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Tribological Performance of Some Pennzane® Based Greases for Vacuum Applications

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Abstract

Commercial greases for space applications usually fulfill the requirements imposed by the severe conditions of use. The main requirement is their ability to create an EHL film, boundary film, or both under speed, load and temperature conditions that the mechanisms will operate. Three greases, all based on a multiply alkylated cyclopentane (Pennzane®) base oil, were studied. The thickeners were an n-octadecylterephthalamate soap, a lithium soap, and a urea derivative. A Four-Ball Tribometer and a Spiral Orbit Tribometer were employed to evaluate the greases under ultrahigh vacuum. Results indicated that all three greases yielded very low wear rates and extended lifetimes. In addition, routine physical property data is reported for each grease.

I- Introduction

Extended mission lifetimes and improvements to other spacecraft components, such as electronics, batteries, and computers have placed increased burdens on space lubrication systems [1]. Fluid lubrication, either as a liquid or a grease, is commonly used to extend lifetimes and minimize wear, torque, and noise [2]. Because of these demands, the reliability of spacecraft moving mechanical assemblies (MMAs) clearly depends on the lubricant employed to cope with the increased lifetime. In order to assure that mission lifetimes will be completed, accelerated testing has become mandatory and critical.

Although full scale life testing [3] or actual component testing [4, 5, 6] is desirable, they are costly and time consuming. Various accelerated tests are available to evaluate the torque, wear rate, friction coefficient or degradation rate of the lubricant. These include the eccentric bearing test apparatus [7], the vacuum four-ball tribometer [8], and the spiral orbit rolling contact tribometer (SOT) [9].

The eccentric bearing apparatus employs an actual bearing in which an intentional misalignment is introduced to accelerate the degradation rate of the lubricant. Results with this device have correlated well with actual space experience [10].

The vacuum four ball tribometer consists of a rotating ball sliding against three stationary balls that are immersed in a lubricant, either liquid or grease. The wear rate of the balls is calculated from the wear scars generated on the stationary balls. There is an inverse correlation between wear rate and component lifetimes [11].

The spiral orbit tribometer (SOT), fully described in a former study [9], reproduces the kinematics of an angular contact bearing. Data, such as the lubricated lifetime, friction coefficient, contact resistance, and degradation products can be determined, monitored, and analyzed. The relative lifetimes of lubricants measured with the SOT have correlated well with actual bearing life tests [4]. Previously limited to solid lubricants and liquid lubricants, the SOT's ability was recently extended to greases [12], which represent many of the lubricants used in MMAs.

The objective of this work was to compare the wear rates of steel and the lubricated lifetimes of three different vacuum greases based on Pennzane® using the vacuum four-ball apparatus and the spiral orbit tribometer. Each contained a different thickener: an n-octadecylterephthalamate soap, a lithium soap, and a urea derivative. In addition, the terephthalamate based grease contained an antiwear additive and two antioxidants.

II- Materials and Experimental Conditions

1- The Four-Ball Tribometer

This tribometer is used to test the lubricants' ability to reduce wear of bearing elements under high loads and operates with standard bearing balls. It is illustrated in Figure 1. The specimens are made of AISI 440C stainless steel. Balls were grade 25, 9.5 mm (3/8 inch) diameter. A 200 N load was applied through a pneumatic system (corresponding to an initial Hertz stress of 2.7 GPa). The top plate rotated and the sliding generated a wear scar on each stationary ball. The tests were conducted at a speed of 100 rpm. The test automatically began when the pressure dropped below $1.3.10^{-4}$ Pa. The tests were performed at room temperature. The wear scars were measured after each hour with a microscope and the wear volume was calculated. This value is calculated considering the material worn has the shape of a spherical zone. Wear volume was plotted as a function of sliding distance and a wear rate calculated from a linear regression. At least four tests were run for each grease.

