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The Performance of Perfluoropolyalkylether Oils Under Boundary Lubrication Conditions

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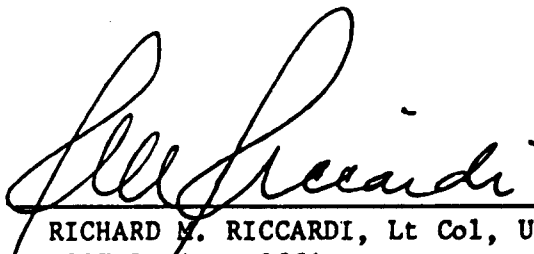Los Angeles, CA 90009-2960
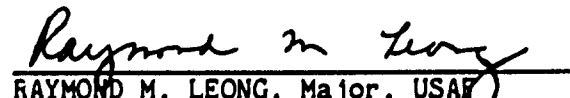
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Perfluoropolyalkylether (PFPE) oils and oil-based greases are being used with increasing frequency on spacecraft as a result of their purported outstanding properties. We demonstrated that, under boundary lubrication, these materials degrade by way of an autocatalytic mechanism. As a result, they have serious limitations in this tribological regime primarily because of the inability of the PFPE fluids to dissolve antiwear additives. To augment the fundamental studies, a series of wear tests comparing PFPE oils and greases with hydrocarbon fluids under boundary conditions were performed. As predicted, the performances of the PFPE fluids were below that of the hydrocarbon fluids. The results of the wear tests are discussed in terms of the effect of additives on the PFPE degradation mechanism and the overall usefulness of the PFPE fluids under boundary lubrication conditions.

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PREFACE

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I. INTRODUCTION

Perfluoropolyalkylether (PFPE) lubricants have become popular in recent years for spacecraft applications because of their very favorable properties, including low vapor pressure, high viscosity index, and general chemical inertness. Another property of PFPE materials that can, however, limit their usefulness is that they dissolve very few materials; i.e., conventional anti-oxidant and antiwear additives do not exhibit significant solubility. As a result, presently available additives must be suspended and not dissolved, which is not the most desirable situation. Without antiwear additives, the boundary lubrication performance of PFPEs is suspect.

The realization that soluble additives are needed for the PFPE materials has resulted in attempts to synthesize appropriate materials. Moderate success has been achieved in that some candidates have shown antioxidative capacity (1). Our interest is in the usefulness of the PFPE materials under boundary conditions and the limitations imposed by the space environment. Under space conditions, at low oxygen partial pressures, antioxidants are not critical. However, antiwear additives are required, but, to date, reasonable candidates have not been produced. In the case of the PFPE fluids, in the absence of additives, it is the chemical reactivity of the materials under the high temperatures generated in the contact area (2) that controls their performances.

The chemical reactivity of PFPE fluids has been addressed in the literature. Although the PFPE materials exhibit inertness at low temperatures, they have been shown to oxidatively decompose at higher temperatures and to corrode metal through the formation of reactive degradation products (3,4). In addition, Lewis acids, such as $AlCl_3$, have been shown to catalyze cleavage of the carbon-oxygen bonds resulting in degradation at moderate temperatures (5-7).

In a previous series of static experiments run under inert atmosphere in the absence of oxygen, the reactions of a branched PFPE fluid with FeF_3 were studied (8). FeF_3 is a weak Lewis acid that was suspected of being formed from the reaction of PFPE with iron under tribological conditions and that might participate catalytically in PFPE degradation. We determined that

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reaction occurred at a significant rate at temperatures 25°C below the onset of thermal degradation. Thermal degradation becomes significant at ~380°C. Infrared spectroscopy indicated that the reaction produced carbonyl-group-containing products which are consistent with ether bond cleavage (8).

In a separate set of experiments, FeF_x compounds, products of the reaction between PFPE and iron, were shown to be left on interacting bearing surfaces (9). Thus, the possible generation of FeF_3 as a catalytically active reaction product could lead to autocatalytic degradation of the PFPE material through Lewis-acid-assisted cleavage of the carbon-oxygen bond. Although the degradation of other hydrocarbon lubricants might commence at lower temperatures, the autocatalytic nature of the PFPE degradation and the inability to dissolve antiwear additives could make it less suitable than hydrocarbon lubricants under conditions in which asperity contact generates high localized temperatures and bare, reactive metal.

