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THERMAL AND OXIDATIVE STABILITY OF A
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STABILITY OF A FLUORINATED POLYETHER AND
A PERFLUOROPOLYETHER TRIAZINE

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ABSTRACT

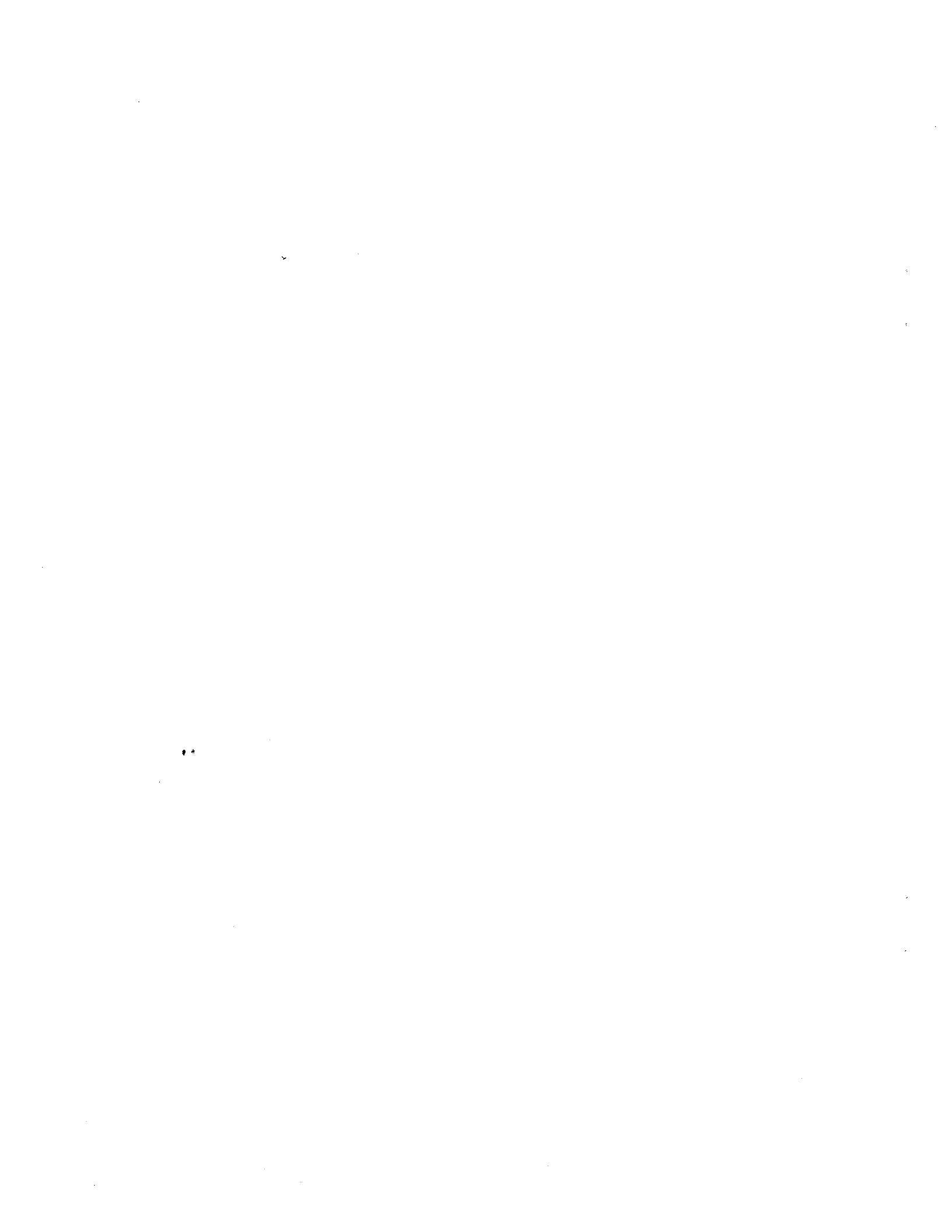
Boundary lubricating characteristics, thermal stability, and oxidation-corrosion stability were determined for a fluorinated polyether and a perfluoropolyether triazine. A ball-on-disk apparatus, a tensimeter, and oxidation-corrosion apparatus were used. Results were compared to data for a polyphenyl ether and a C-ether. The polyether and triazine yielded better boundary lubricating characteristics than either the polyphenyl ether or C-ether. The polyphenyl ether had the greatest thermal stability (443° C) while the other fluids had stabilities in the range 389° to 397° C. Oxidation-corrosion results indicated the following order of stabilities: perfluoropolyether triazine > polyphenyl ether > C-ether > fluorinated polyether.

INTRODUCTION

Advanced aircraft and future aerospace systems will place increased thermal and oxidative stresses on lubricants and hydraulic fluids. Maximum fluid temperatures in excess of 260° C (500° F) have been estimated for future applications (1-6). At these elevated temperatures, fluids must also provide effective lubrication for bearings and hydraulic system components.

State-of-the-art fluids (esters, hydrocarbons, silicones, fluorinated polyethers, C-ethers and polyphenyl ethers) have one or more deficiencies which would limit or prevent their use at high temperatures (i. e., 260° C). Esters, hydrocarbons, and silicones are not oxidatively stable at 260° C (7). The C-ethers have excellent thermal stability (390° C), good oxidation stability to 260° C and lower pour points

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(-29° C) than the polyphenyl ethers (5° C) (8, 9). However, both the C-ethers and polyphenyl ethers are rather poor boundary lubricants (10, 11, 12). Both fluids have also exhibited poor wetting characteristics (13, 14). The fluorinated polyethers have excellent thermal and oxidative stability to 370° C (15) but have experienced corrosion problems with certain ferrous and titanium alloys above 260° C (16).

A combination of the heterocyclic *s*-triazine system with fluorinated polyether substituents has yielded a variety of potential high temperature lubricants and/or hydraulic fluids (17). By varying the ratio of carbon to oxygen in the polyether substituents, wide variations in physical properties can be obtained. However, since these fluids are available only in research quantities (typically <100 g), little is known about their thermal and oxidative stability, and boundary lubrication properties.

The objectives of this investigation were twofold: (1) to determine the boundary lubrication characteristics of a perfluoropolyether substituted *s*-triazine and a commercially available fluorinated polyether and to compare these results with those previously obtained with both a C-ether (12) and a polyphenyl ether (18), and (2) to compare the thermal and oxidation-corrosion characteristics of the above noted fluids.