2- The Spiral Orbit Tribometer (SOT)

A Spiral Orbit Tribometer (SOT) simulates an angular contact bearing (Figure 2). A 12.7 mm (1/2 inch) ball rolled between a fixed plate and a rotary plate, running at 210 rpm. The load, providing a mean Hertz stress of 1.5 GPa, was applied through the fixed plate. The combination of the high load, the moderate speed, and of the small amount of lubricant (approximately 50 μg) allowed the system to operate in the boundary lubrication regime. The ball was rolling and pivoting in a spiral and maintained in the orbit by the guide plate. The force the ball exerted on the guide plate was used to determine the friction coefficient, since the ball was sliding between the disks at this moment. The resistance of the contacts between the ball and the plates was calculated from the voltage drop across the plates. The evaluation of the greases was conducted at room temperature (≈ 23 °C), and under ultrahigh vacuum ($1.3.10^{-6}$ Pa). As the lubricant was tribologically stressed, it was degraded and eventually consumed. Test conclusion was defined when a friction coefficient of 0.28 was attained. Normalized lubricant lifetime (or inversely, its degradation rate) was then defined as the number of orbits divided by the amount of lubricant in micrograms.

3- Materials preparation

The greases are all based on a multiply alkylated cyclopentane (Pennzane®) oil [13]. A summary of the composition and physical properties is provided in Table 1. Rheolube® 2000 is a commonly used grease for space applications, containing both anti-wear and anti-oxidant additives [14]. The grease MULTEMP 1C408 is manufactured with a lithium soap of 12-hydroxystearate (8% by weight), while the MULTEMP 1C409 contains a thickener made with a urea derivative (13% by weight). Neither MULTEMP grease contains any additives.

For the SOT tests, the greases were applied only on the ball by rolling it several times between two elastic membranes made of polyethylene. The small amount of grease deposited on the ball (30-60 μg) was determined using a balance with an accuracy of ± 2 μg. The edges of the wear tracks on the SOT plates, where some of the deposited lubricant was pushed aside during the test, were analyzed with an infrared micro-spectrometer. It confirmed that both oil and thickener were present on the ball surface.

All specimens were made of AISI 440C stainless steel. For tribological purposes, ball and plate surfaces were polished to a roughness Ra of 0.05 μm. The parts were first rubbed with an alumina slurry and rinsed under running deionized water. Then they were ultrasonically cleaned for ten minutes each first in a bath of hexane, followed by deionized water. All drying was done with filtered nitrogen. The procedure was completed by exposing the specimens to UV/ozone for 15 minutes. More details can be found in reference [15]. In the case of the Four-Ball Tribometer, the cleaning baths were hexane, acetone and methanol, respectively (10 minutes each).

III- Results

1- Surface analysis

X-Ray Phoelectron Spectroscopy

An XPS analysis of some ball surfaces was conducted for the greases based on the lithium soap and urea derivative. The objective was to look for lithium and nitrogen traces on the surfaces after the tests. In both cases, no traces of lithium nor of nitrogen were detected. The ball surfaces had a slight brown coloration. The XPS spectra revealed the presence of a thick carbon layer, created by degradation of the greases. The thickness of this layer was great enough (> 40 Å) to obscure the iron substrate peaks. Moreover, in the case of lithium, the Li 1s peak is usually weak and interferes with one of the iron peaks. A similar interference problem occurred with an EDAX analysis of the same balls. This implies that the either Li-soap and urea derivative were completely consumed, or that the amounts left, either in its virgin state or after degradation, were too small to be detected.

Infrared Spectroscopy

An infrared analysis was made inside and adjacent to the wear tracks on the disks from the SOT (Figure 3). It confirmed the presence of organic degradation products (broad and weak bands) in the track and of the original grease on the edges of the tracks (sharp and strong bands) (Figure 4).

Raman Spectroscopy

Raman spectroscopy was conducted mainly in the scrub area on the bottom plate. The scrub (Figure 2) is the straight line portion of the orbit where the ball is sliding against the plates when it strikes the guide plate. Lubricant degradation mainly takes place in this area. Examples of this area are shown in Figure 3. The appearance of the sliding area is different between the greases. The degradation is more pronounced for the Rheolube® 2000 than for the other two greases. This was confirmed by the difficulty to detect a signature of degraded lubricant from MULTEMP 1C409 and any signal in the case of MULTEMP 1C408. A comparison of the Raman spectra for all the greases is presented in Figure 5. The spectra have a high fluorescence background. The ones for Rheolube® and MULTEMP 1C409 were obtained under the same conditions. No significant spectral characteristics of degradation products were detected with MULTEMP 1C408 after several attempts in different locations of the scrub. Hence, no comparable spectra were obtained on this grease.

Two broad peaks are evident with Rheolube® 2000, one centered near 1580 cm^{-1} and a smaller peak around 1360 cm^{-1}. These are related to the so called "G" and "D" peaks assigned to amorphous carbon [16], the final stage of the lubricant degradation. This has been observed in other friction polymers [17] and in an in-situ Raman study [18].

