disc was performed on a fixed-ball-on-disc tribometer

(*16*) shown in Figure 1. All tests were performed using a relative sliding speed of 0.05

ms^{-1} and a 3-N normal load which generated an initial Hertzian contact stress of ~ 0.7 GPa.

All tests were run at ambient temperatures ($\sim$ 25 °C). The majority of the tests were run

in dry nitrogen (< 2% RH), while others were run in ambient air. The friction force as a function of disc revolutions (cycles) was recorded for each test (one wear track).

After a test, the ball support fixture and ball were rotated to expose an unworn surface on the ball and the fixture was extended so the ball would contact the disc at a different radius. By doing this, three virgin wear tracks, 1.6 mm apart, could be worn on each disc. Each test was run until the desired value of the friction coefficient was attained. The experiment was then stopped and the specimens disassembled. The wear scars and unworn areas on both specimens were then examined with FTIR and Raman spectroscopies. After spectroscopic analysis, all specimens were ultrasonically cleaned in trifluorotrichloroethane until no PFPE was detected on the surfaces with FTIR.

RESULTS

Specimens were run on the ball-on-disc apparatus until one of three criteria were attained. These criteria correspond to different values of the mean friction coefficient and are referred to as Regions I, II and III. During Region I, the friction coefficient remains essentially constant at approximately 0.20 ± 0.06. Region II represents a transition region which typically both begins and lasts several hundred cycles. In this region, the friction coefficient gradually increases from 0.2 to approximately 0.63. Region III represents total loss of lubrication function, as the friction coefficient is ≥ 0.63. This corresponds to an unlubricated metal-metal contact (*16*). A typical friction trace as a function of number of cycles for a 600 Å PFPE film appears in Figure 2. Micrographs of typical wear tracks for each region appear in Figure 3.

FTIR Analysis: Region I

Typically, infrared analysis of wear tracks in this region indicated no chemical changes, only a thinning of the lubricant layer. This thinning corresponds to about a 40 % decrease in the original film thickness. Lubricant accumulation at the sides of the wear track was not observed. Therefore, the majority of displaced lubricant was transferred to the unlubricated ball. Lubricant thickness in the wear tracks varied from 240 to 400 Å. Figure 4 contains infrared spectra of the original film (a) and of a wear track (b) from Region I.

FTIR Analysis: Region II

In this transition region, the test was stopped after the friction coefficient reached a value of 0.36 ± 0.03. In general, IR analysis indicated that the lubricant had thinned further, but there was still no indication of any chemical changes. Film uniformity varied greatly. Some wear tracks yielded relatively uniform films. Others had some areas, at least 100 μm in diameter, completely void of lubricant. This wide variation in lubricant thickness, together with the increase in friction and wear, are indicative of the gradual loss of lubricity that occurs in this region.

FTIR Analysis: Region III

In some places in the wear scar of a failed track IR analysis indicated a complete absence of lubricant. In other places, IR analysis indicated small patchy amounts of lubricant. This has been observed previously (16) and was used to explain oscillations in the friction coefficient due to local reservoirs of lubricant among the surface asperities in the wear track.

A typical IR spectrum of the wear track in this region appears in Figure 5. This signature is quite different from that of unused lubricant. A brown material was visually observed on the wear track. This brown material yields a broad band at 3000 to 3600 cm^{-1} and another set of bands at 1669 cm^{-1} and 1436 cm^{-1}. The broad band can be assigned to hydrogen bonded hydroxyl groups. The other two bands can be assigned to carboxylic acid species. There are also C-F vibrations from the original lubricant at 990, 1127, 1270 and 1310 cm^{-1}.

The growth of the 3000-3600, 1669 and 1436 cm^{-1} bands simultaneously occurs with the change in appearance of the wear track. When the brownish material is obvious in the wear track, these three bands are easily detected. These bands were present in the wear track after both nitrogen and air runs. Relative abundance was directly related to the original film thickness. Intense bands were present after runs using thicker initial lubricant films.

Figure 6 shows the IR spectra of the track surface before and after rinsing with trichlorotrifluoroethane. After rinsing, only the species associated with the carboxylic acid remained. Most of the C-F signal associated with the unaltered PFPE has disappeared.

Ball

Ball wear scars were also analyzed. Since the balls were not lubricated prior to testing, all material found on the scar must come from the contact area. The entrance and exit regions of the scar show an accumulation of wear debris and the same brown material found in the wear tracks. This brown material is abundant in these regions. When the apparatus was run with thick lubricant films, this material could be removed and

transferred to a gold coated slide for further examination. A micrograph of a typical ball wear scar with accompanying debris appears in Figure 7. The IR spectrum of the brown material is shown in Figure 8. Like its counterpart on the disc's wear tracks, this transformed material is insoluble in fluorinated solvents or hexane at room temperature and is strongly attached to the surface. Sometimes, there is a build-up of this material in the entrance to the wear scar and along the track. This brown, insoluble material is termed "friction polymer." When a large amount is present, it can be removed and placed on a glass slide for spectral analysis. The Raman analysis of such a sample is described below.

