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ABSTRACT

The friction and wear characteristics of two formulated perfluoropolyether based greases were compared to their non-additive base greases. One grease was developed for the electronics industry (designated as GXL-296A) while the other is for space applications (designated GXL-320A). The formulated greases (GXL-296B and GXL-320B) contained a proprietary antiwear additive at an optimized concentration. Tests were conducted using a vacuum four-ball tribometer. AISI 52100 steel specimens were used for all GXL-296 tests. Both AISI 52100 steel and 440C stainless steel were tested with the GXL 320 greases. Test conditions included: a pressure $< 6.7 \times 10^{-4}$ Pa, a 200N load, a sliding velocity of 28.8 mm/sec (100 rpm) and room temperature ($\cong 23$ °C). Wear rates for each grease were determined from the slope of the wear volume as a function of sliding distance. Both non-additive base greases yielded relatively high wear rates on the order of 10^{-8} mm³/mm using AISI 52100 steel specimens. Formulated grease GXL-296B yielded a reduction in wear rate by a factor of ~ 21 , while grease GXL-320B had a reduction of ~ 12 times. Lower wear rates (~ 50 %) were observed with both GXL-320 greases using 440C stainless steel. Mean friction coefficients were slightly higher for both formulated greases compared to their base greases. The GXL-296 series (higher base oil viscosity) yielded much higher friction coefficients compared to their GXL-320 series (lower base oil viscosity) counterparts.

INTRODUCTION

Perfluoropolyethers (PFPE) are a class of liquid lubricants that have been used extensively for space applications (1) and more recently for magnetic recording media (2). In addition, these fluids are also candidates for advanced gas turbine engine applications (3). Several different structures are commercially available. One branched fluid is made by CsF catalyzed polymerization of hexafluoropropene oxide (HFPO)(K fluids) (4). A similar polymer is made by the UV catalyzed oxidation of HFPO (FY fluids) (5). A linear fluid (FZ fluids) is prepared by a similar process, but utilizing tetrafluoroethylene (6). A fourth material (D fluids) is produced by polymerization of tetrafluorooxetane followed by direct fluorination (7). Some of these materials are available as greases thickened with polytetrafluoroethylene.

There is a multiplicity of papers demonstrating the high wear rates generated with unformulated PFPE fluids in vacuum and under high loads (8)-(18). Others have shown the degradation characteristics of PFPEs in contact with a variety of surfaces in air, oxygen and vacuum (19)-(21). Several mechanisms of catalytic degradation have been invoked, including attacks by Lewis acids (22)-(23), oxides and nascent metals (24)-(26) and surface hydroxyl groups (27)-(28). The linear PFPEs are more prone to degradation than the branched polymers (29), but do exhibit very low volatility and excellent low temperature performance important for space applications (1). For semiconductor applications, low volatility is crucial, but low temperature fluidity is not a requirement.

Early work with antiwear or antidegradation additives was hampered by the insolubility of conventional additives in the PFPE basestocks. In recent years, a number of studies have involved the synthesis of PFPE soluble additives (11),(30)-(35). Some of

these studies showed little or no wear reduction while others showed substantial reductions. However, none were performed on PFPE grease-based systems.

The objective of this study was to formulate two commercially available PFPE greases (one for space applications and the other for use in the semiconductor industry) with an antiwear additive and compare the friction and wear characteristics with the unformulated base greases in vacuum using a four-ball apparatus.

EXPERIMENTAL

Lubricants

Two different base greases were used in these studies. GXL-296A designed for semiconductor applications and GXL-320A for space applications. A compilation of their properties are listed in Table 1. The base fluid for the 320 series was an FZ PFPE. The base fluid for the 296 series was a mixture of an FZ and a K fluid. The antiwear additive used in this study is generically described as a carbonic acid calcium salt, 1:1 at a 10 wt % concentration in the 320 series and 5 wt % in the 296 series.

Tribometer

A four-ball tribometer (Figure 1), operating in the boundary lubrication mode, was used to measure steady state wear rates and friction coefficients for each lubricant. Specimen configuration (Figure 2) is essentially the same as the ordinary four-ball apparatus, except for the use of 9.5 mm (3/8 in.) diameter 440C stainless steel (grade 10) or AISI 52100 chrome steel bearing balls (grade 25). A complete description of this device appears in reference 36.