2- Wear rates

The wear rates of the steel for each grease (based on four tests) appear in Figure 6 along with reference data for Pennzane® 2001A base oil and formulated Pennzane® 2001. As can be seen, all greases yield low wear rates compared to fluorinated lubricants [8, 11]. These rates are only slightly higher than the formulated oil and base oil.

3- Friction coefficient and Lifetime in the SOT

At least four tests were conducted for each grease. All of them have large normalized lifetimes (number of orbits per microgram of lubricant) compared to fluorinated lubricants [9]. Results are shown in Figure 7. However, the lifetime of Rheolube® 2000 is several times greater than the MULTEMP 1C408 and 1C409 ones. The initial friction coefficients (Figure 8) are low in most cases (0.09-0.10), except for the MULTEMP 1C408 (0.12), but this value was stable during most of the test. For comparison, the initial friction coefficient for Pennzane® base oil is about 0.08.

The friction traces of the greases could be divided into two different behaviors. The first one is the grease with a urea-derivative thickener. The friction coefficient of this grease increased progressively and slowly until failure. The second one included the Li-soap and the Rheolube® 2000 greases. Both of these have also shown a progressive failure, but the increase in friction began at approximately half the lifetime. Thus, these two greases have shown a precursor of the failure, indicated by the arrows in Figure 8.

IV- Discussion

In general, wear rates from the vacuum four-ball tests have correlated well with the spiral orbit tribometer and full-scale bearing tests. That is, high wear rates of the steel are associated with short relative lifetimes of the lubricant (high degradation rates) and short bearing lives. However, in this study, all greases yielded similar and low wear rates ($\approx 2.0.10^{-10}$ mm^3/mm) compared to fluorinated lubricants [8, 11]. Fluorinated lubricants have generally yielded steel wear rates, at least one order of magnitude higher.

On the other hand, the relative lifetimes from the SOT show a clear distinction. The thickener made with sodium n-octadecylterephthalamate soap has yielded the greatest lifetime (more than 15000 orbits/μg). The greases evaluated use the same base oil. Therefore, the ability of these greases to lubricate a contact mainly depends on the ability of the grease to release the oil it contains. The oil is usually enclosed in a three dimensional network made by the thickener, the reaction to create the thickener sometimes taking directly place within the oil. So the oil is released when the grease is "squeezed" by the passage of the ball. This aspect was accurately detailed in reference [19]. Nevertheless, in spite of the evidence of the presence of both the oil and the thickener on the grease applied to the ball, it cannot be determined that the weight percentages are the ones of the original lubricant. It is also clear, from the Raman analysis, that the degradation of the MULTEMP greases has left very little chemical products. The only parameters that change are the presence of additives, the thickener nature or its weight percentage, and, as a consequence, the nature of the interactions between it and the oil.

The additives in Rheolube® 2000 generally have higher vapor pressures compared to the base oil. Therefore, even though "trapped" they are within the structure of the grease, they are not designed to stand ultrahigh vacuum.

The SOT operated with only 30 to 60 μg of grease. Assuming the lubricant is evenly distributed over the surface of the ball, the grease thickness is around 15 nm, while the surface roughness is Ra=0.05 μm. Considering the Stribeck criteria (lambda ratio, lubricant thickness/roughness), the tests were operated in the boundary regime. A resistance equal to zero confirmed this when the tests started, indicating direct contact between the plates and the ball surface. Neither inlet nor outlet were created and the Hamrock and Dowson model cannot be applied to calculate the film thickness. The only supply of lubricant was the initially charge present on the ball surface. Thus, it is clear that the grease composition and its structure will determine if the lubricant will be able to last. According to these results, the method that the small amount of oil present within the grease structure is released changed between the three lubricants evaluated. Also, the thickeners of the MULTEMP greases are not as capable as the one of the Rheolube® 2000 to cope with the severity of the conditions. This is consistent with the worked penetration data of the greases (Table 1). This test gives the depth of penetration of a cone falling in grease under defined conditions [19]. The MULTEMP 1C408 has the lowest one (Table 1), but also with the images and the spectroscopic analysis (Figures 3, 4 and 5) the grease leaving few residues. Grease 1C408 (Li-soap) and 1C409 (urea derivative) have not left residues on the wear tracks. The degradation of the MULTEMP thickeners occured and lead to more volatile products. This would explain the shorter lifetimes and the lower amount of residue on the wear track.