APPARATUS AND PROCEDURE

Sliding Friction Apparatus

The ball-on-disk sliding friction apparatus is shown in Fig. 1. The test specimens were contained inside a stainless-steel chamber. A stationary 0.476-centimeter-radius ball was placed in sliding contact with a rotating 6.3-centimeter-diameter disk. A sliding speed of 17 meters per minute (100 rpm) was maintained. A normal load of 1 kilogram (initial Hz stress of 10^9 Pa) was applied with a deadweight. Balls and disks were made of CVM M-50 steel having a Rockwell C hardness of 62 to 64. The apparatus and procedure have been previously described (12, 13).

Frictional force and bulk lubricant temperature were recorded continuously. Disk temperature was monitored continuously. Each experiment was terminated after a 25-minute duration and the rider (ball) wear scar was measured and recorded. Tests were normally run in triplicate and an average calculated for plotting.

Tensimeter

The tensimeter apparatus is shown in Fig. 2. The basic function of the tensimeter consists of heating a liquid sample and of plotting either the vapor pressure, or the isothermal time rate of increase of vapor pressure (due to thermal decomposition), as a function of temperature.

The sample cell is a glass bulb having a 5-milliliter ($5 \times 10^{-6} \text{ m}^3$) volume, with a stem extending through the oven wall to a valve and pressure transducer mounted outside the oven. Three to four milliliters (3×10^{-6} to $4 \times 10^{-6} \text{ m}^3$) of test fluid are placed in the sample cell. The cell is attached to the cell assembly and the fluid is degassed and refluxed under vacuum. The cell assembly is then placed in the oven. The sample is heated to an initial temperature about 50° C (90° F) below the suspected decomposition temperature. After a 5-minute stabilization period, the increase in vapor pressure, if any, is recorded as a vertical bar during a fixed time interval. Then the programmer raises the sample temperature by a preset amount, usually 5° C (9° F), and the previous process is repeated.

A typical plot appears in Fig. 3. This is essentially a plot of the logarithm of the isothermal rate of vapor pressure increase as a function of reciprocal absolute temperature. A straight line is drawn connecting the tops of the recorded bars at the higher temperatures. The intersection with the temperature reference axis is the thermal decomposition temperature T_D . This temperature axis corresponds to a pressure rise of 50 torr per hour, which is the definition of the T_D .

Oxidation-Corrosion Apparatus

The oxidation-corrosion glass apparatus is shown schematically in Fig. 4. A flow rate of 20 liters per hour of dry air was maintained for 24 hours. The metal corrosion coupons were: Ti(6Al4V), Al(2024), AISI M-10, Ag, and 301 stainless steel. Tests were run with and without a reflux condenser. A 20 milliliter test sample was used. More detailed information about the apparatus and procedure has been previously described (19).

Pre-test and post-test fluid characterizations were performed to determine fluid viscosity changes at 38° C (100° F), fluid weight loss (weight percent) and acid number changes (mg of KOH/g of fluid). In addition, the weight change per unit area

of the metal coupons (mg/cm^2) was determined. Maximum allowable weight change is $\pm 0.2 \text{ mg}/\text{cm}^2$. This upper limit was determined from corrosion rates measured in gas turbine engine tests.

Experimental Lubricants

Typical properties of the two experimental fluids along with the two reference fluids appear in table I. The fluorinated polyether is one member of a class of commercially available fluids (hydraulic fluid grade). These fluids are based on the polymerization of hexafluoropropylene oxide (HFPO). A higher viscosity grade of the fluorinated polyether, but chemically identical, was used for the oxidation-corrosion tests only. The second experimental fluid is a symmetrical triazine having perfluoropolyether substituents derived from HFPO. Members of this fluid class have exhibited excellent high temperature properties (20). However, they are available only in research quantities. Therefore, the particular member of this fluid class chosen for these tests was done so because of its ease of synthesis and high yield. Its physical properties are not optimum (such as viscosity, pour point, volatility, etc.). The chemical name for this fluid is 2,4,6-Tris [2,5,8,11,14-pentaoxa - 1,4,7,10,13-penta (trifluoromethyl) - eicosylfluoroheptadecyl] - 1,3,5-triazine. Its chemical structure and that of the fluorinated polyether appear in Fig. 5. Neither fluid contained any additives.

RESULTS

Friction and Wear

Average rider (ball) wear rate and the coefficient of friction for the fluorinated polyether and perfluoropolyether triazine as a function of disk temperature appear in Fig. 6. In general, the wear rate increased as disk temperature increased. However, in the temperature range, 100° to 300° C, the wear rates for both fluids were reasonably level. The fluorinated polyether exhibited a level coefficient of friction as a function of temperature (0.13 to 0.16). A gradual increase in the coefficient of friction as disk temperature increased was noted for the triazine (except at the highest disk temperature, 300° C).

Friction as a function of time for the fluorinated polyether and triazine at three disk temperatures appear in Fig. 7. Unusually smooth friction traces were obtained

for both fluids. Only at 300° C with the triazine did erratic friction take place. After rig disassembly, it was evident that this behavior was a result of the high volatility of the triazine at 300° C which caused a loss of the fluid. In separate experiments, thermogravimetric analysis indicated a 100 percent weight loss for this fluid at 315° C.

The wear results of Fig. 6 are replotted in Fig. 8 along with the reference data for a five-ring polyphenyl ether (18) and a C-ether base fluid (12). In general, both the fluorinated polyether and triazine yielded lower wear rates than either of the aromatic reference fluids over almost the entire temperature range.

Thermal Stability

Table II contains thermal decomposition temperatures (T_D) for the fluorinated polyether, triazine, and two reference fluids, five-ring polyphenyl ether and the C-ether base fluid. The polyphenyl ether exhibited the highest T_D of 443° C. Intermediate values of 397°, 390°, and 389° C were obtained for the triazine, C-ether and fluorinated polyether, respectively.

Oxidation-Corrosion Stability

Oxidation-corrosion data for all four fluids at temperatures of 260°, 288°, and 316° C appear in table III in overboard configuration (no reflux condenser). Similar data obtained at 316° C with a reflux condenser is shown in table IV. In the first configuration, the fluid stabilities are in the order: triazine > polyphenyl ether > C-ether \cong fluorinated polyether. In the latter configuration a similar sequence was obtained: triazine > polyphenyl ether > C-ether > fluorinated polyether.

DISCUSSION

Friction and Wear

As stated previously, the two fluorinated fluids (s-triazine and polyether) not only yielded lower wear rates than the polyphenyl ether and C-ether reference fluids over most of the temperature range, but also exhibited unusually smooth friction-time traces. This is to be compared to the higher, broader, and sometimes erratic friction behavior of polyphenyl ethers (13) and C-ethers (12). For example, C-ether friction traces at 100°, 200°, and 300° C are compared to the fluorinated polyether data (Fig. 7) in Fig. 9. It is apparent that the fluorinated fluids generate a boundary film which yields a smooth coefficient of friction throughout the test.