Testing Procedure

Cleaning and Preparation

The test balls were ultrasonically cleaned in baths of hexane, acetone and ethanol for 10 minutes each, respectively. The lubricant cup was also ultrasonically cleaned in a similar manner. The balls and cup were then blown dry with nitrogen. Further cleaning of the balls occurred with a 15 minute UV/ozone treatment, rotating the balls every 5 minutes. Within 5 minutes of this treatment, one of the balls was placed in the nitrogen purged tribometer and was used as the rotating ball. The other three balls were secured into the lubricant cup and covered with the test grease. The cup was then placed in a bell jar and evacuated for about 30 minutes at 0.01 Torr. The cup was then placed on the stage inside the tribometer and the chamber evacuated.

Testing

After reaching a pressure of less than 6.7×10^{-4} Pa, the experiment was started. The stage was pneumatically loaded against the upper ball and rotation was initiated. All tests were performed at room temperature, a load of 200 N (an initial Hertzian mean stress of 3.5 GPa), and a sliding speed of 28.8 mm/sec (rotating ball speed of 100 rpm). Frictional torque was monitored and recorded throughout the experiment by means of a Hall-effect position sensor. A minimum of four tests were run for each grease.

Wear was determined by measuring the wear scar diameters on the three stationary balls by using an optical microscope. A sample stage on the microscope was designed so that the wear scars could be measured without disassembling the balls from the cup. The experiment was continued using the same set of balls. A completed test run was four hours in length with interruptions every hour for wear measurements. After completion of

the test, a wear rate (mm^3/mm) was calculated from the slope of the line which was obtained from a plot of wear volume as a function of sliding distance. An example of this data from a typical test is shown in figure 3.

RESULTS

Wear

Wear rate results, including standard deviations, appear in Table 2 for all greases tested. The number of tests performed with each grease is contained within the parentheses in the respective lubricant column. The space applications greases (320 series) were tested with both AISI 52100 steel and 440C stainless steel specimens. The electronics grade greases (296 series) were only studied with 440C steel specimens. Both base greases (GXL-296A and GXL-320A) yielded high wear rates in the range of 10^{-8} mm^3/mm using AISI 52100 steel specimens. In contrast, both formulated greases (GXL-296B and GXL-320B) exhibited more than an order of magnitude decrease in wear rate. These results are shown graphically in figure 4, where error bars represent one standard deviation. In addition, wear rates for 320A and 320B with 440C steel were 50 % less than that obtained with 52100 steel. For comparison, the base oil FZ-25 with 440C steel specimens also appears in figure 4.

Friction

Friction coefficients are also shown in Table 2. The mean friction coefficients are indicated, while the ranges are within the parentheses. Values for greases 320A and B with 440C specimens were not measured. The mean friction coefficients are also shown in figure 5, where the error bars represent the minimum and maximum for each grease. All greases had mean friction coefficients between 0.11 to 0.20. Both formulated greases

yielded small increases in mean friction coefficient compared to their base greases. A much larger increase was seen when comparing the 320 greases with their 296 counterparts. The higher base oil viscosity of the 296 series contributes to this difference.

DISCUSSION

PFPE base lubricants normally operate in the corrosive wear regime under boundary lubrication conditions (1). In boundary contacts, PFPEs react with bearing surfaces producing a series of corrosive products. These products can then react with the existing surface oxides on the steel to produce metal fluorides (12). Metal fluorides are effective solid lubricants which provide protection for the surfaces in contact by reducing friction and wear. However, these fluorides can also attack and decompose PFPEs which results in the production of even more reactive species. The surface fluorides are constantly removed during the wear process which is the reason for the high substrate wear (i.e. corrosive wear). Overall, the very reaction that enables PFPEs to protect surfaces eventually leads to their total destruction.

The incorporation of additives into PFPE base fluids and greases reduces this corrosive wear phenomenon to more acceptable levels, thus prolonging the life of the lubricated couple. The mechanism of wear inhibition of the additive in this study is not known, but the additive probably acts as a getter for the corrosive acidic products produced during the tribological process. This would reduce the corrosive wear mechanism.