The results of the aforementioned mechanistic studies have suggested that the PFPE fluids should generally perform poorly under boundary conditions. We recently ran lubricant tests in which the performances of PFPE lubricants were compared to those of commercially available hydrocarbon lubricants under identical conditions of load, speed, and sample size. The tests were designed in conjunction with several satellite programs for use in lubricant selection and, as a result, must be interpreted with the understanding that they were not designed, nor were the lubricant and additive types and quantities selected, specifically to address the PFPE mechanistic issues. The results of these tests are the subject of this report.

II. EXPERIMENTAL

A. MATERIALS

The lubricants used in the tests were commercially available products. The lubricants used were as follows: a **linear** PFPE oil-based grease thickened with polytetrafluoroethylene telomer and with sodium nitrite as rust inhibitor; a **branched** PFPE oil; a paraffinic hydrocarbon oil with either lead naphthenate (PbNp) or antimony dialkyldithiocarbamate (SbDDC) as additives; a poly-alpha-olefin (PAO) oil-based grease with a bentonite clay thickener, a PAO oil with the SbDDC additive; a different PAO oil with either the SbDDC or PbNp additives; and a neopentylester oil. The PbNp and SbDDC additives were commercial products and were added at 5 percent (v/v) so that the relative performances of the lubricants with the additives could be assessed. The products were received, additive-free, from the suppliers except for the PFPE grease which contained sodium nitrite and the neopentylester oil which contained a proprietary antioxidant. The lubricants and their viscosity properties are listed in Tables 1 and 2.

B. WEAR-TEST FACILITY

The wear-test facility used in these tests is shown in Fig. 1 and has been described in detail previously (9). The basic concept is to force boundary conditions through the eccentric interaction between a flat disk that is rotated and the balls and lower raceway of a thrust bearing. The disks are 32-mm-diameter, 440C stainless steel polished to a mean surface finish of 0.3 μm. The thrust bearing is a standard F1 bearing in which the balls have been replaced with grade 10, 440C stainless steel balls. The ball diameter is 4.72 mm and the ball complement is 10. The eccentricity is produced by positioning the lower raceway, the commercial F1 bearing part, ~1 mm off-axis. This results in a calculated skid/roll ratio of ~ 0.16. The tests were run at 1750 rpm under ambient temperature conditions. No attempt was made to control the temperature.

The interaction members are housed in a vacuum chamber that is pumped by a turbomolecular pump so that low pressures, simulating space conditions in which the oxygen partial pressure is low, could be attained. With the PFPE

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Table 1. Selected Grease Properties for Set A Samples

BASE FLUID PROPERTY	VALUE
Specific gravity at 20°C (68°F)	1.80-1.90
Viscosity at:	
37.8°C (100°F), cSt	110-170
98.9°C (210°F), cSt	40-50
Viscosity index	340

GREASE PROPERTY	VALUE
Penetration at:	
25°C (77°F), unworked, mm^{-1}	270-295
25°C (77°F), worked, mm^{-1}	270-295
Dropping point, °C (°F)	182.2 (360)
Oil separation, 30 h at 204.4°C (400°F), wt. %	15
Evaporation loss at 204.4°C, wt. %	2

Table 2. Sample Identification and Viscosity Data for Set B Samples

NUMBER	TYPE	ADDITIVE	VISCOSITY, cST at 37.8°C	VISCOSITY INDEX (100°F)
1	Paraffinic hydrocarbon	PbNp[*]	90[‡]	100
2	Paraffinic hydrocarbon	SbDDC[†]	90[‡]	100
3	PAO[§]	SbDDC	440	145
4	PAO	PbNp	98-102[‡]	135
5	PAO	SbDDC	98-102[‡]	135
6	PAO oil-based grease	-	32[∥]	132
7	Neopentylester	-	20	139
8	PFPE[#]	-	85	113