Moreover, a change in the friction coefficient trace appeared in the case of grease MULTEMP 1C408 and Rheolube® 2000 at about half of the lifetime (see Figure 8). Therefore, we have a precursor of the failure, as observed before [12]. That implies that the degradation process of the lubricant (oil and thickener) is taking place later with the greases based on the ester soaps. However, the grease with a urea-derivative thickener has shown a constant increase in the friction coefficient, as did the neat oil. The consumption of the lubricant started immediately with the grease based on the urea derivative. The behavior of the urea grease can also be linked to the ability of the thickener to release the lubricant within the contact. Thus, since the behavior of the MULTEMP 1C409 (urea derivative) was very close to that of the neat oil, we can assume that this grease quickly released the main part of the oil it contained, although the thickener percentage is higher, while the Rheolube® 2000 and the MULTEMP 1C408 (Li soap) greases released it more progressively. This could show that the interaction between the soap and the oil is stronger with the esters soaps.

It is also interesting to note the way the lubricant detected on the edge of the wear track behaves. The grease is supposed to provide oil from the edge to the tracks. But no oil was detected on the track after the conclusion of the test. It appears that the lubricant pushed out of the track is unavailable for the duration of the test. This aspect would reinforce the idea that a lubricant reservoir would still be necessary.

V- Conclusion

The greases based on Pennzane® oil have demonstrated a good ability to operate under severe conditions. The accelerated tests have shown that the grease based on the sodium n-octadecylterephthalamate soap has a greater lifetime than the ones with a lithium or a urea derivative thickeners. All these greases are able to provide good surface protection against wear and provide low friction coefficients. Without structural and rheological data, the effect of different interactions between the thickener and the oil to explain their tribological behaviors could not yet be confirmed. An increase in the thickener mass percentage of the Li-grease could improve its lifetime.

The Spiral Orbit Tribometer, an angular contact bearing simulator, has now clearly confirmed its ability to evaluate and optimize greases. It would also be able to establish a relationship between the amount of lubricant used and the lifetime of a mechanism.

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	GREASES		
	Rheolube® 2000	**MULTEMP 1C408**	**MULTEMP 1C409**
Appearance	light brown	light brown	light brown
Additive(s)	a phosphate, an amine and a hindered phenol	none	none
Thickener	soap of sodium n-octadecylterephthalamate	soap of lithium 12-hydroxy stearate	urea derivative
Mass percentage of thickener	≈ 15	≈ 8	≈ 13
Dropping point (°C)	> 260	209	260
Worked penetration, 60 strikes	276	227	300
Oil separation (100 °C, 24 h) (mass %)	3.3	1.1	1.7

