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Tribochemistry of Multiply-Alkylated Cyclopentane Oils on DLC-Coated Thrust Bearings

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14. ABSTRACT

In contrast to typical DLC coatings, hydrogenated DLC (H-DLC) coatings exhibit extremely low friction in vacuum and dry atmospheres, suggesting their potential importance for spacecraft applications. We have conducted a study of H-DLC-coated steel thrust bearings, lubricated with a multiply-alkylated cyclopentane oil, either unformulated, or formulated with lead naphthenate or an aryl phosphate ester mixture. Data on uncoated steel thrust bearings were obtained for comparison. The surface chemistry of the additives on worn H-DLC surfaces was evaluated along with chemical analysis of the residual lubricant. In contrast with results on uncoated steel bearings, minimal additive-based tribofilm formation was detected on the surfaces of the H-DLC coatings in the wear tracks. The results indicate that additives optimized for steels may not be appropriate for H-DLC coatings. Although there were indications that H-DLC coatings increase endurance, the high roughness of the bearings contributed to statistical uncertainty. Future studies are planned with higher quality bearings.

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1. Introduction

Diamond-like carbon (DLC) coatings are increasingly being used in a wide range of applications, including razor blades, hard drives, automotive parts, and space mechanisms.[1–4] DLC films have demonstrated high sensitivity to water vapor in the environment[5–8] and hydrogen content of the films.[9–12] In particular, typical DLC with low hydrogen content exhibits low coefficients of friction (COFs) in humid air and higher friction in dry and vacuum environments. In contrast, appropriately hydrogenated DLC (H-DLC) exhibits very low friction in dry and vacuum environments (i.e., COF < 0.01), and higher friction in humid air (i.e. ~ 0.07).

Because of its low friction in vacuum, along with its inherent high hardness, H-DLC coatings are an attractive possibility for spacecraft mechanisms. Much of the research into DLC coatings, in general, has involved sliding friction/wear tests, and there have been relatively few studies investigating the applicability of H-DLC (or even non-hydrogenated DLC) coatings for use in ball bearings.[13–16] In addition to their hardness and low friction, they also are attractive for liquid-lubricated ball bearing applications because their relative inertness could reduce tribochemical breakdown of the lubricant due to metal-to-metal contact, functioning as a barrier coating as long as they remain intact.

Most spacecraft heritage lubricant formulations have additive packages that are intended for use with steel. As such, the eventual application of H-DLC coatings in spacecraft bearings will require data on how they behave with heritage lubricants. These data may show that development of more appropriate additives is required. The primary goal of this study is to evaluate chemical effects of spacecraft lubricants with H-DLC-coated bearings, and compare to their chemistry on uncoated steel bearings. To this end, we conduct post-test surface analysis of the bearing surfaces, as well as chemical analysis of residual lubricant. Another goal is to determine whether H-DLC coatings can provide performance improvements for spacecraft ball bearings, including increased endurance.

2. Experimental Method

The bearings were GT-1 thrust bearings from INA. The races were made of 1117 steel, and the balls were 52100 steel. The ball retainer was the steel-ribbon retainer furnished with the thrust ball bearing. The surface roughness of raceways was in the range R_a = ~0.25–0.41 μm (10–16 μin), which is greater than typical spacecraft bearing components. However, R_a actually overstates the surface quality because of the presence of a relatively small number of large surface asperities; R_z (which is a better measure of the average height of a small number of asperities) for these bearings is in the range 1.2–1.9 μm (50–75 μin). The components were washed using heptane, cleaned using Brulin 815GD detergent (diluted in H_2O), rinsed in distilled H_2O, and dried using nitrogen gas under pressure. H-DLC coatings were deposited to a thickness of 1 μm on some of the bearing race surfaces. They were deposited by plasma-enhanced chemical vapor deposition (PACVD) as described in Reference 10.

The bearings were lubricated with multiply-alkylated cyclopentane (MAC) oils. Three formulations obtained from Nye Lubricants, Inc., were used for the testing: unformulated MAC (NSO2001A), MAC formulated with a 1% w/w mixture of aryl phosphate esters (NSO2001), and MAC formulated with 3% w/w lead naphthenate (Pbnp) (these lubricants are commonly known as "Pennzane"). Testing was conducted with either 10 or 20 μl of oil. The results indicated that the bearing endurance was not affected by which volume was used.