[*]Lead naphthenate

[†]Antimony dialkyldithiocarbamate

[‡]Viscosity at 40°C (104°F)

[§]Poly-alpha-olefin

[∥]Viscosity of base oil

[#]Perfluoropolyalkylether
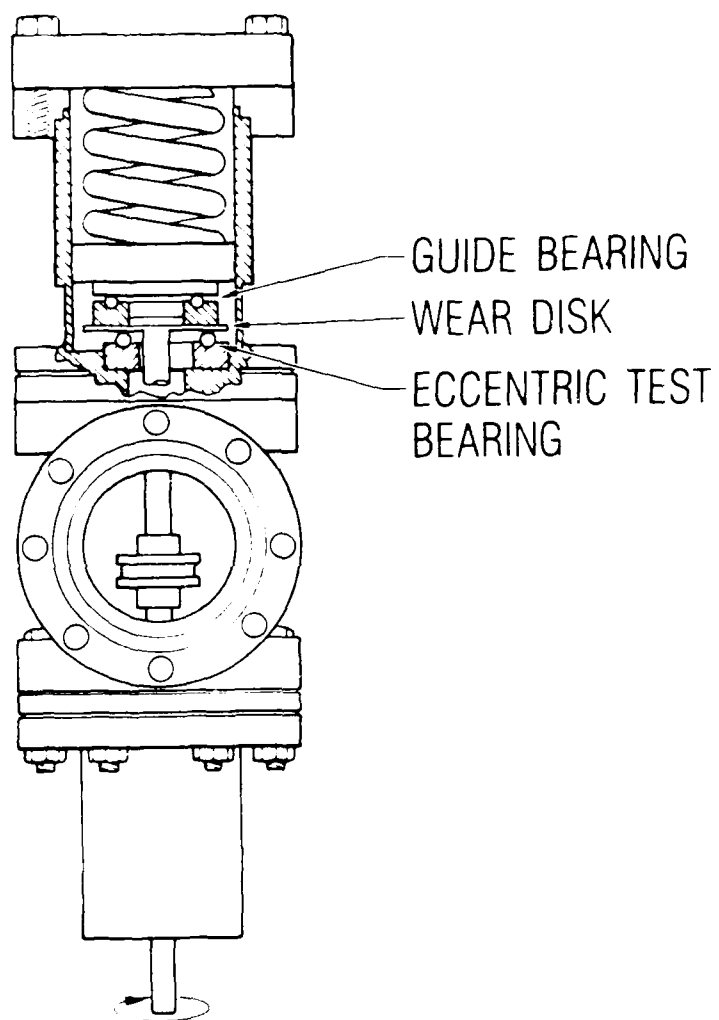
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GUIDE BEARING
WEAR DISK
ECCENTRIC TEST
BEARING

Fig. 1. Eccentric Bearing Wear-Test Facility

fluids, a liquid nitrogen trap was used to intercept any potentially hazard-
ous, volatile degradation products. With the pumping capacity of the liquid-
nitrogen-cooled trap, pressures of ~1.3 × 10^{-5} Pa were attained. (The
excellent vapor pressure properties of the PFPE fluids allowed the use of low
pressures without concern for significant lubricant loss through evaporation.)
For the other materials, the facility was used without the liquid nitrogen
trap, and pressures of 1.3 × 10^{-3} to 1.3 × 10^{-2} Pa were routinely achieved.
Several of the oils tested were relatively volatile and their vapor pressure
most likely resulted in the higher pressures, ~ 1.3 × 10^{-2} Pa, observed when
they were tested.

C. PROCEDURE

Two sets of tests were run: set A, in which the performance of the
PFPE grease was compared to the performance of the PFPE grease contaminated
with a PAO oil containing the SbDDC additive (the contaminant corresponded to
5 percent of the total lubricant); and set B, in which the performance of the
branched PFPE oil was compared to the performance of the other lubricants
(with the exception of the PFPE grease).