Table 1: Greases compositions and properties

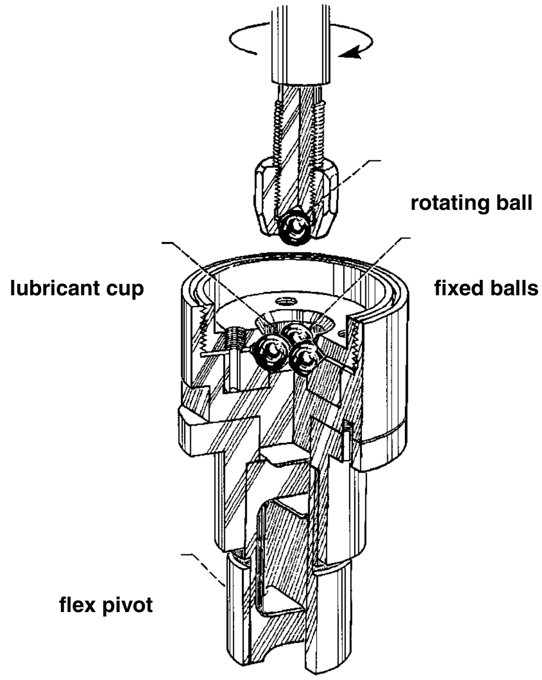


Figure 1: The Four-Ball Tribometer

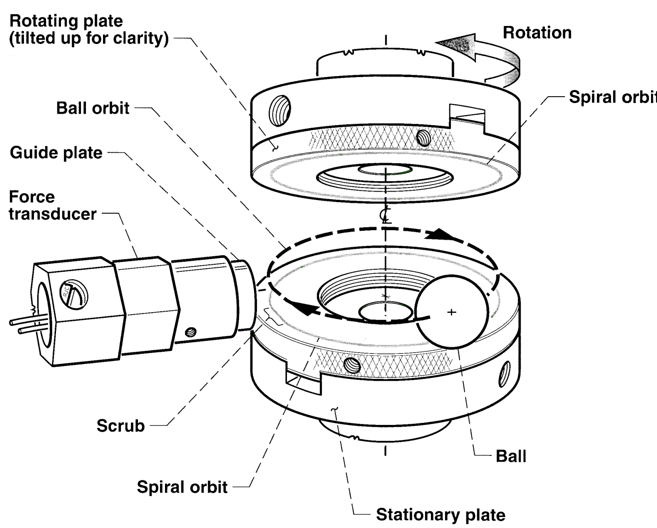
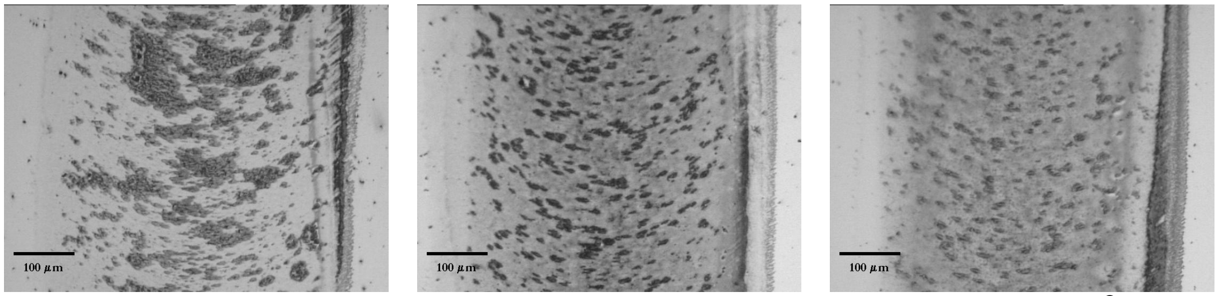
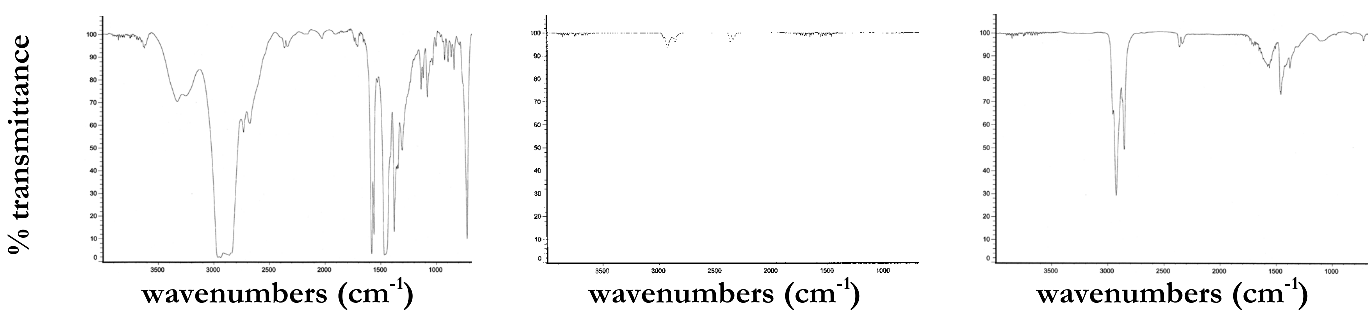