The base oil used for the GXL-296 series of tests was not tested by itself. However, in previous work (37), the base oil for the GXL-320 series was tested in the same tribometer using 440C specimens, under identical conditions. As shown in Figure 4,

this base oil yielded a much lower wear rate (about one fourth) than its equivalent base grease (GXL-320A). This is probably the result of contact starvation.

SUMMARY OF RESULTS

1. Two unformulated base PFPE greases (GXL-296 (base) and GXL-320A) yielded high wear rates under vacuum at room temperature on the order of 10^{-8} mm³/mm using AISI 52100 steel specimens.
2. A carbonic acid calcium salt additive at an optimized 10 wt % in GXL-320 series and 5 wt % in GXL 296 series greases, reduced wear rates by more than an order of magnitude.
3. Substituting 440 stainless steel specimens reduced wear another 50 per cent for greases (GXL-320A and 320B) compared to AISI 52100 steel.
4. Mean friction coefficients were in the range of 0.11 to 0.20. Small reductions were observed between the formulated and unformulated pairs. A large increase (~ 50 %) was observed between the GXL-320 and GXL-296 counterparts, which is probably related to the higher base oil viscosity for the 296 series of greases.

CONCLUSION

Formulated PFPE greases are now available that retain low temperature fluidity and low volatility, while exhibiting acceptable wear rates, for both space and semiconductor applications.

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Table 1. Typical Properties of Test Materials

| TEST | TEST METHOD | GXL-320A | GXL-320B | GXL-296A | GXL-296B |
|---|--------------------------------|---|---|---|---|
| BASE OIL DATA | | | | | |
| Viscosity, cS @ 100C 40C -40C -54C | ASTM D445 | 45 148 1123 12000 | 45 148 1123 12000 | 44 208 28000 167000 | 44 208 28000 167000 |
| VI | | 350 | 350 | 256 | 256 |
| Pour Pt., C | | <-73 | <-73 | -60 | -60 |
| Vapor Press, Torr @ 20C 100C | ASTM D92 Knudsen | <10 ⁻¹³ <10 ⁻⁹ | <10 ⁻¹³ <10 ⁻⁹ | <10 ⁻¹³ <10 ⁻⁹ | <10 ⁻¹³ <10 ⁻⁹ |
| GREASE DATA | | | | | |
| Penetration,mm /10 worked | ASTM D1403 | 290 | 285 | 280 | 272 |
| Dropping Pt., F | | | | | |
| Four Ball Wear, mm (1200 rpm, 75C, 1 hr, 40 kg, N ₂ , 52100) | ASTM D2265 ASTM D2266 | 460 0.99 | 488 0.5 | 456 1.06 | 492 0.49 |
| (600 rpm, 74C, 1 hr, 60 kg, N ₂ , 52100) | | 1.57 | 1.08 | 1.56 | 1.27 |
| (368 rpm, 300C, 1 hr, 5.5 kg, N ₂ , 52100) | | 0.52 | 0.35 | 0.49 | 0.27 |
| Evap. Loss, 22 hr @ 20C, % | ASTM D2595 | 1.4 | 2.0 | 0.19 | 0.14 |
| Oil Separation, 22 hr @ 204C | | | | | |
| Oil Sep., % | FTM 321 | 12.4 | 10.3 | 6.3 | 6.3 |
| Evap. Loss, % | | 0.11 | 0.16 | 0.14 | 0.14 |
| Wt. Loss, % | | 12.5 | 10.5 | 6.4 | 6.4 |