Raman Analysis

Raman spectra for the region from 1000 to 1900 cm^{-1} of several surfaces appear in Figure 9. A spectrum for the 440C surface after application of the PFPE film is shown in Figure 9a. This is the signature of the carbon sp^3 backbone of the original PFPE. Spectrum 9b was obtained from a wear track run to failure (Region III). For comparison, a spectrum of a commercially available fluorinated amorphous carbon powder appears in Figure 9c. Figure 9d contains the spectrum of tribologically degraded PFPE (friction polymer) removed from a ball (figure 7) and deposited on a gold slide. The peak at 1364 cm^{-1} can be assigned to the so called "D" band. It can be attributed to the scattering by disorder activated optical zone edge phonons of graphite (18) which is due to various orientations of graphitic micro or nanocrystallites. Thus, the "D" band is a direct measure of the order (disorder) of this tribologically created structure. The broad band at 1604 cm^{-1} is the original "G" peak found in highly oriented pyrolytic graphite at

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1576 cm^{-1}, previously assigned to scattering by graphitic optic zone center phonons (*18*). This band shifts and broadens as a result of the change from mostly C sp^3 to C sp^2 bonding. Thus, Figure 9b shows both the broad doublet from the "G" and "D" bands, and the signature of residual, undegraded PFPE lubricant.

DISCUSSION

Infrared Spectra

The infrared spectra of the degraded lubricant are consistent with a chain scission process resulting in the eventual formation of an adsorbed carboxylate species, as indicated by the presence of the 1669 and 1436 cm^{-1} peaks. These represent the asymmetric and symmetric stretching frequencies of the carboxylate. Liang and Roselius (*19*) observed a somewhat similar spectrum while studying the adsorption of a perfluoroacyl fluoride onto alumina under vacuum conditions. However, they concluded that the acyl C-F bond was not broken and, therefore, a carboxylate species was not formed. However, tribotests with a commercial PFPE fluid under ambient conditions yielded a high molecular weight carboxylic acid species (*20*). Other researchers have reported the formation of carboxylic acid species from tribotests (*10*), (*11*), as well as static tests with Lewis acids (*12*), (*13*).

For comparison, Figure 10 shows the IR spectra of neat trifluoroacetic acid, both in transmission as well as adsorbed onto a 440C steel disk. The asymmetric carbonyl stretching frequency shifts from 1786 to 1660 cm^{-1}. The symmetric carbonyl stretching band also shifts from 1444 to 1473 cm^{-1}. The adsorbed carboxylate must be hydrogen bonded, as suggested by Liang and Helmick (*20*), as indicated by the broad band from

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3000 to 3500 cm^{-1}. It is well known that hydrogen bonding and conjugation shift the C=O stretching band to lower frequencies.

Raman Spectra

The Raman effect (*21*) can be thought of as a collision process between photons and the molecules or lattice units. The loss of energy, due to these collisions, can cause the amount of vibrational or rotational energy to increase. Therefore, changes in the material due to stress, orientation, and environment are reflected as changes in the Raman spectra. Strong Raman scattering arises from non-polar groupings as opposed to IR absorptions from polar groups. Therefore, the Raman spectrum of a polymeric material will yield information about the carbon chain, whereas the IR spectrum produces information about the substitutients on the chain. Thus Raman spectroscopy was also used to study the nature of the "friction polymer" formed in the tribocontacts.

As the PFPE is progressively degraded in the tribocontacts, the Raman spectra indicated the formation of C=C sp^2 type bonding. This would represent a C-C network produced by a cross linking process. A very similar phenomenon was observed by Sugimoto and Miyake (*8*) for fluorinated polymers.

The spectroscopic evidence presented here confirms the degradation pathway suggested by others. The mechanism involves defluorination, chain scission, and the formation of carbonyl end groups. The carbonyl groups are humidity labile, reacting with residual water to form carboxylic acid groups. These acidic groups, in turn, interact with the surface metal oxides to produce carboxylates. It has been suggested (*22*) the presence of both asymmetric and symmetric stretching modes in the IR spectra indicates that the

COO⁻ groups are asymmetrically bonded, in a monodentate fashion. This configuration could easily lead to a C-C framework that includes -OH species through hydrogen bonding and some fractions of the original fluoroether chain. The resulting fluorine deficient network could explain the graphitic nature of the Raman signal. This high molecular weight "polymeric network" formed on the surface would not be soluble in fluorinated solvents. The nature of the bonding of this network to surfaces is not clear given the complexities of the surface chemistry and the polymeric material.