Figure 2: The Spiral Orbit Tribometer



(a) grease 1C408 (b) grease 1C409 (c) grease Rheolube® 2000

Figure 3: Pictures of the scrub area for the three Pennzane® based greases



(a) virgin grease (b) within the track (c) edge of the track

Figure 4: Examples of infrared signatures of the grease 1C408

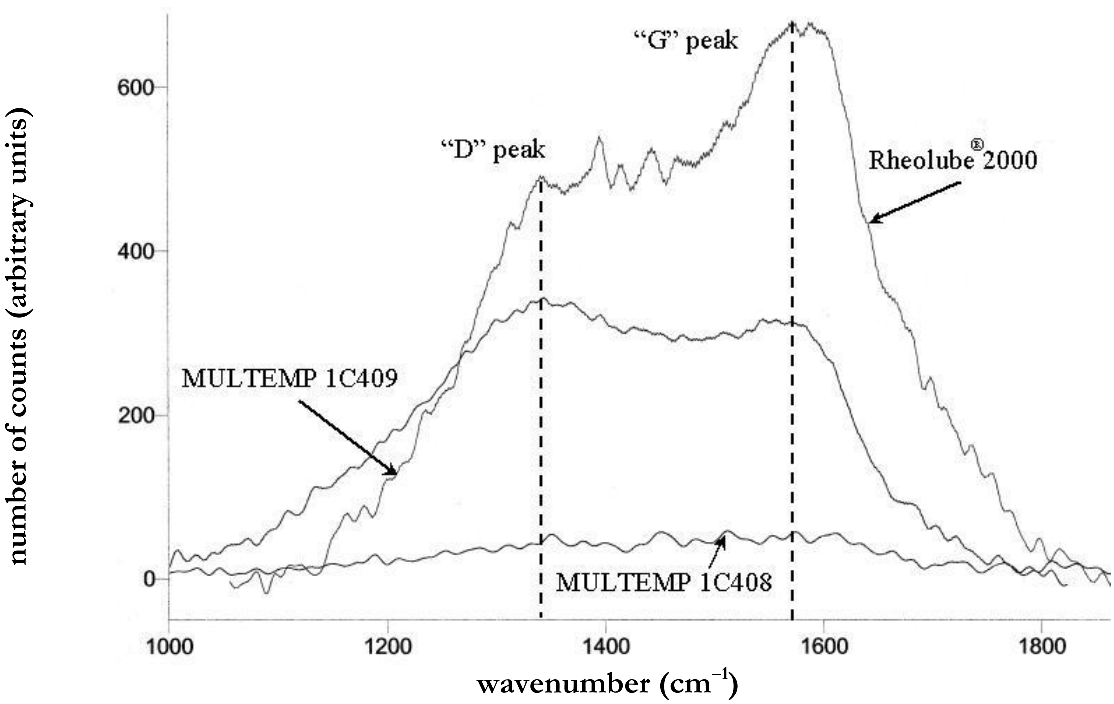


Figure 5: Raman spectra of the scrub area for the different Pennzane® based greases