Dynamic tests were performed using our in-house, eccentric bearing wear test facility (see Figure 1), which has been described previously.[17] Because two grooved thrust bearing races were used, the

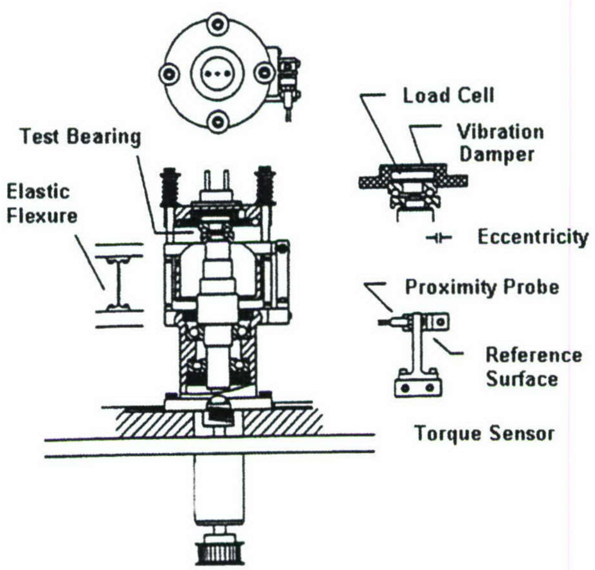

Figure 1. Schematic of the thrust bearing tester used in this work. It can be used with an eccentricity (i.e., the axis of a flat thrust washer can be offset from the axis of the opposing thrust bearing race), but the eccentricity was set to zero for the present study.

eccentricity was set to zero (i.e., the rotation axes of the races coincided). A load of 107 N was used (8.9 N per ball), giving a maximum Hertzian contact stress of ~0.668 GPa (97 ksi). The wear tester was contained inside a turbomolecular-pumped vacuum chamber. Bearing tests were run at pressures below 1.3×10^{-4} Pa (1×10^{-6} Torr). A rotation speed of 1800 rpm was used. The bearing temperatures were controlled using a water-cooled fixture to about ~20°C (temperature not measured). Torque values were monitored using LabView-based software, and the tests were stopped if the torque increased to greater than 30% of the initial running torque. A number of tests were stopped prior to reaching the failure criterion, either to allow their pre-failure chemical state to be evaluated, or because they reached the maximum test duration of 17–20 million cycles (20 million cycles takes a week to complete).

After each test was completed, the lubricant was removed from the ball bearings by rinsing with heptane solvent, followed by evaporation of the solvent at room temperature with a stream of dry nitrogen to reduce the analysis volume and concentrate the samples. Some of the lubricant samples were analyzed using a Nicolet Magna-IR 550 FTIR spectrometer in the internal reflectance mode. The spectra were taken with a resolution of 2 cm^{-1}. "Difference spectra" were obtained by converting the spectra from percent transmittance to absorbance, and subtracting two spectra. Prior to subtraction, the spectra were normalized so that the resulting difference spectrum gave equal areas above and below the zero line.

Some of the raceways (with the residual lubricant removed by heptane washing, as described above) were analyzed using a Physical Electronics 680 Auger Nanoprobe, from which secondary-electron images (similar to scanning electron microscope images), and Auger electron spectra were obtained. Composition analysis of the bearing surfaces were obtained from the Auger electron spectra. The depth sensitivity is in the range 0.5 to 2 nm, depending on which element is being analyzed.

3. Results

3.1 Testing of H-DLC-Coated and Uncoated Thrust Bearings with MAC/Pbnp Oil

For thrust bearings tested with MAC/Pbnp, no failures were seen for either H-DLC-coated or uncoated races. Four of the bearings were allowed to continue to the test maximum of 17 to 20 millions cycles (see Figure 2). Our tests were not long enough to distinguish endurance between coated and uncoated bearings with MAC/Pbnp. However, there were marked differences in running torques: the average running torques for the H-DLC-coated samples were about half that of the uncoated samples.