In addition, copious amounts of "friction polymer" or "surface resin" are generated by polyphenyl ethers (21) and C-ethers (18). An example of this material is shown in Fig. 10(a) around the wear scar for a C-ether fluid run at 225^o in dry air. It is felt that the production of this material is related to the tendency of aromatics to produce free radicals or possibly radical anions (21). The latter may be sufficiently corrosive to be detrimental to the wear process (22).

Neither of the fluorinated fluids generated any such material in these wear tests. An example of a wear scar obtained with the triazine at 200^o C is shown in Fig. 10(b). No "friction polymer" or other debris was noted around this or any other triazine wear scar in this study even though this fluid does have an aromatic nucleus.

Thermal Stability

These thermal stability tests, which take place in the absence of oxygen and catalytic metals measure the inherent stability of the chemical bonds in the lubricant. The T_D is an idealized or maximum temperature at which a lubricant could operate. The high T_D 's obtained for the two experimental fluids (389^o to 443^o C) attest to the stability of carbon-fluorine bonding and of the aromatic rings.

Oxidation-Corrosion Stability

A direct comparison of the data and subsequent ranking of the experimental fluids is difficult. This is due to the fact that two distinct chemical classes are being compared (aromatics and highly fluorinated structures). Because of these chemical differences, these fluids undergo significantly different degradation mechanisms under autoxidative conditions at elevated temperatures. Smith et al. (23) reported extensively on the oxidation mechanism of polyphenyl ethers. It is probable that the C-ethers (which are a mixture of aromatic sulfides and are therefore structurally similar to the polyphenyl ethers) degrade similarly. Gumprecht (24) described the autoxidative and metal-catalyzed oxidation of fluorinated polyether (HFPO type) fluids. Recently, Paciorek and Kratzer (25) demonstrated that one must take into account the small amounts of incompletely fluorinated polyalkylether polymers that occur during the synthesis of this fluid. These materials are significantly less stable than the totally fluorinated species. In the case of the perfluoropolyether substituted triazine, there is no possibility of incomplete fluorination. In addition, the s-triazine

ring is extremely stable to oxygen at high temperatures. Therefore, it is felt, that the mechanism proposed by Gumprecht for the HFPO fluorinated polyethers is also applicable to the HFPO substituted s-triazines.

When comparing changes in some of the fluid properties, the nature of the fluid must be considered. For example, if the test fluid consists of a distillation fraction containing a wide range of molecular weights, viscosity increase accompanied by a large fluid weight loss when tested without a reflux condenser, may indicate volatilization of the lower molecular weight components leaving behind the higher molecular weight, more viscous species. The fluorinated polyether is an example. The use of a reflux condenser minimizes this effect. Therefore, the results with and without the condenser will be considered separately.

Overboard Configuration (No Reflux Condenser)

In this configuration, the triazine appears to be the most stable with the fluid property changes being related only to volatilization. The polyphenyl ether was the next most stable fluid in terms of property changes and metal compatibility to 316° C. No distinction can be made between the fluorinated polyether and C-ether because of their high volatility. The C-ether did exceed the maximum allowable metal corrosion limit at 260° C and the polyether at 316° C. The reason that the C-ether exhibited greater metal corrosion at 260° C than at 288° or 316° C is not known. One might speculate that the degradation products responsible for the corrosion would have a higher residence time at the lower temperature allowing more interaction with the metals. At the high temperatures these products may volatilize before they can react.

Reflux Configuration

Tests with the water cooled reflux condenser were conducted at 316° C only. Again, the triazine appeared superior with the polyphenyl ether close behind. The fluorinated polyether yielded significantly smaller changes in fluid properties but the magnitude of the metal corrosion would cause it to be ranked behind the C-ether. Therefore, taking into account both test configurations the following oxidation-corrosion stability ranking was obtained: perfluoropolyether triazine > five-ring polyphenyl ether > C-ether > fluorinated polyether.

SUMMARY OF RESULTS

Boundary lubricating characteristics to 300° C, thermal stability, and oxidation-corrosion stability were determined for a fluorinated polyether and a perfluoropolyether triazine. A sliding ball-on-disk apparatus, a tensimeter (isoteniscope) and a micro oxidation-corrosion apparatus were used. Results were compared to data previously obtained for a five-ring polyphenyl ether and a C-ether. The major results were:

1. The fluorinated polyether and the perfluoropolyether triazine yielded better boundary lubricating characteristics than the polyphenyl ether or the C-ether.
2. The thermal stabilities of the polyphenyl ether, perfluoropolyether triazine, C-ether, and fluorinated polyether were 443°, 397°, 390°, and 389° C, respectively.
3. In general, the order of oxidation-corrosion stability was perfluoropolyether triazine > polyphenyl ether > C-ether > fluorinated polyether.

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TABLE I. - TYPICAL PROPERTIES OF EXPERIMENTAL AND REFERENCE FLUIDS

Property	Reference fluids		Experimental fluids	
	Five-ring polyphenyl ether	C-ether	Fluorinated polyether	Perfluoropolyether triazine
Kinematic viscosity, m^2/sec (cS) at: 0° C	-----	-----	1.2×10^{-3} (1200)	7×10^{-4} (700)
38° C	3.6×10^{-4} (360)	2.5×10^{-5} (25)	9.7×10^{-5} (97)	6×10^{-5} (60)
99° C	1.3×10^{-5} (13)	4.1×10^{-6} (4.1)	1.1×10^{-5} (11)	7×10^{-6} (7)
204° C	-----	-----	1.9×10^{-6} (1.9)	-----
260° C	-----	-----	-----	6×10^{-7} (0.6)
300° C	9.5×10^{-7} (0.95)	6.9×10^{-7} (0.69)	-----	-----
Pour point, °C	5	-29	-40	-21
Flash point, °C	288	230	None	None
Fire point, °C	350	285	None	None
Density at 38° C, kg/m^3 (g/ml)	1.19 (1.19×10^3)	1.18 (1.18×10^3)	1.89 (1.89×10^3)	-----
Vapor pressure at 371° C, torr	28	140	-----	-----
ASTM slope	-----	-----	0.67	0.76
Surface tension at 23° C, dynes/cm (N/cm)	46.0 (4.6×10^{-4})	44.8 (4.48×10^{-4})	17.3 (1.73×10^{-4})	-----

TABLE II. - THERMAL DECOMPOSITION
TEMPERATURE (T_D) FOR SEVERAL FLUIDS

Fluid	Decomposition temperature, °C
Fluorinated polyether	389
C-ether	390
Perfluoropolyether triazine	397
Five-ring polyphenyl ether	443

TABLE III. - OXIDATION-CORROSION TEST DATA (NO REFLUX CONDENSER)

[20 Liters/hr, dry air, 24-hr test.]