CONCLUSIONS

The following conclusions can be drawn from these experiments.

1. The PFPE lubricant is progressively degraded to an insoluble "friction polymer" containing a mixture of carboxylic acid species, partially fluorinated carbonaceous material and amorphous carbon.

2. Contact failure is associated with almost complete conversion of the fluorinated polymer to amorphous carbon.

3. The Raman and infrared signatures can be used to give a clear indication of the approach to contact failure.

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Table 1. Selected Properties of the
PFPE Used in the Study

Average molecular weight	11000 amu
Vapor pressure at 20 °C at 50 °C at 100 °C	4×10^{-12} Pa 3×10^{-10} Pa 1×10^{-7} Pa
Kinematic viscosity at 20 °C at 50 °C at 100 °C	2717 mm^2s^{-1} 444 mm^2s^{-1} 63 mm^2s^{-1}
Density at 20 °C at 50 °C at 100 °C	1.92 gcm^{-1} 1.87 gcm^{-1} 1.78 gcm^{-1}
Surface tension at 25 °C	19 $dynescm^{-1}$
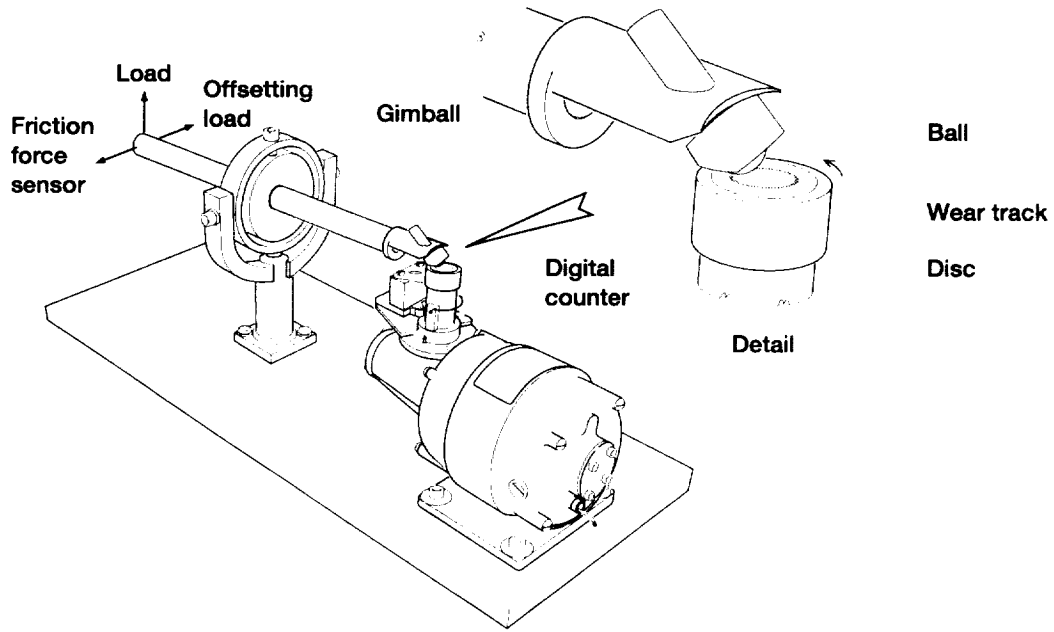
Figure 1.—Fixed ball-on-disc tribometer.
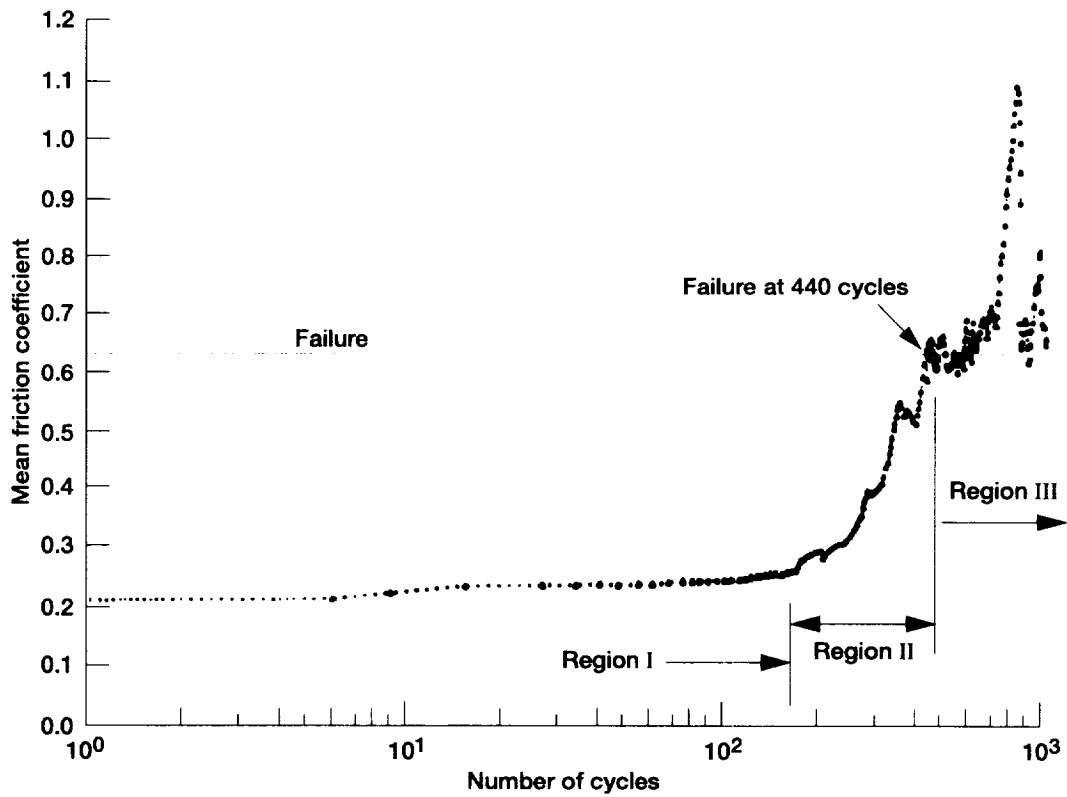
Figure 2.—Typical friction coefficient as a function of number of cycles for a 600 Å PFPE film.
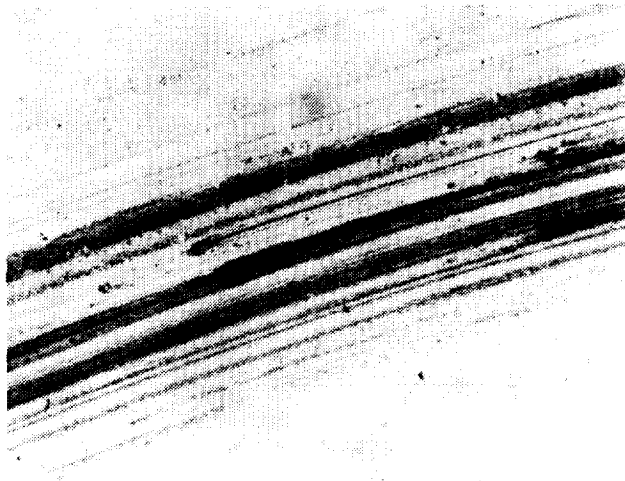
Region I
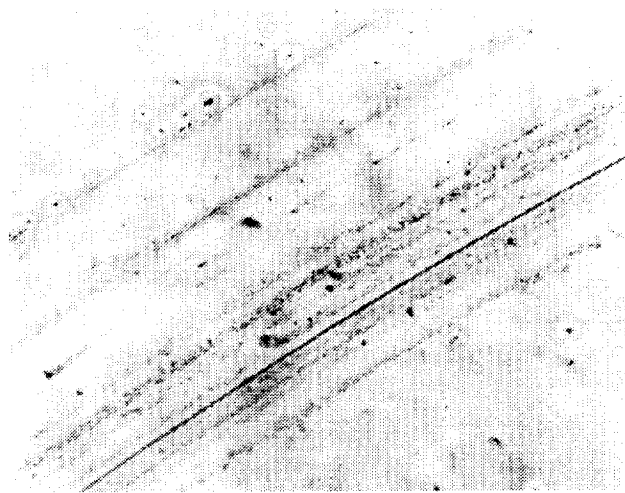
Region II
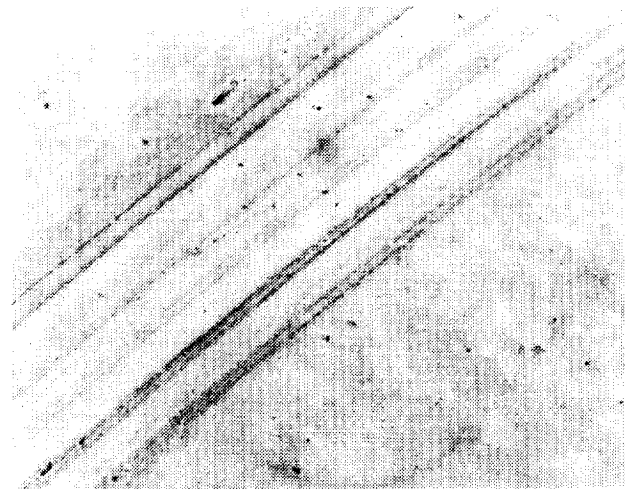
Region III