An additional uncoated thrust bearing was tested with MAC/Pbnp oil for only 2.4 million cycles (without failure). Secondary-electron images (see Figure 3a and higher magnification Figure 3b) show a light wear pattern on the race surface. AES (Figure 4) of the two types of wear features shown in Figure 3b indicate the deposition of a tribofilm containing Pb from the Pbnp additive. In both regions, the amount of Pb was ~2 at% (some Fe and O was also detected, along with large amounts of C), indicating minimal formation of additive-based tribofilm. A bearing similarly tested for 20.7 million cycles (see Figure 5 showed a more significant wear pattern, with the production of debris. The amounts of Pb in the Areas 1 to 3 in Figure 5 were 10, 9, and 7 at%, respectively, representing worn regions of the surface (see AES results in Figure 6). Area 4 is a debris particle, and

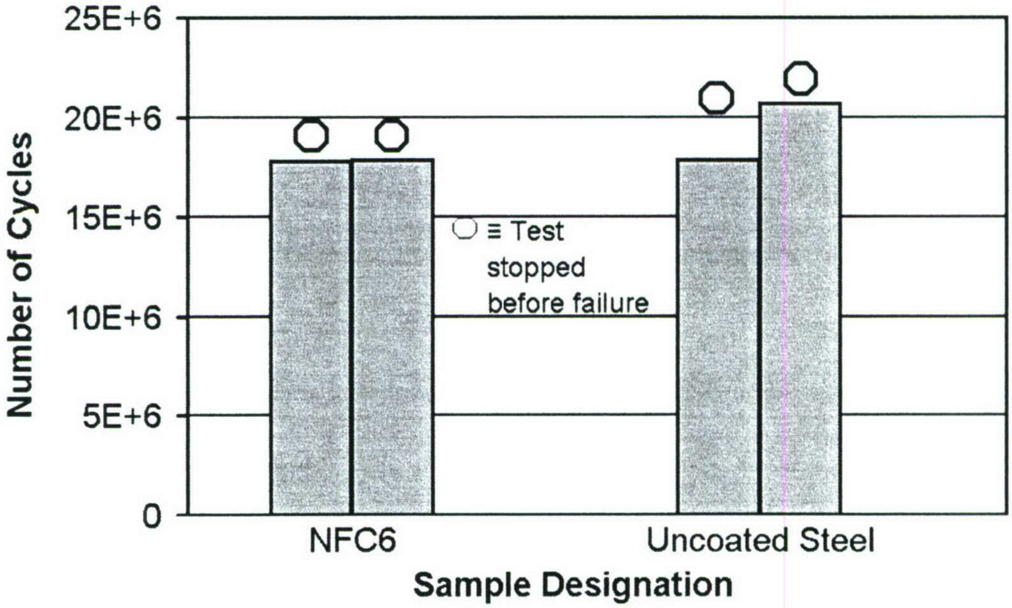

Figure 2. Summary of endurance testing of thrust bearings lubricated with MAC oil formulated with Pbnp. Tests were allowed to run until either failure occurred, or 17 to 20 million cycles were reached. H-DLC-coated bearings were tested along with uncoated steel.