Fluid	Temperature, °C	Δ Vis. 100° F, %	Δ Acid NR., mg/g	Fluid loss, wt %	Metal weight change, mg/cm ²				
					Ti(6Al4V)	Al(2024)	M-10	Ag	301
C-ether	260	+11.3	0.0	10.2	^a -1.340	-0.098	-0.118	^a -0.238	-0.112
		+9.7	0.0	8.2	-0.086	-0.118	-0.146	^a -0.220	-0.126
	288	+13.7	0.0	6.0	-0.008	-0.022	-0.018	-0.134	-0.014
		+23.1	0.0	13.1	-0.002	-0.030	-0.014	-0.144	-0.016
	316	+95.1	(b)	37.0	+0.014	+0.088	+0.024	-0.118	+0.050
		+80.6	(b)	33.5	-0.006	+0.092	+0.026	-0.184	+0.044
Fluorinated polyether	260	+56.0	0.0	24.3	+0.006	+0.026	+0.040	+0.026	+0.052
		+55.8	0.0	24.0	+0.038	-0.020	+0.034	+0.038	+0.056
	288	+107.3	0.0	42.2	+0.060	+0.046	+0.018	-0.070	-0.016
		+90.8	<0.1	36.5	+0.084	+0.010	+0.016	-0.078	-0.008
	316	+12.1	(b)	6.3	^a +0.230	+0.066	+0.124	-0.154	+0.040
		+14.5	(b)	8.2	^a +0.262	+0.094	+0.098	-0.158	+0.026
Five-ring polyphenyl ether	260	+4.6	0.0	3.0	0.00	-0.020	+0.020	+0.004	0.00
		+4.5	0.0	2.4	+0.006	-0.044	0.00	-0.028	+0.002
	288	+5.4	(b)	9.0	+0.02	0.00	+0.01	-0.03	+0.05
		+6.6	(b)	9.6	0.0	0.00	+0.02	0.00	+0.01
	316	+12.3	(b)	2.4	-0.02	-0.01	+0.01	-0.02	0.00
		+11.8	(b)	3.6	-0.02	-0.01	+0.02	-0.02	+0.01
Perfluoropolyether triazine	316	-1.1	<0.1	32.7	+0.05	+0.03	+0.05	^a -0.7	+0.03
		-1.1	<0.1	34.2	+0.05	0.00	+0.03	-0.09	-0.02

^aExceeds 0.2 mg/cm².^bNot determined.

TABLE IV. - OXIDATION-CORROSION TEST DATA (WITH REFLUX CONDENSER)

[20 Liters/hr dry air 24-hr test.]

Fluid	Temperature, °C	Δ Vis. 100° R, %	Δ Acid NR., mg/g	Fluid loss, wt %	Metal weight change, mg/cm ²				
					Ti(6Al4V)	Al(2024)	M-10	Ag	301
C-ether	316 ↓	+29.0	(a)	12.4	-0.032	+0.020	-0.022	-0.130	+0.004
		+31.5	(a)	15.5	-0.028	+0.028	+0.006	-0.166	-0.002
Fluorinated polyether		+7.3	(a)	9.3	^b +0.254	+0.176	^b +0.228	-0.142	+0.020
		+9.1	(a)	9.0	^b +0.220	+0.120	^b +0.272	-0.038	+0.024
Five-ring polyphenyl ether		+27.1	(a)	2.1	+0.014	+0.016	+0.028	+0.058	-0.006
		+14.0	(a)	2.5	+0.052	+0.048	+0.020	-0.006	+0.022
Perfluoropolyether triazine		+1.0	(a)	16.6	+0.016	+0.050	-0.006	-0.184	+0.194
		+2.2	(a)	19.3	+0.012	+0.078	+0.002	-0.120	-0.004

^aNot determined.

^bExceeds 0.2 mg/cm².

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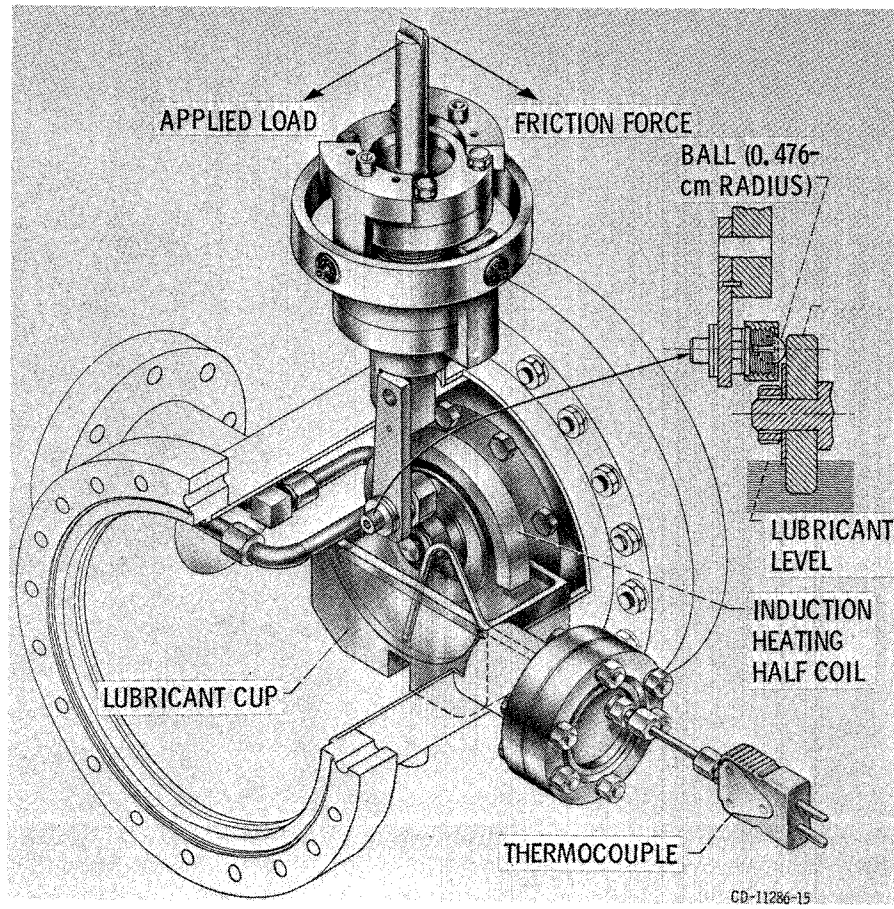