Figure 3.—Micrographs of the wear track for each of the regions labeled in the friction trace (Fig. 2).
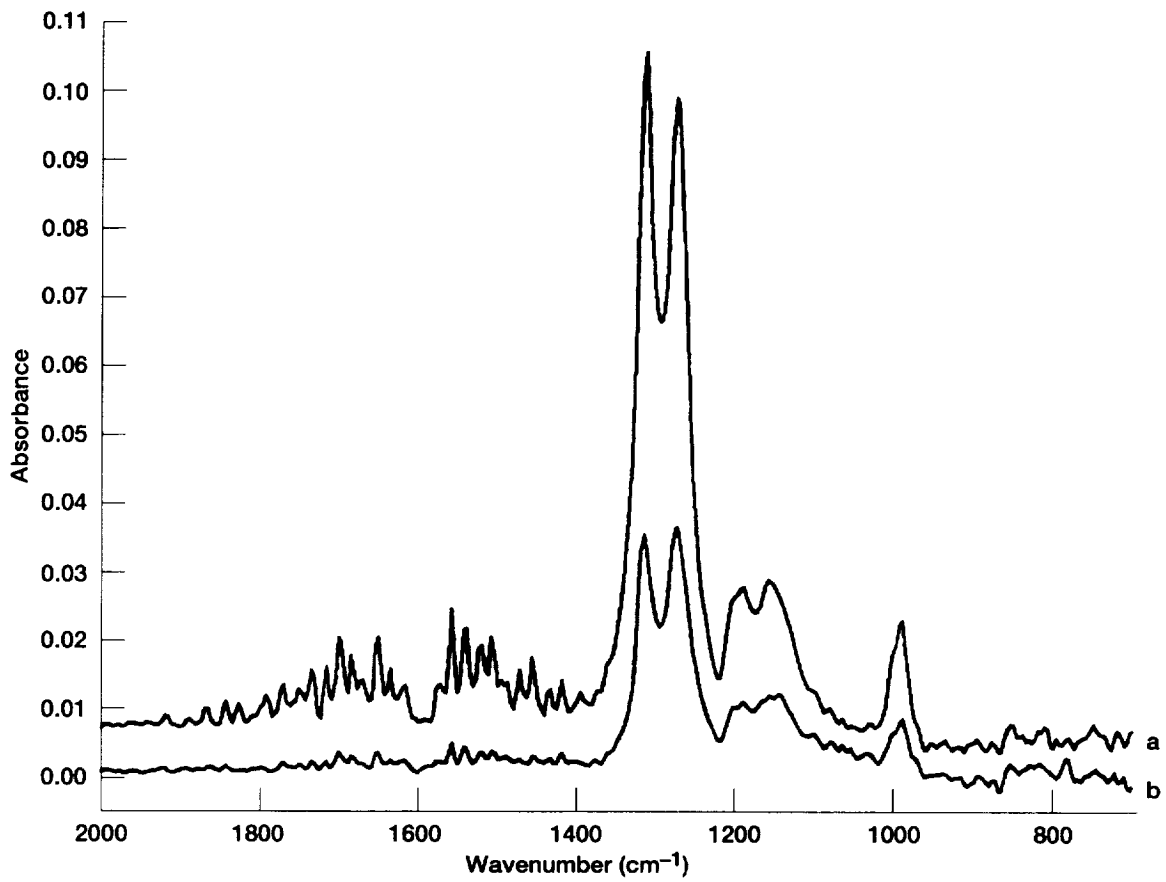
Figure 4.—FTIR spectra of the original