For the tests in set A, in which the PFPE grease was compared to the
contaminated PFPE grease, the load was varied between 89.0 N (20 lb) and
225.5 N (50 lb). These loads resulted in calculated Hertzian stress values of
1.00 × 10^9 Pa (1.45 × 10^5 psi) to 1.36 × 10^9 Pa (1.97 × 10^5 psi). In the
set A tests, 200 mg of grease was used as the sample size. The contaminated
grease samples consisted of 200 mg of grease with 10 microliters of contami-
nant oil. To be certain that the contaminant oil contacted the test surfaces
and did not mix into the grease prior to testing, the grease was first applied
to the thrust bearing raceway, followed by placement of the retainer and
balls, and then application of the contaminant oil onto the bearing balls.
The disk was then located on top of the balls, ensuring that contact between
the oil and the balls and disk occurred at the beginning of testing. [This
method of application was dictated by a program requirement. The reviewers
felt that applying the PAO to the system in this manner would result in the
PFPE grease acting as a "barrier film" trapping the PAO oil in the interaction
area and that intimate mixing of the lubricants would be preferred. While
this may be true, I feel that intimate mixing of the PAO into the PFPE grease

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would lead to the same results because a microemulsion would be formed in which the reactive additives would be transported to the interacting surfaces during running. Previously, we have demonstrated that a PFPE oil intimately mixed with a hydrocarbon oil contaminant performed significantly better under boundary conditions than did the PFPE oil by itself (9)].

The set B tests were run using a load of 133.5 N (30 lb), which resulted in a calculated Hertzian stress of 1.15×10^9 Pa (1.66×10^5 psi). The lubricant quantity was 10 microliters, except for sample 6 in which 15 mg of grease was used.

The tests were terminated when the torque increased to 150 percent of the initial steady-state torque value. (The 150 percent torque increase was arbitrarily chosen to represent system failure.) The torque of the system was measured indirectly by using the current drawn by the motor, measured as the potential drop across a precision 1-ohm resistor in series with the motor. The "torque" of the system in the absence of the wear members was determined independently so that its value could be subtracted from the total potential drop. The difference is due to the wear members. (The torque of the guide bearing in the system was considered to be insignificant compared to the wear members.) The test parameters are given in Table 3.

Table 3. Wear Test Conditions

	SET A	SET B
Speed (rpm)	1750	1750
Axial load (N)	89.0 - 225.5	133.5
Hertzian stress (Pa)	$1.00 \times 10^9 - 1.36 \times 10^9$	1.15×10^9
Pressure (Pa)	1×10^{-5}	$1 \times 10^{-5} - 1 \times 10^{-2}$
Test temperature[*]	Ambient	Ambient

[*]The temperature was not controlled.

III. RESULTS AND DISCUSSION

A. SET A - CONTAMINATED PFPE GREASE PERFORMANCE

The results of the set A tests are given in Table 4 and Fig. 2. It is clear that the PFPE grease, contaminated with the PAO oil, outperformed the uncontaminated PFPE grease by approximately a factor of 2 over the range of loads tested. Despite the fact that the wear-test facility primarily promotes system failure through lubricant starvation, the substantial grease sample size, 200 mg, would have resulted in a significant quantity of oil supplied to the interaction area so that early lubricant starvation was unlikely. It is significant to note that the wear life of the contaminated grease was essentially the same as that for the contaminant oil by itself under the identical test conditions (set B). Despite the fact that a significant quantity of PFPE oil was available at the beginning and during the testing, the contaminant oil with its additives controlled the lubrication of the system.

B. SET B - COMPARISONS OF OIL PERFORMANCE

The results for the set B tests are given in Table 5 and Fig. 3. Three conclusions are obvious from the examination of these data: The synthetic hydrocarbons, samples 3-7, outperformed both the PFPE oil, sample 8, and the petroleum-based hydrocarbon oils, samples 1 and 2; the SbDDC additive performed better than the PbNp additive; and the best performance was given by the PAO oil-based grease. A possible explanation is that the petroleum based oils have unsaturated aliphatic and aromatic impurities that are more reactive than the base saturated oils and could lead to premature degradation.