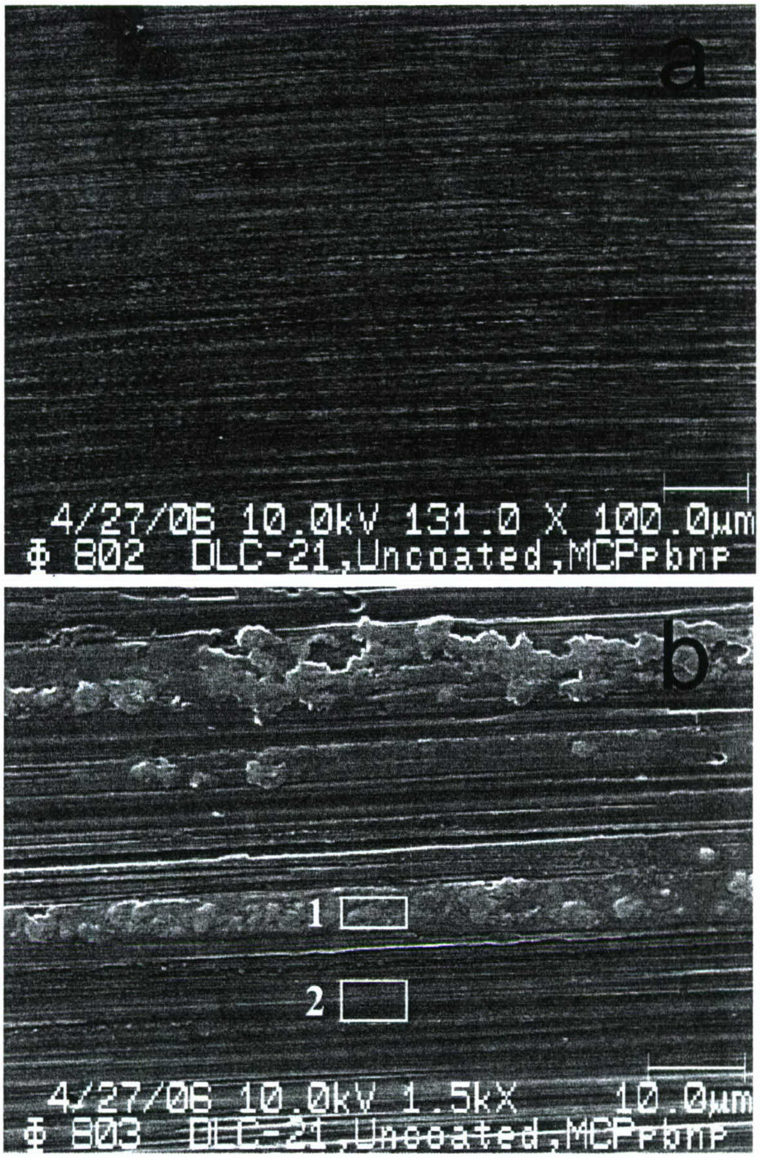

Figure 3. Scanning electron micrograph of the surface of a race of an uncoated steel thrust bearing, lubricated with MAC/Pbnp oil after running for 2.4 Mcycles without failure. Micrographs are shown near the center of the wear track, at magnifications of (a) 131X and (b) 1500X.

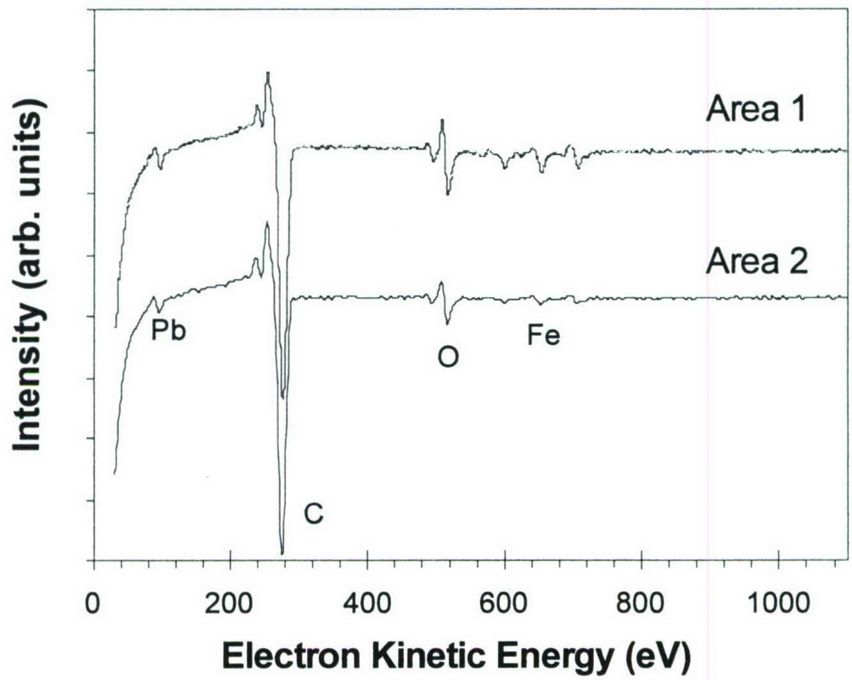

Figure 4. AES analysis of the surface of a race of an uncoated steel thrust bearing lubricated with MAC/Pbnp oil after running for 2.4 Mcycles without failure. Areas are shown in the SEM photomicrograph in Figure 3b.