film (a) and of a wear track from region I (b).
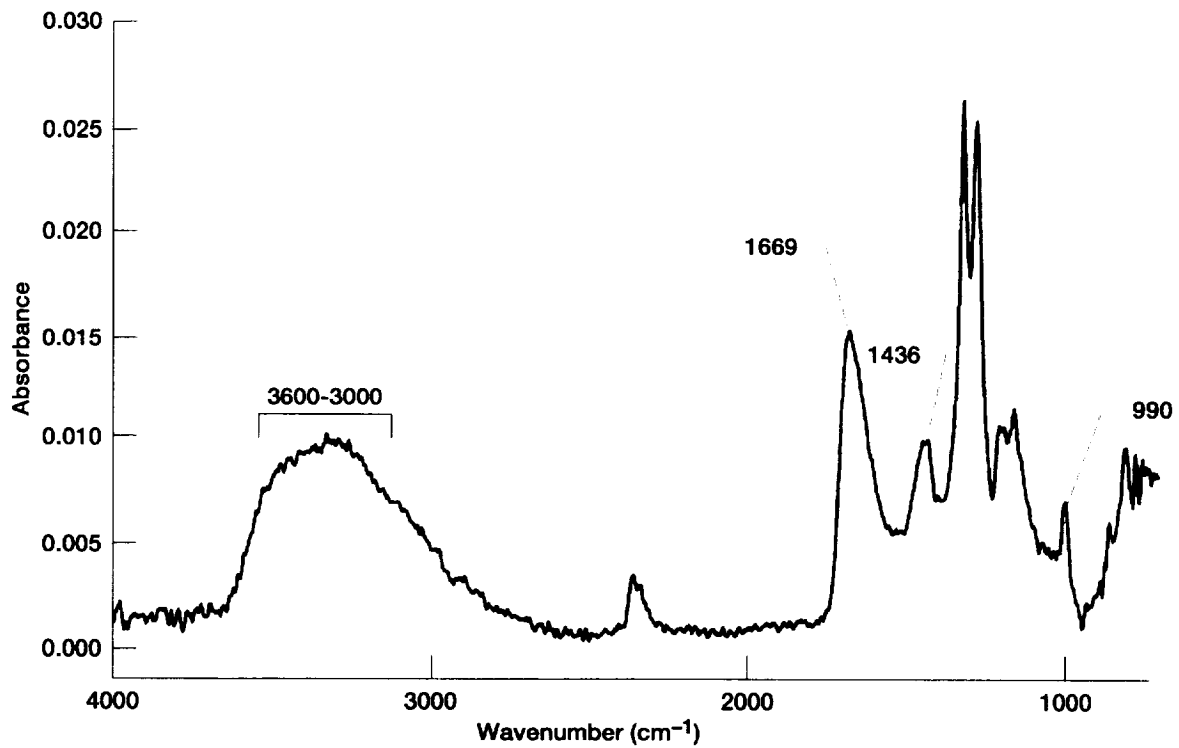
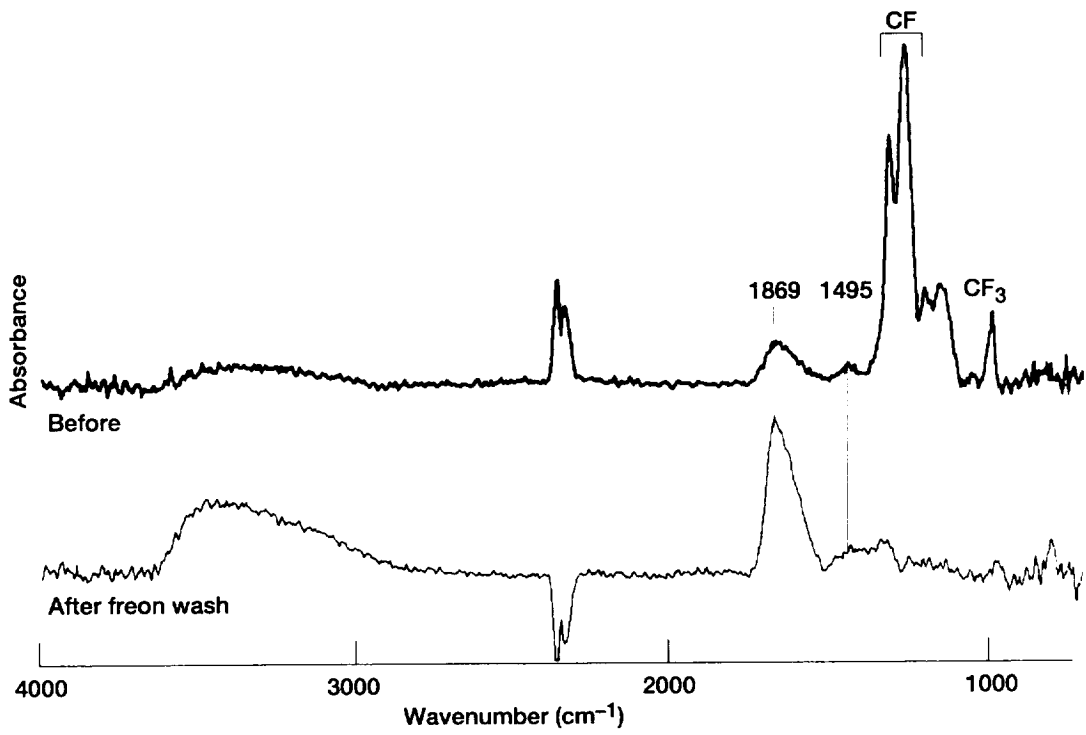Figure 5.—FTIR spectra of a wear track from region III.