Figure 1. - Friction and wear apparatus

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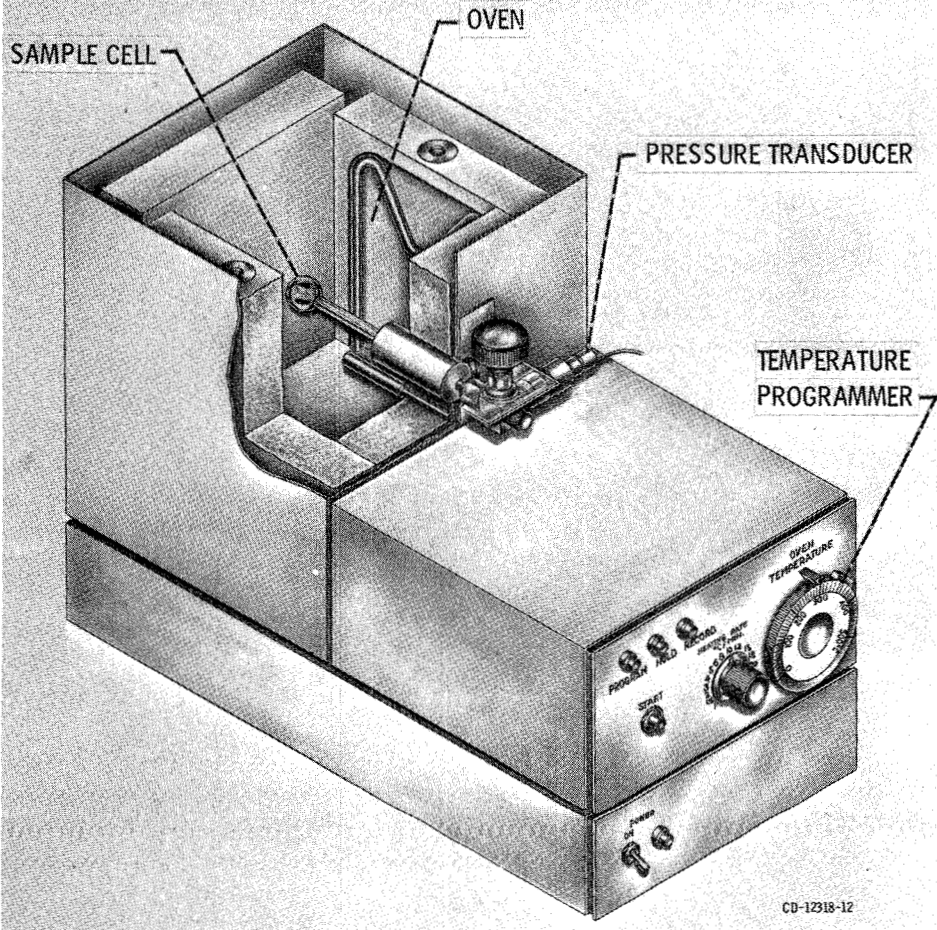


Figure 2. - Recording tensimeter.

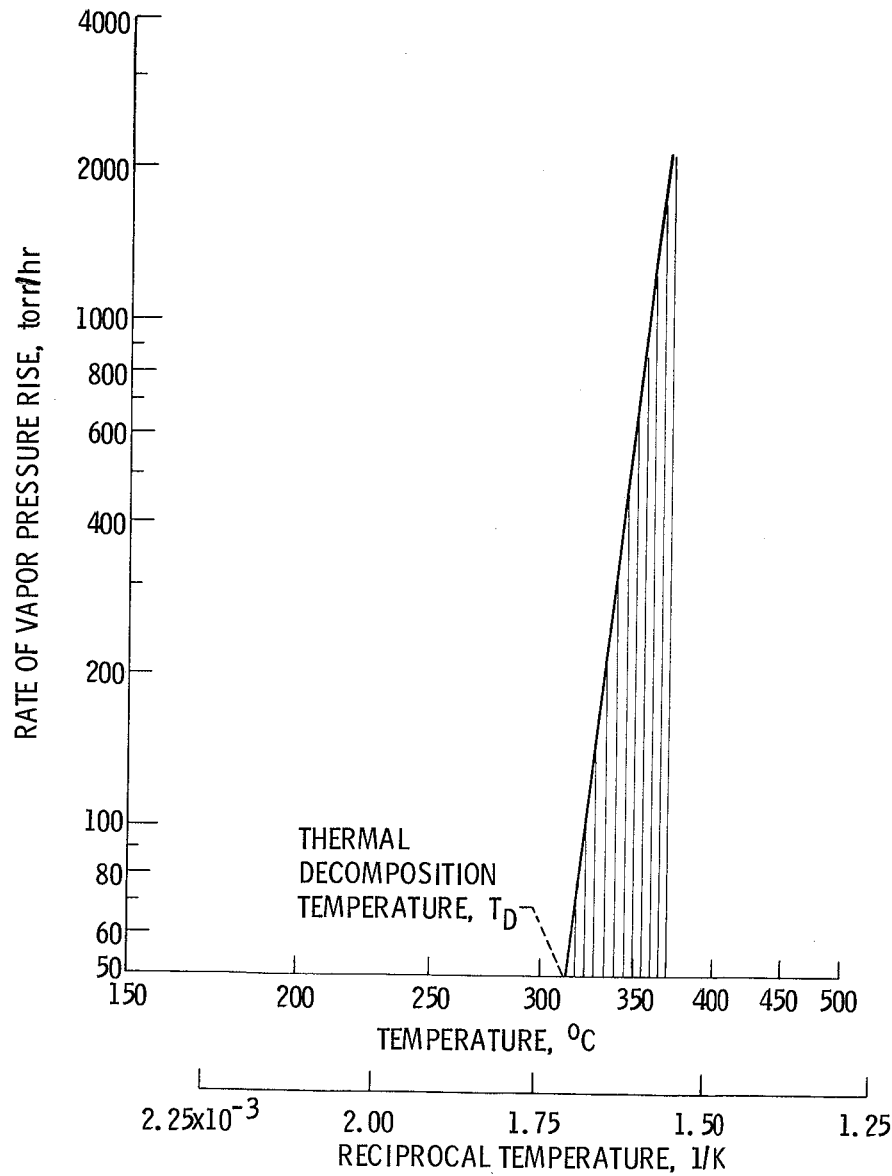


Figure 3. - Typical thermal decomposition curve, rate of vapor pressure rise as function of reciprocal of absolute temperature. Heating interval, 5°C (9°F).