The SbDDC additive outperformed the PbNp additive by approximately a factor of 2 for the two oils used in the comparison. Without knowledge of the chemistry at the interface between lubricant and metal surfaces, the reasons for this difference in performance cannot be determined.

The outstanding performance of the PAO oil-based grease is really an anomaly of the test facility. Since lubricant starvation is used to bring about failure, an oil cannot be compared directly with a grease. While the oil can be removed by centrifugal action, the reservoir capability of the

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Table 4. Wear Test Results for Set A Tests[*]

APPLIED LOAD (N)	HERTZIAN STRESS (Pa)	TOTAL REVOLUTIONS	
		CONTAMINATED	UNCONTAMINATED
89.0	1.00×10^9	$6.54 \pm 2.75 \times 10^6$	$6.64 \pm 1.55 \times 10^6$
133.5	1.15	7.79 ± 4.36	1.43 ± 0.74
178.0	1.26	3.14 ± 1.37	1.40 ± 0.63
222.5	1.36	1.74 ± 0.21	0.76 ± 0.03

[*]The wear life values are reported as the average of
three runs ± one standard deviation.

Table 5. Wear Test Data for Set B Tests

NUMBER	AVERAGE TOTAL REVOLUTIONS[*]	STANDARD DEVIATION
1	0.96×10^6	0.25×10^6
2	1.39	1.05
3	2.63	0.57
4	1.63	0.76
5	4.27	3.92
6	9.51	5.84
7	4.42	1.56
8	0.85	0.64

[*]The wear life values are reported as the
average of three runs ± one standard
deviation.