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Figure 6.—FTIR spectra of the track surface on the disc before and after rinsing with trichloro-trifluoroethane.

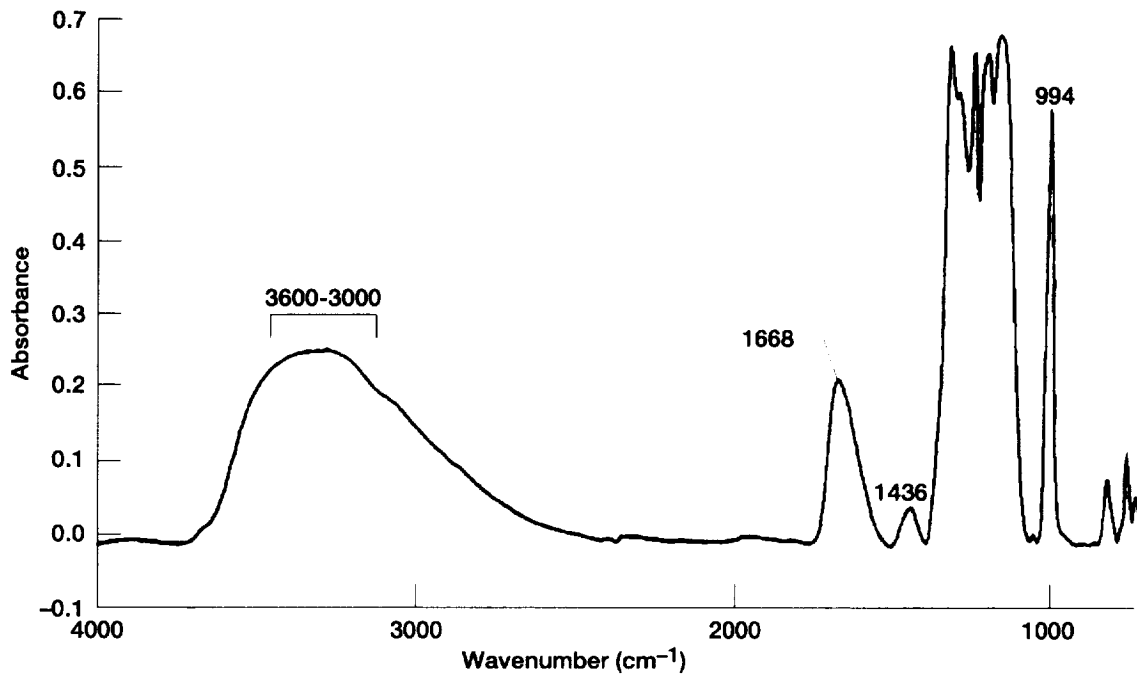

Figure 7.—Typical ball wear scar with accompanying debris.

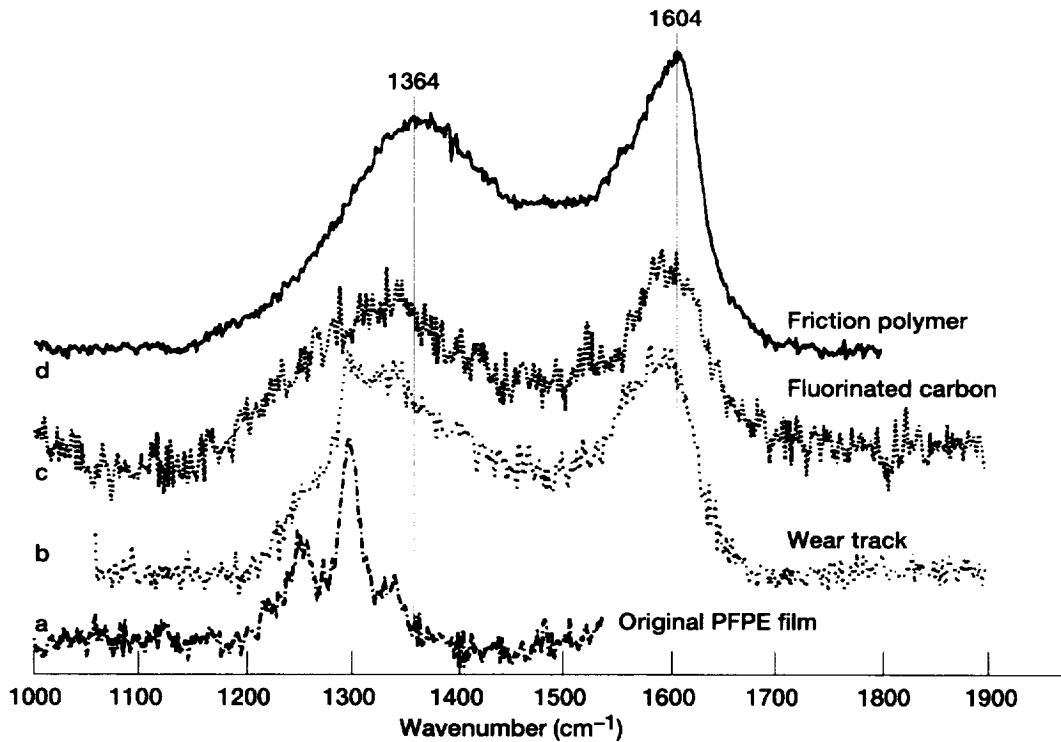Figure 8.—FTIR spectrum of the brown colored material found on the wear scar.