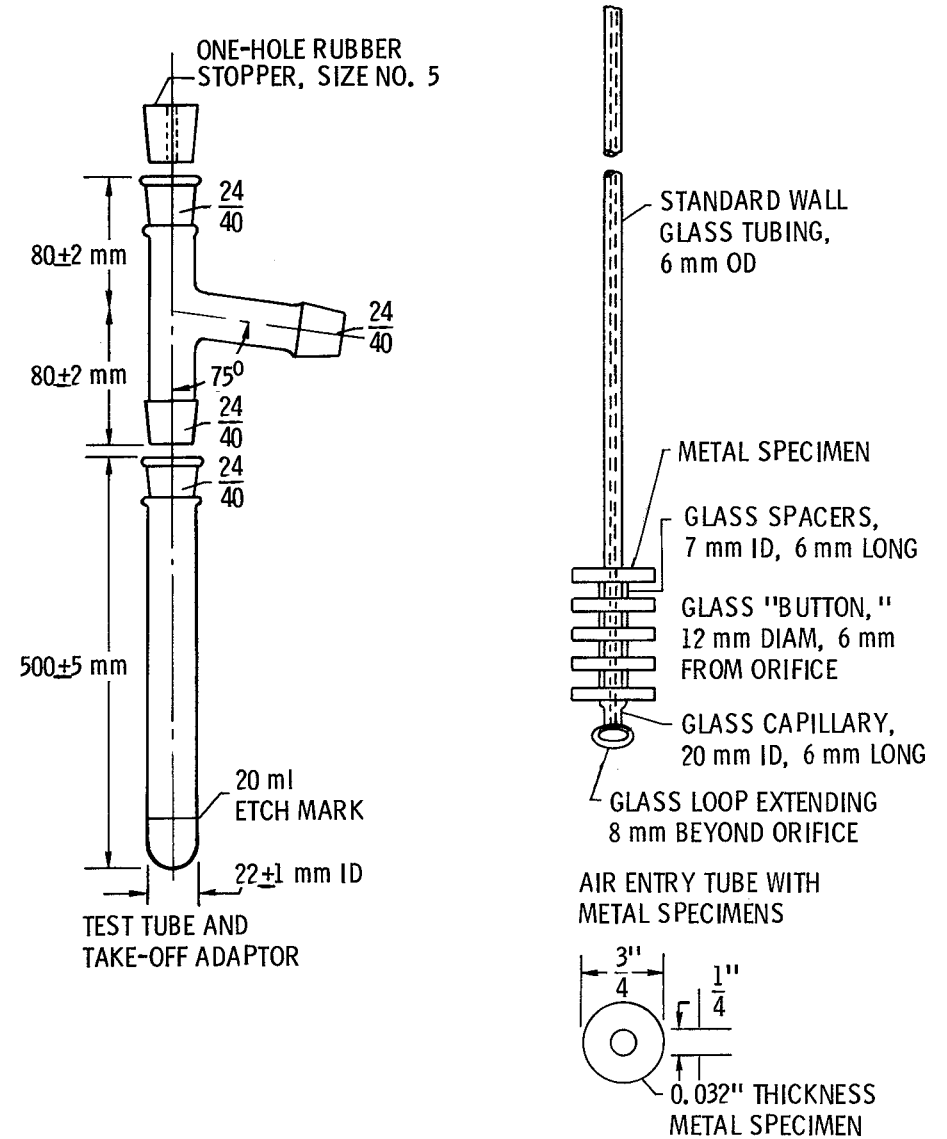
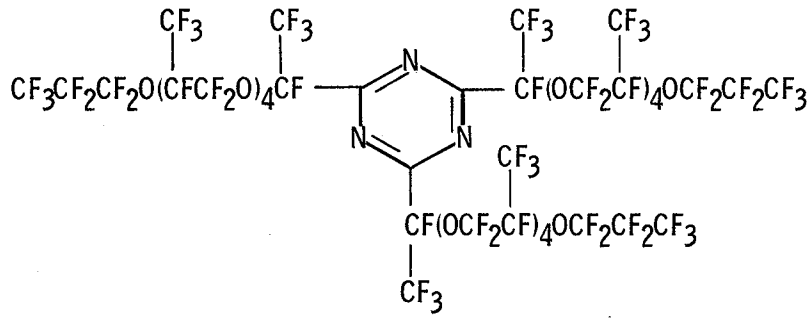
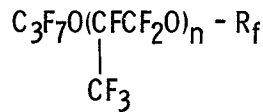


Figure 4. - Micro-oxidation-corrosion apparatus.



(a) PERFLUOROPOLYETHER S-TRIAZINE.



(b) FLUORINATED POLYETHER.

Figure 5. - Chemical structures of the experimental fluids.