| | | | | | |
|--|---|---------------------------------|---------------------------------|---------------------------------|---------------------------------|
| Low Temp, Torque, g-cm @ -73C, starting Running, 1 hr | ASTM D1478 | 1430 637 | 1040 650 | 53000 froze | 38000 froze |
| Outgassing Vapor Press, torr @ 20C, TML, 48 hr @ 150C, % | ASTM E1559 Lockheed | <10 ⁻¹⁴ 0.040 | <10 ⁻¹⁴ 0.039 | <10 ⁻¹⁴ 0.063 | <10 ⁻¹⁴ 0.073 |
| Vacuum Stability TML, % Collected Condensable, % | ASTM E595 | 0.15 0.02 | 0.0 0.0 | 0.14 0.0 | 0.08 0.03 |
| Bearing Test, Hrs first darkening | Kaydon Bearing | <60 | 140 | 160 | >290 |
| Copper Strip, 24 hr @ 100C Four Ball Weld Point, kg Liquid Oxygen Impact Test Rust Preventative | ASTM D130 ASTM D2595 ASTM D2512 ASTM D1743 | 1B >800 Pass Pass | 1B 620 Pass Pass | 1B 800 Pass Pass | 1B 620 Pass Pass |

Table 2. Wear rates and friction coefficients of the test lubricants

| <i>Lubricant (# of runs)</i> | <i>Ball Material</i> | <i>Mean Wear Rate x10⁻⁹ (mm³/mm)</i> | <i>Wear Rate Std Deviation x10⁻⁹ (mm³/mm)</i> | <i>Mean (Range) of Friction Coefficient</i> |
|----------------------------------|----------------------------|--|---|---|
| GXL-320A (Base) (4) | AISI 52100 Steel | 15 | ±1.1 | 0.11 (0.09-0.23) |
| GXL-320B (Formulated) (4) | AISI 52100 Steel | 1.2 | ±0.3 | 0.14 (0.08-0.19) |
| GXL-320A (Base) (4) | 440C Stainless Steel | 6.2 | ±1.3 | NM |
| GXL-320B (Formulated) (4) | 440C Stainless Steel | 0.74 | ±0.44 | NM |
| PFPE Z-25 (Liquid) (4)* | 440C Stainless Steel | 1.7 | ±0.16 | 0.08 (0.05-0.10) |
| GXL-296A (Base) (3) | AISI 52100 Steel | 11 | ±0.43 | 0.17 (0.14-0.32) |
| GXL-296B (Formulated) (3) | AISI 52100 Steel | 0.53 | ±0.17 | 0.20 (0.13-0.25) |

* From Reference 37

NM (not measured)