Figure 9.—Raman spectra. (a) 440C surface after PFPE film deposition. (b) Wear track run to failure (region III). (c) Fluorinated amorphous carbon. (d) Tribologically degraded PFPE deposited on a gold slide.
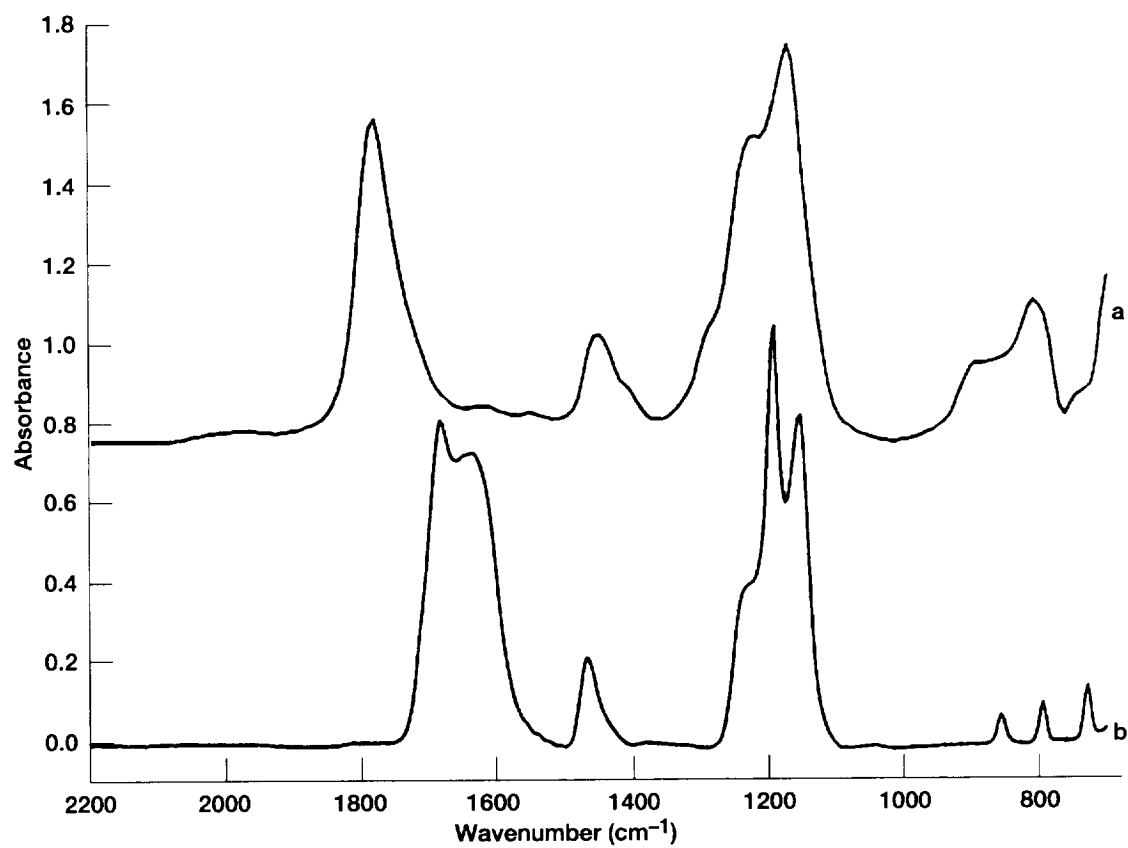
Figure 10.—FTIR spectra of fluoroacetic acid. (a) In transmission. (b) Adsorbed on 440C steel.

REPORT DOCUMENTATION PAGE

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1. AGENCY USE ONLY (Leave blank)	2. REPORT DATE September 1996	3. REPORT TYPE AND DATES COVERED Technical Memorandum

4. TITLE AND SUBTITLE

Spectroscopic Analysis of Perfluoropolyether Lubricant Degradation During Boundary Lubrication

6. AUTHOR(S)

Pilar Herrera-Fierro, Bradley A. Shogrin, and William R. Jones, Jr.

5. FUNDING NUMBERS

WU–505–63–5A

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

National Aeronautics and Space Administration
Lewis Research Center
Cleveland, Ohio 44135–3191

8. PERFORMING ORGANIZATION REPORT NUMBER

E–10381

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

National Aeronautics and Space Administration
Washington, D.C. 20546–0001

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

NASA TM–107299

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Unclassified - Unlimited
Subject Category 27

This publication is available from the NASA Center for AeroSpace Information, (301) 621–0390.

12b. DISTRIBUTION CODE

13. ABSTRACT (Maximum 200 words)

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14. SUBJECT TERMS

Perfluoropolyethers; Boundary lubrication; Degradation

15. NUMBER OF PAGES
23

16. PRICE CODE
A03

17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT

NSN 7540-01-280-5500