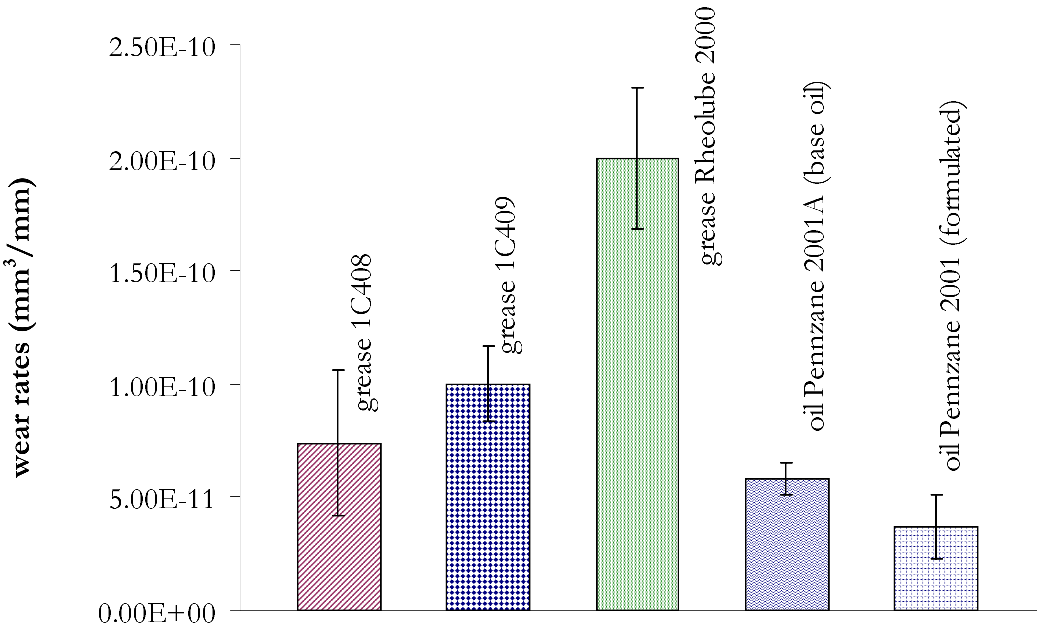


Figure 6: Wear rates (mm^3/mm) of the different Pennzane® oils and greases

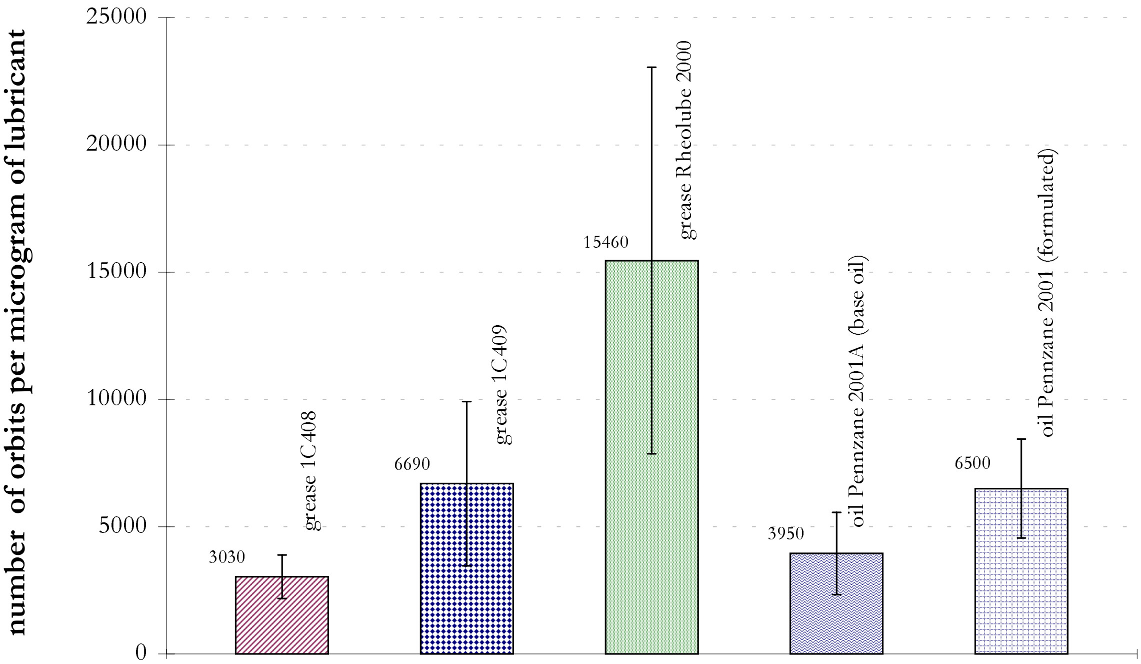
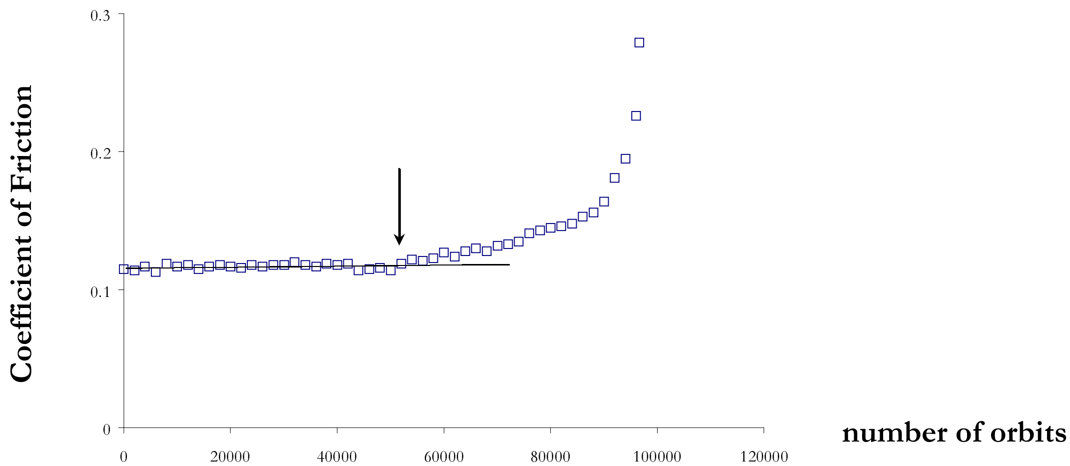
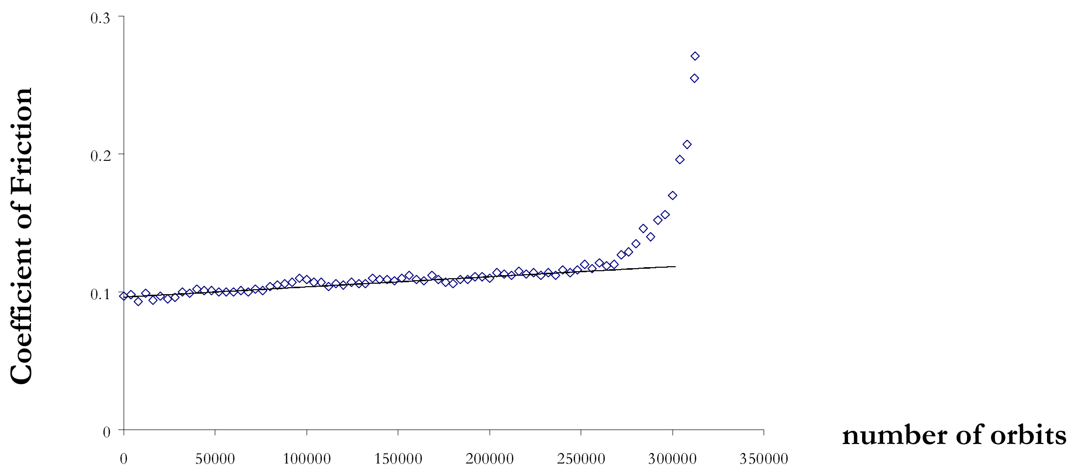