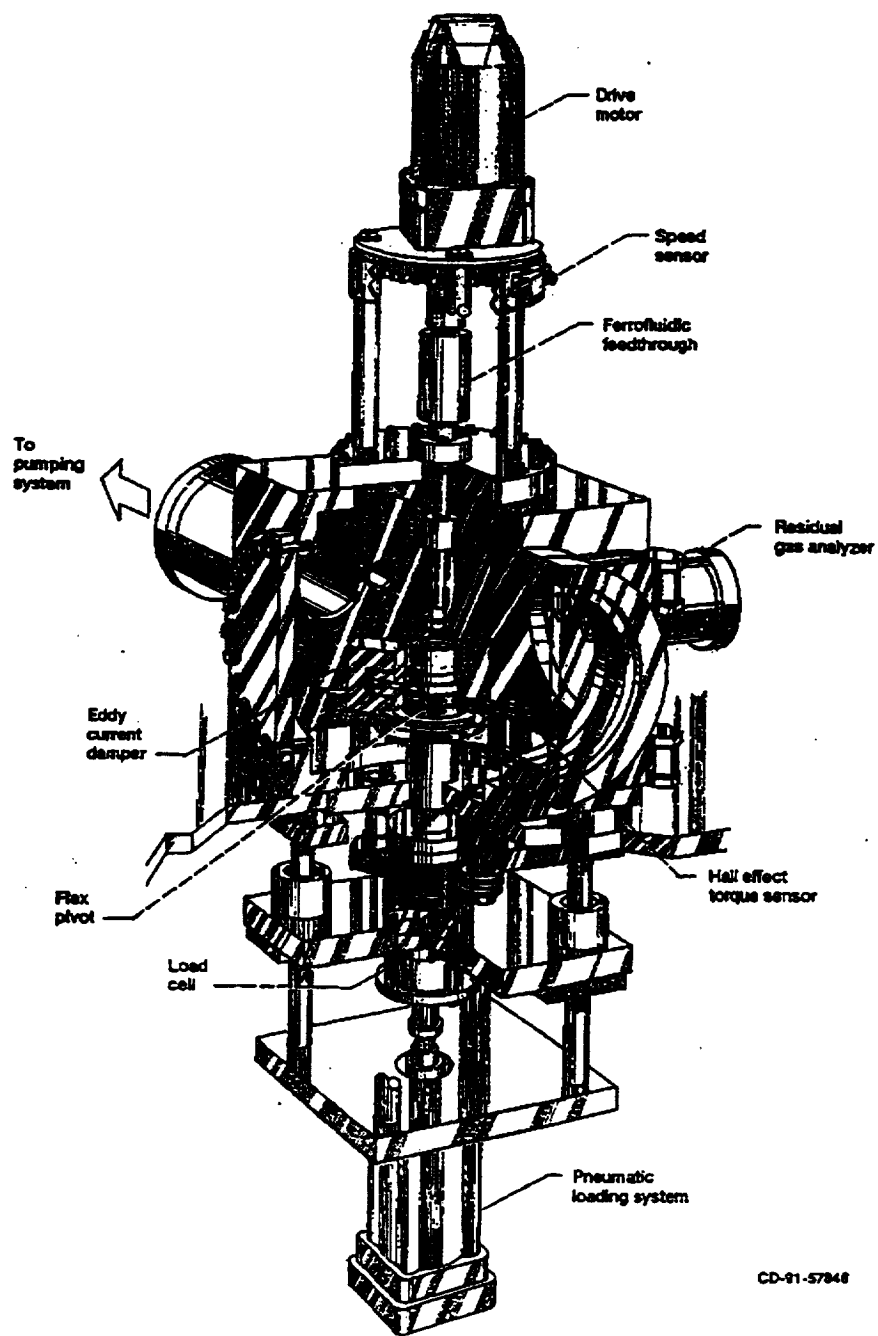


Figure 1.—Vacuum tribometer.