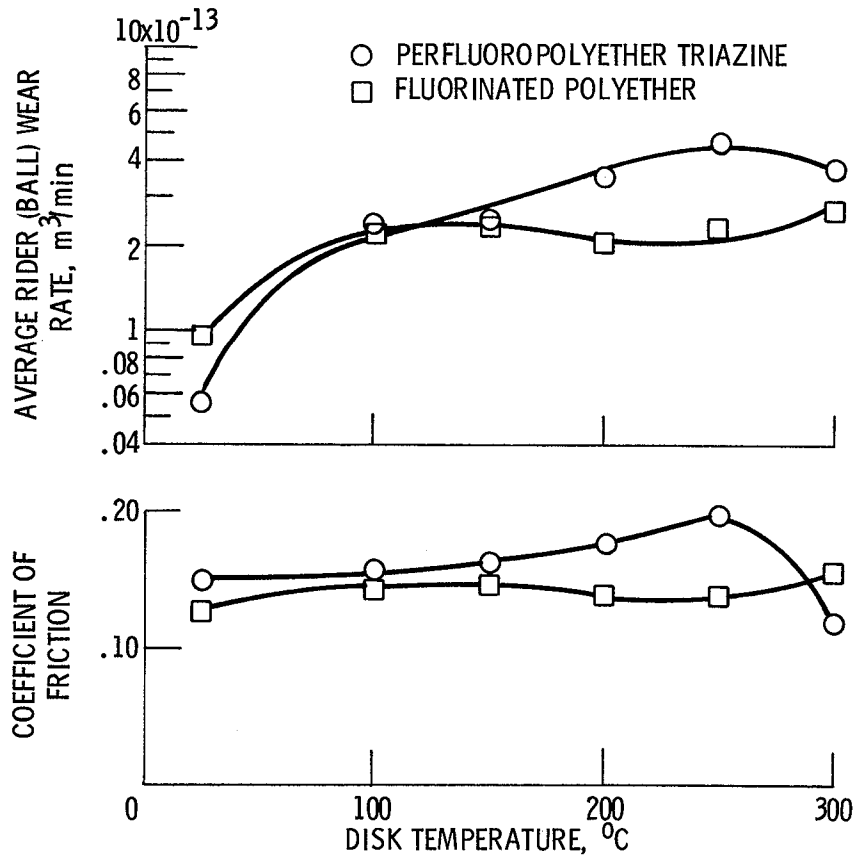
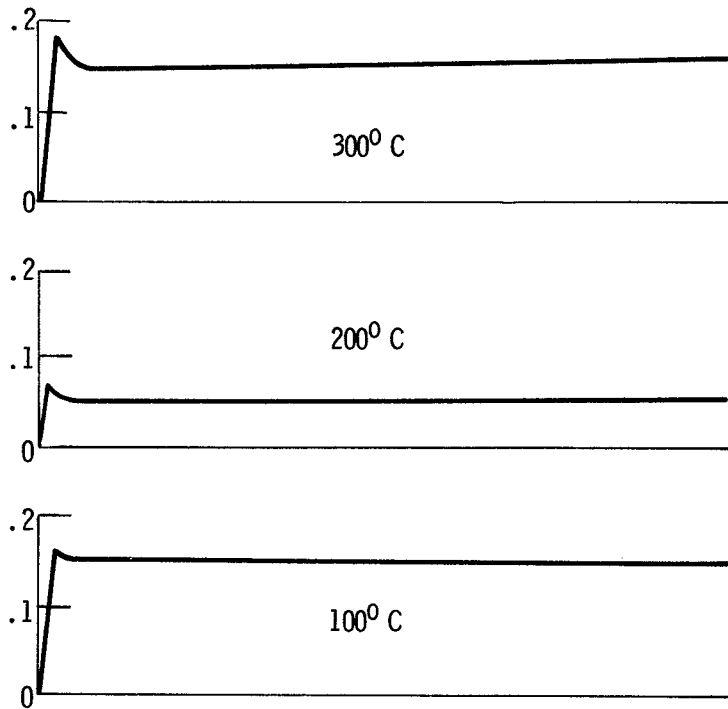
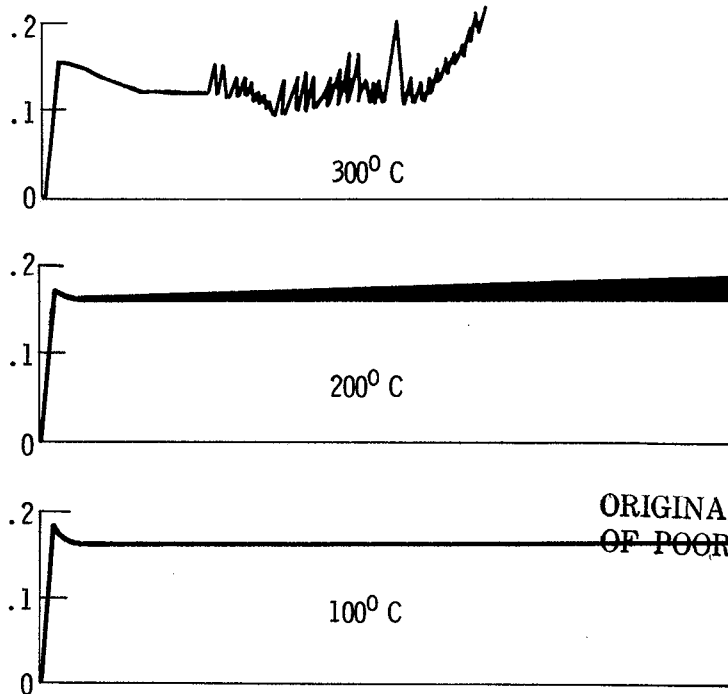


Figure 6. - Average rider wear rate and coefficient of friction for a perfluoropolyether triazine and a fluorinated polyether as a function of disk temperature (dry air atmosphere, 1 kg load, 25 minute test duration).



(a) FLUORINATED POLYETHER IN DRY AIR (TEMP - 100°, 200°, AND 300° C, 1 kg LOAD, 17 m/min SLIDING SPEED, 25 min TEST DURATION).



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(b) PERFLUOROPOLYETHER TRIAZINE IN DRY AIR (TEMP - 100°, 200°, AND 300° C, 1 kg LOAD, 17 m/min SLIDING SPEED, 25 min TEST DURATION).

Figure 7. - Coefficient of friction as a function of time.

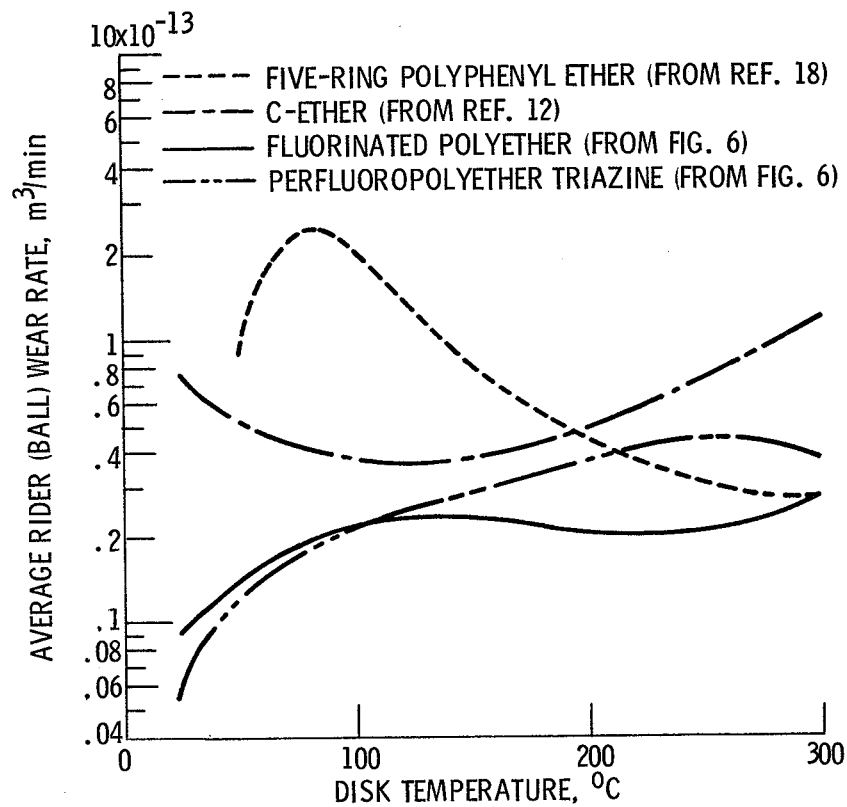


Figure 8. - Average rider wear rate as a function of disk temperature for four fluids.