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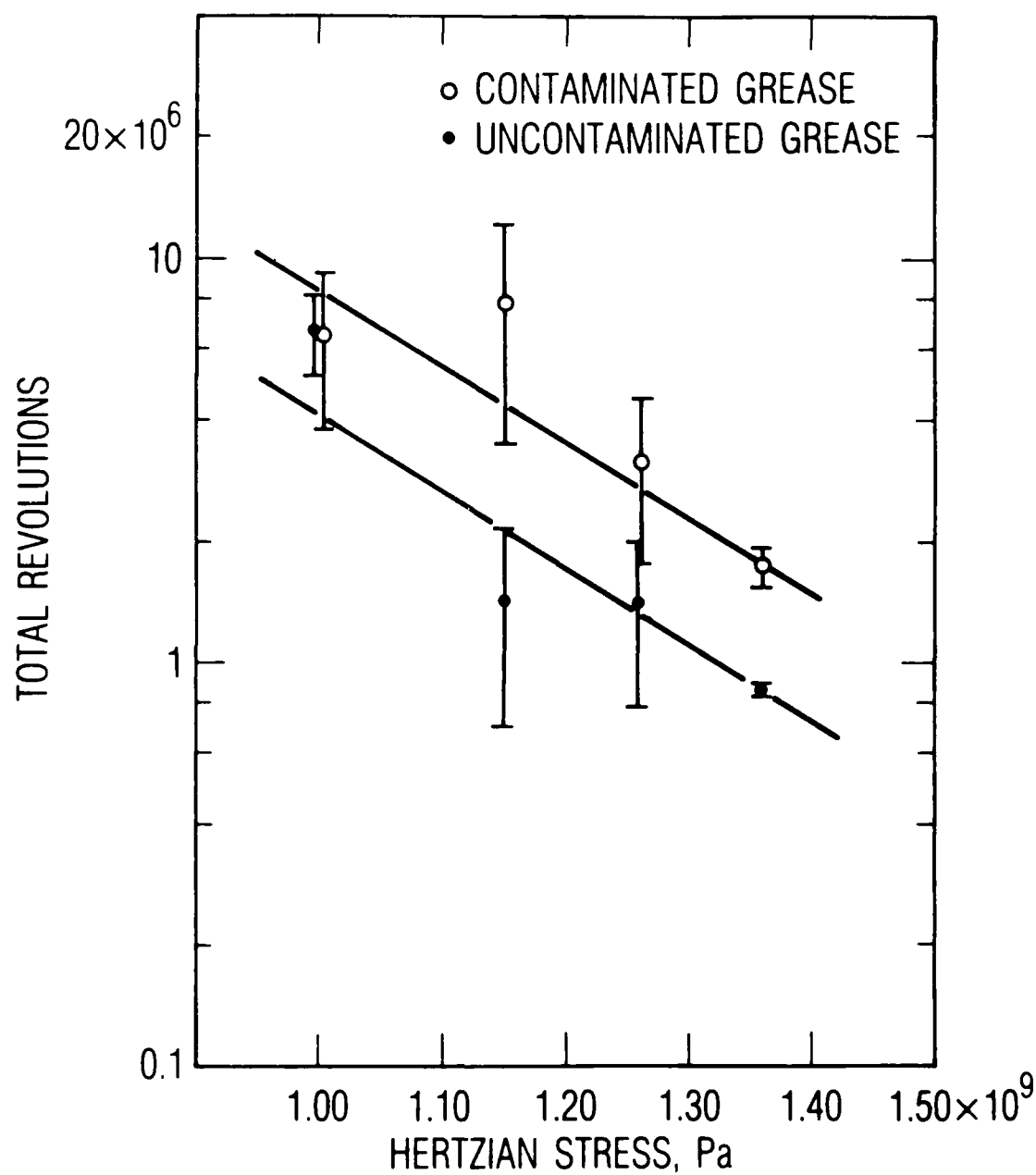
Fig. 2. Wear Life vs Hertzian Stress for
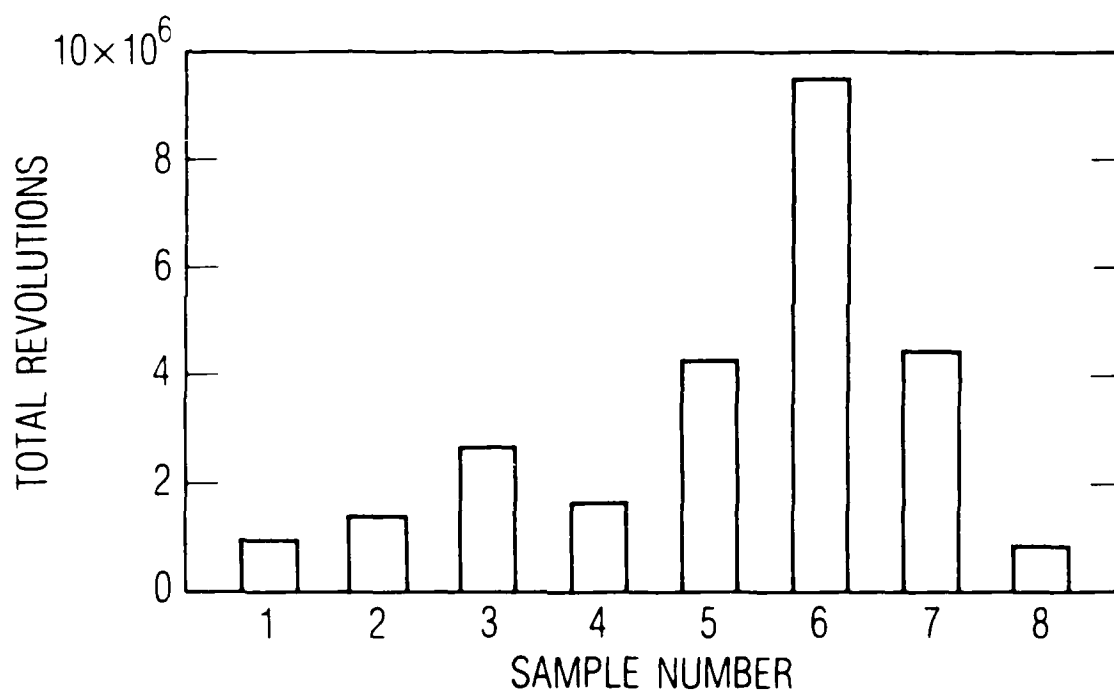Contaminated and Uncontaminated
PFPE Grease, Set A

Fig. 3.   Wear Life vs Sample Number for
Set B Samples

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grease allows for an available oil supply for use in the interaction area after the initial oil is removed. The performance of the grease is considered to be similar to that of the other PAO oils.