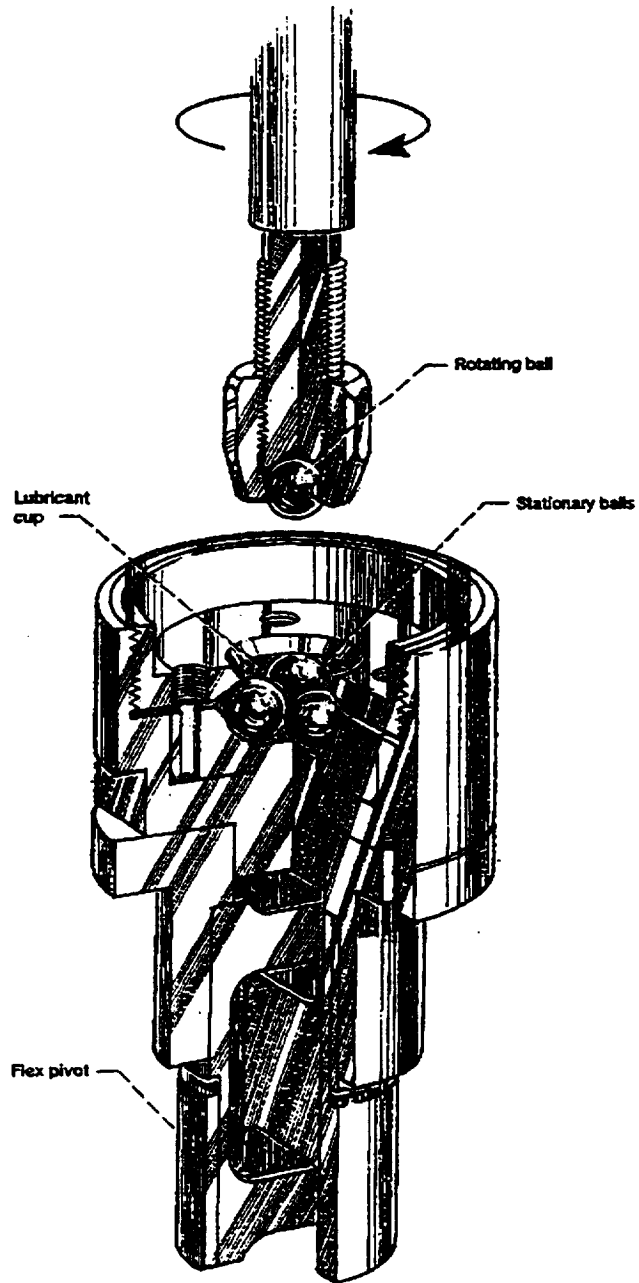
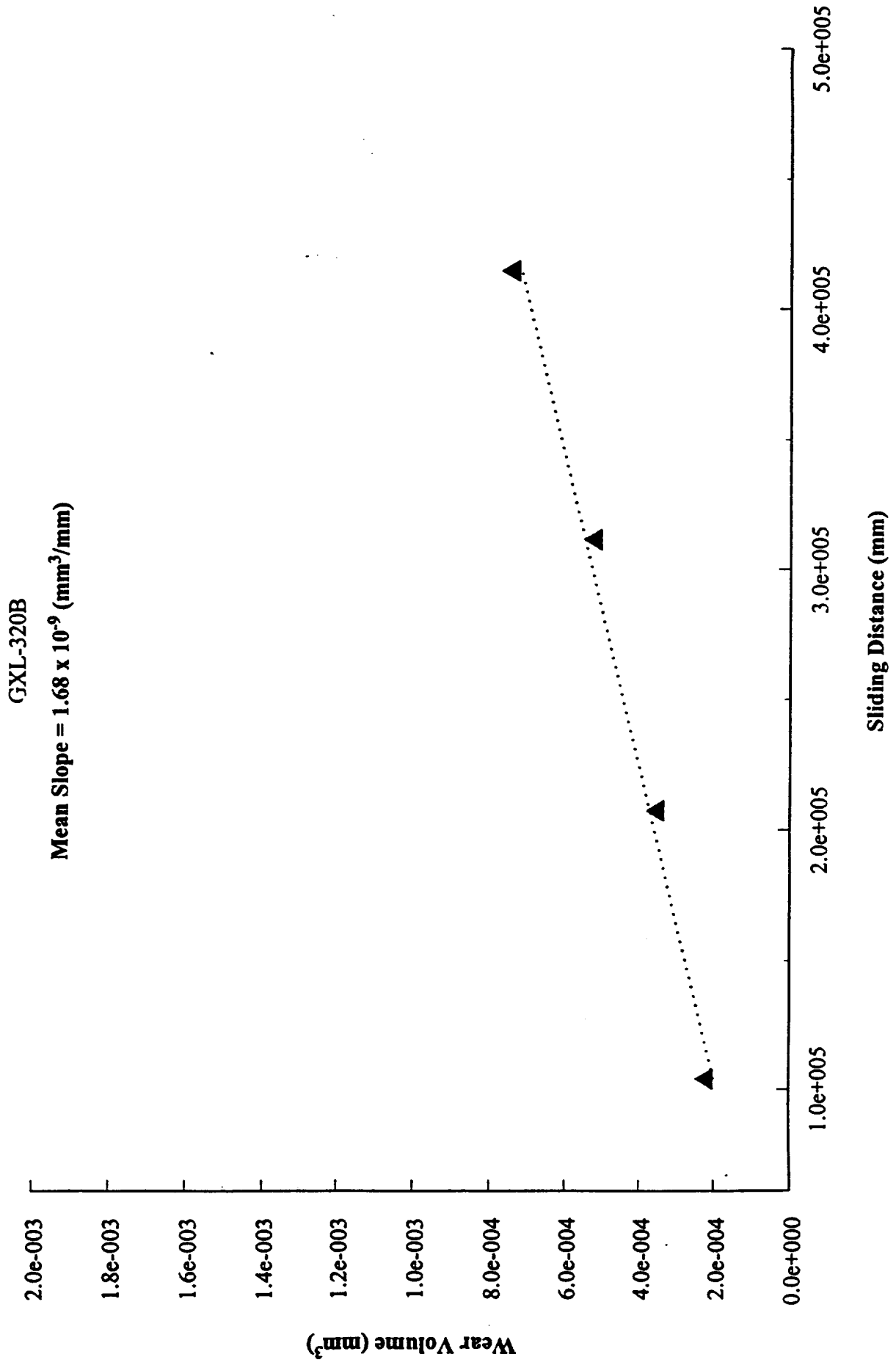


Figure 2.—Specimen Configuration.