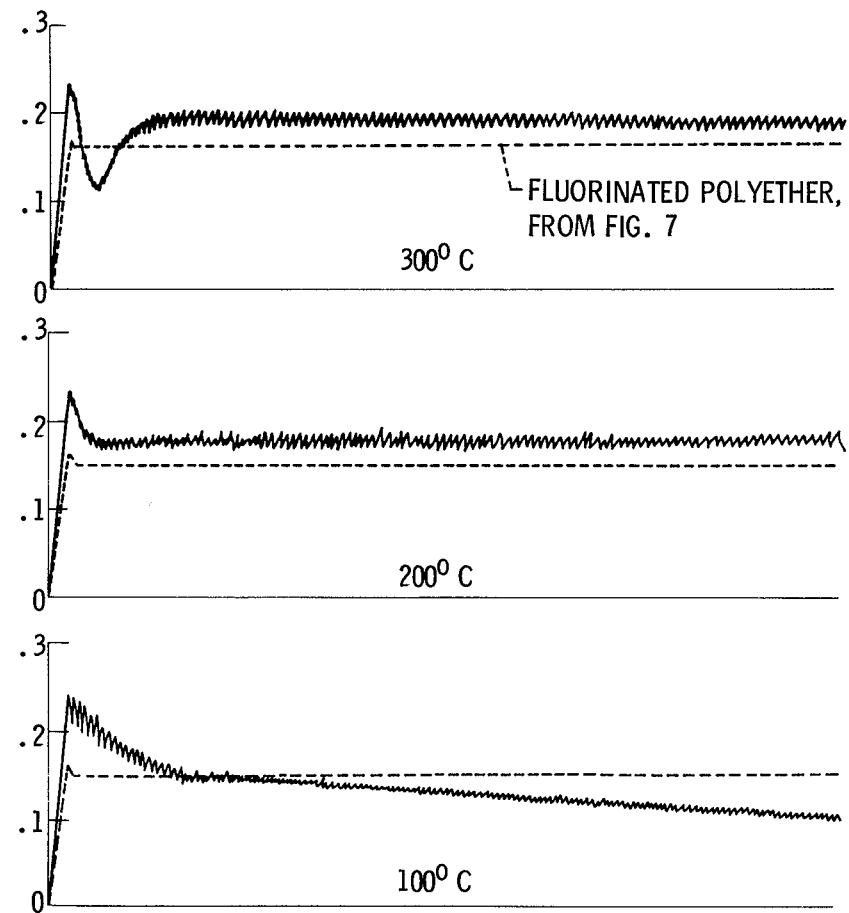
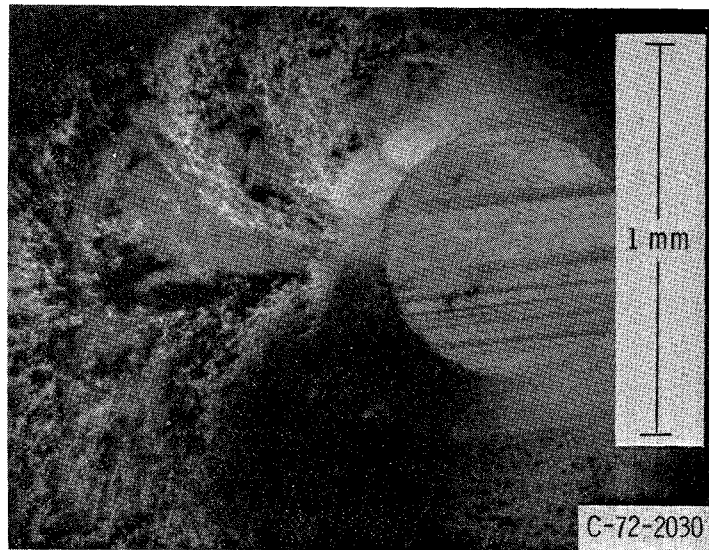
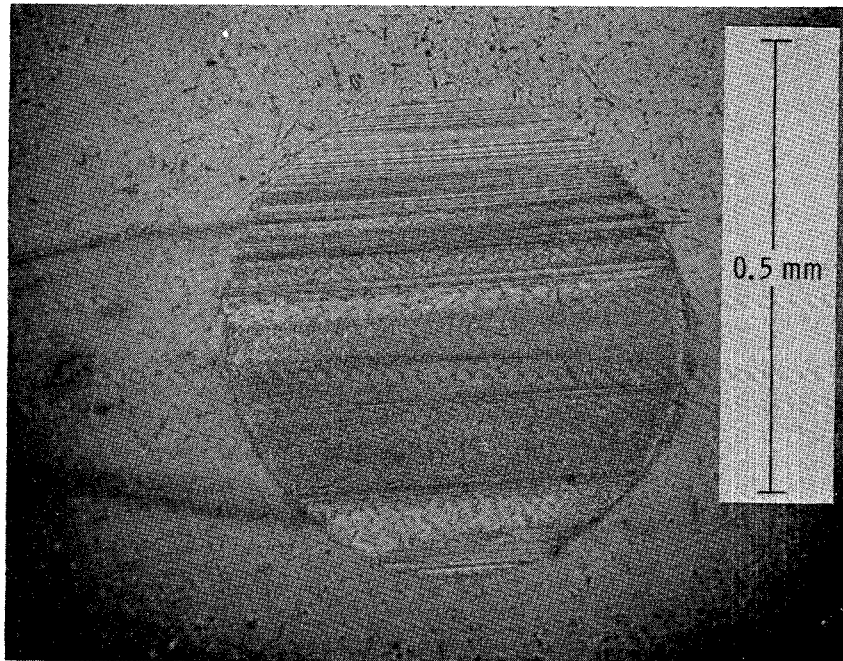


Figure 9. - Friction traces for a C-ether base fluid at 100 $^{\circ}$, 200 $^{\circ}$, and 300 $^{\circ}\text{C}$, 1 kg load, 17 m/min sliding speed, dry air atmosphere, M-50 steel specimens, 25 min test duration.



(a) C-ETHER BASE FLUID, 225⁰ C.



(b) PERFLUOROPOLYETHER TRIAZINE, 200⁰ C.

Figure 10. - Typical rider wear scars for a C-ether base fluid. Test conditions: 1-kilogram load, 17-meter-per-minute sliding speed, dry air (<100 ppm H₂O), M-50 steel test specimens, and 25-minute test duration.

E-9879

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16. Abstract Boundary lubricating characteristics, thermal stability, and oxidation-corrosion stability were determined for a fluorinated polyether and a perfluoropolyether triazine. A ball-on-disk apparatus, a tensimeter, and oxidation-corrosion apparatus were used. Results were compared to data for a polyphenyl ether and a C-ether. The polyether and triazine yielded better boundary lubricating characteristics than either the polyphenyl ether or C-ether. The polyphenyl ether had the greatest thermal stability (443 ^o C) while the other fluids had stabilities in the range 389 ^o to 397 ^o C. Oxidation-corrosion results indicated the following order of stabilities: perfluoropolyether triazine > polyphenyl ether > C-ether > fluorinated polyether.			
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