C. MECHANISTIC CONSIDERATIONS

These wear tests results are pertinent to our hypothesized PFPE degradation mechanism because, in the presence of additives from contaminant hydrocarbon oil, the degradation is inhibited. This can be explained as follows: With hydrocarbon lubricants, and also with the hydrocarbon contaminants in the PFPE grease, it is not the oil that does the work in boundary conditions, but rather the additives. Apparently, the hydrocarbon oil acts as a carrier of the antiwear additive, transporting it to the bearing surfaces. At the metal surface, the additive displaces any fluorocarbon lubricant that is present. Although it is a very low energy material and will substantially lower the energy of the metal surfaces, the PFPE does not adhere or bond strongly to the bearing surfaces. This conclusion is supported by the results from the set B tests. In these tests, 10 microliters of hydrocarbon oil was used as the sample size. This corresponds to the same quantity of PAO oil that was added as the contaminant in the set A tests. Similar wear lives were achieved in the presence or absence of the PFPE grease under identical test conditions. The implication is that there was little or no contribution to the lubrication from the PFPE material. Thus, the resultant "shielding" of the surface by the antiwear additive does not allow the PFPE fluid to be in intimate contact with bare metal and, as a result, the rate of degradation of the PFPE material is reduced through inhibition of the formation of the iron fluoride, Lewis acid, catalyst.

The performance of the neopentylester oil, sample 7, appears anomalous in that no antiwear additives were used. However, it was chosen for the testing because it is suspected of forming its own antiwear agents upon reaction with iron. Neopentylesters have been shown to decompose at elevated temperatures, accompanied by an increase in acidity (10). The fatty acids that result could react with the metal surfaces, forming metal soaps which have been shown to provide boundary protection (11). At the present time, in the absence of other data, this is the best explanation that can be offered.

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In considering these results, it must be kept in mind that they were obtained for a specific range of Hertzian stresses and at specific sample sizes. Also, the failures generated in the eccentric bearing wear-test facility result from forced lubricant starvation by the absence of lubricant reservoirs and the "slinging" action of the rotating disk and the severe wear conditions resulting from the large sliding component. Other test conditions might result in different ordering of lubricant performance. For example, the short duration of the testing does not allow the high vapor pressure lubricants to evaporate significantly. Thus, these materials might give relatively longer wear lives in our facility than would be experienced under conditions that more closely simulate long term applications. However, because evaporation of the lubricant is not a consideration, these tests can give an indication of the "intrinsic" performance of the lubricants in the absence of oxygen, i.e., under vacuum.

The results of these tests also underscore the importance of antiwear additives in boundary lubrication. Whether or not the PFPE lubricants would have outperformed additive-free hydrocarbon lubricants is a point of conjecture that our testing did not address.

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IV. CONCLUSIONS

The results of the wear tests discussed herein lead to the following major conclusion: Synthetic hydrocarbon lubricants with additives outperform PFPE fluids under identical wear-test conditions. Consistent with our degradation hypothesis (9), the poor performance of PFPE fluids stems from their inability to dissolve additives in the parent fluids and the possibility of autocatalytic material degradation.

These conclusions are based upon results obtained on our rather specialized wear-test apparatus, and load and bearing surface finish conditions can probably be found in which adequate performance of PFPE fluids can be attained (most likely lightly loaded systems). However, the thrust of these results is not only to support the degradation hypothesis for the PFPE materials from a scientific standpoint but also to emphasize the importance of understanding lubrication systems and the tribological conditions in an application before making lubricant choices. In the past, too many lubricant selections have been based on inadequate performance data and hearsay pertaining to the general value of a lubricant. In expensive programs, such as satellite programs, the cost of requalifying a lubricant following failure of an inappropriate lubricant choice and the cost of the replacing the equipment can be staggering.

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