Figure 3. Typical Test



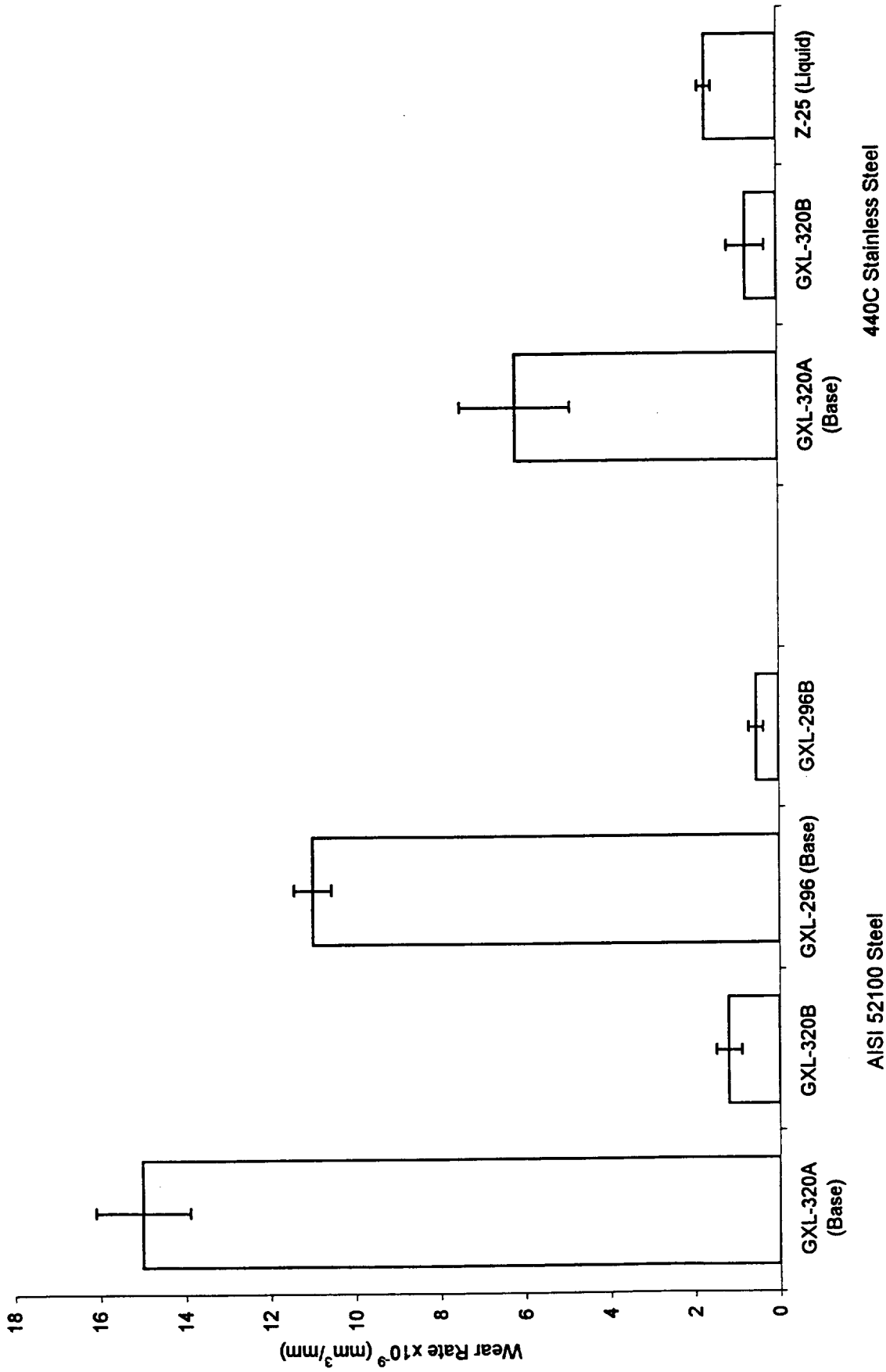


Figure 4. Wear rate results from vacuum 4-ball tribometer

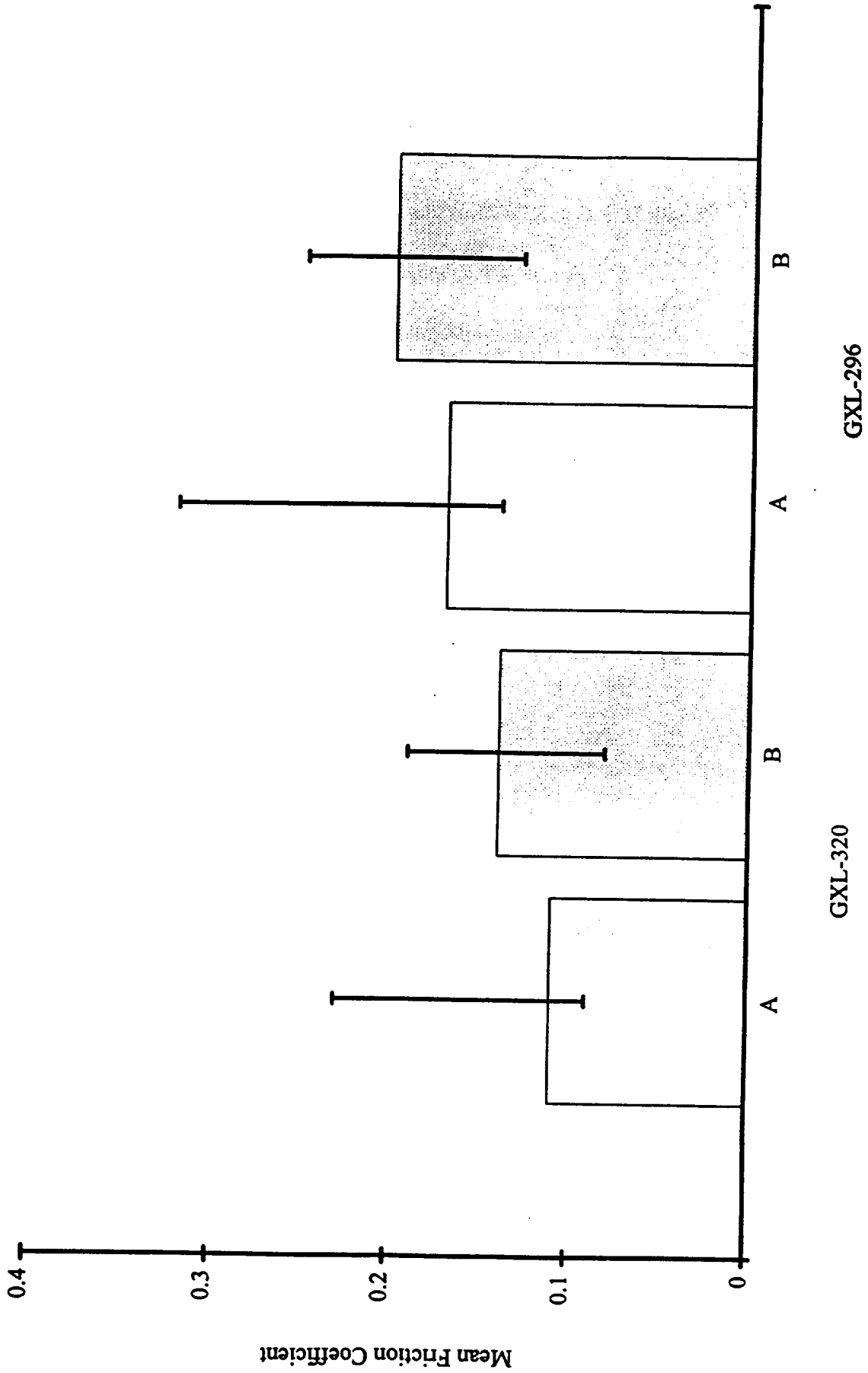


Figure 5. Mean Friction Coefficients of the Formulated and Unformulated Greases with AISI 52100 steel specimens.

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