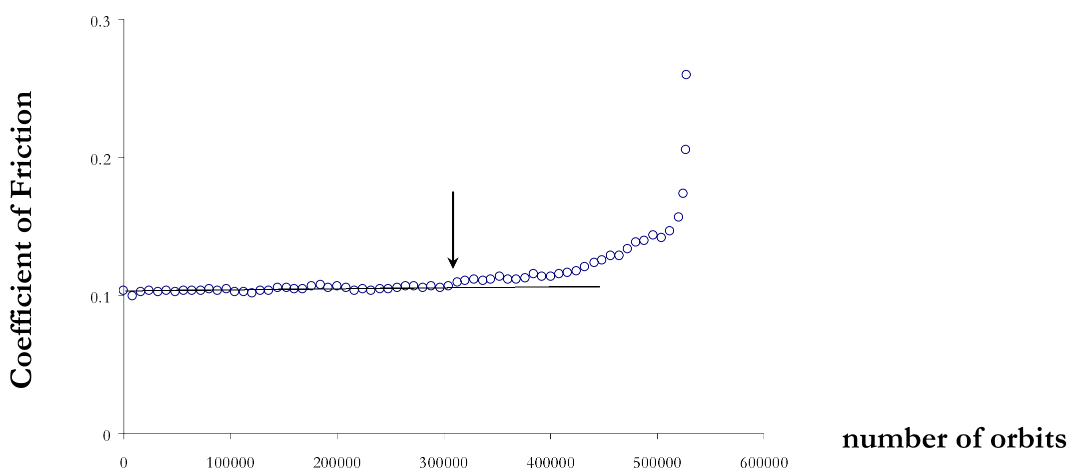
Figure 7: Normalized lifetimes of the Pennzane® greases and oils (with standard deviation for the four tests)



(a) grease 1C408 (Li soap)



(b) grease 1C409 (urea derivative)



(c) grease Rheolube® 2000 (Na soap)

Figure 8: Examples of friction coefficient traces

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13. ABSTRACT ***(Maximum 200 words)***

Commercial greases for space applications usually fulfill the requirements imposed by the severe conditions of use. The main requirement is their ability to create an EHL film, boundary film, or both under speed, load and temperature conditions that the mechanisms will operate. Three greases, all based on a multiply alkylated cyclopentane (Pennzane®) base oil, were studied. The thickeners were an n-octadecylterephthalamate soap, a lithium soap, and a urea derivative. A Four-Ball Tribometer and a Spiral Orbit Tribometer were employed to evaluate the greases under ultrahigh vacuum. Results indicated that all three greases yielded very low wear rates and extended lifetimes. In addition, routine physical property data is reported for each grease.

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