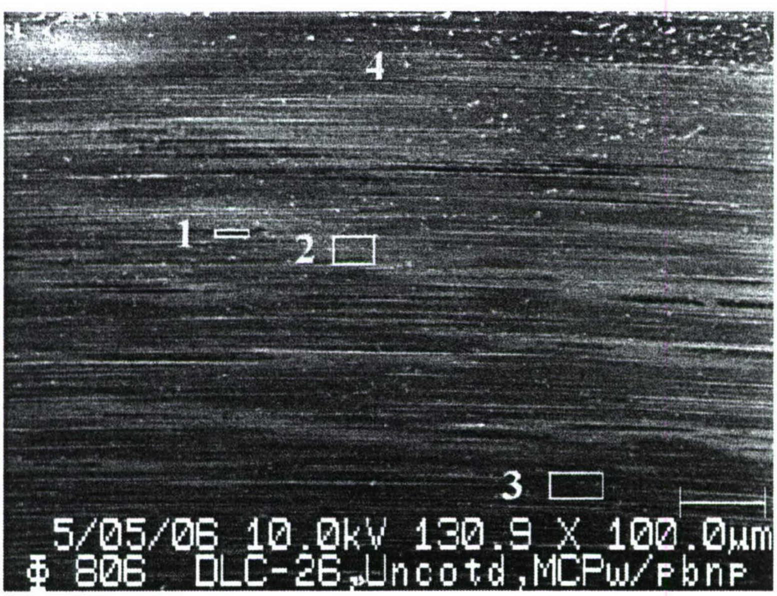

Figure 5. Scanning electron micrograph of the surface of a race of an uncoated steel thrust bearing lubricated with MAC/Pbnp oil after running for 20.7 Mcycles without failure. The area shown is near the center of the wear track. Areas noted on the micrograph were chosen for AES analysis.

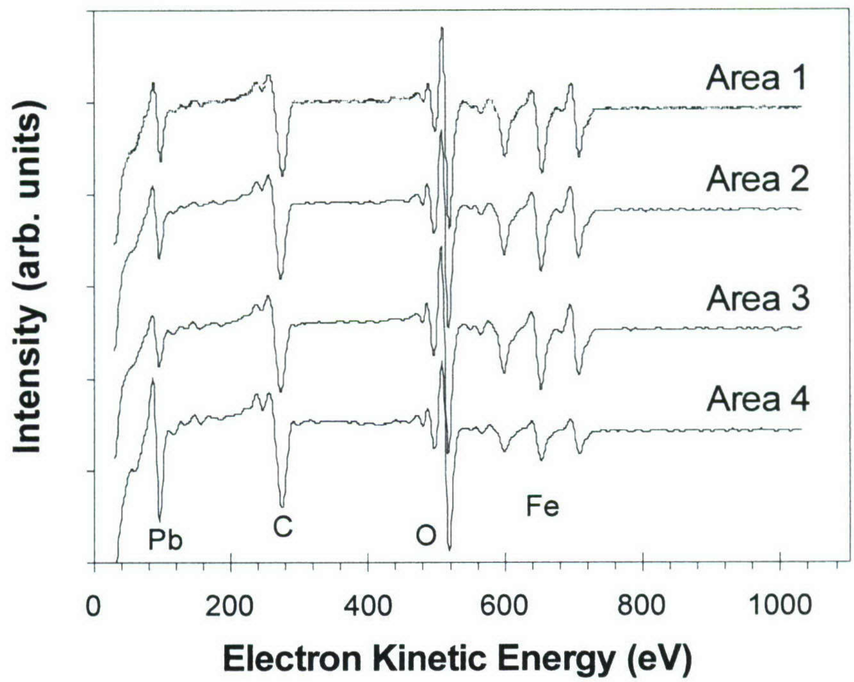

Figure 6. AES analysis of the surface of a race of an uncoated steel thrust bearing lubricated with MAC/Pbnp oil after running for 20.7 Mcycles without failure. Areas are shown in the SEM photomicrograph in Figure 5.

contains a larger amount of Pb (17 at%). The results on the bearing run for 20.7 million cycles describe a significant additive-based tribofilm formation after 20 million cycles, with some wear of the film to produce Pb-containing debris.

Figure 7a shows the surface of a MAC/Pbnp-lubricated, H-DLC-coated thrust bearing after a test was stopped at 2.2 million cycles without failure. The micrograph shows that mild wear of the coating has occurred. AES spectra showed that in Areas 1, 2, and 3, no Fe from the substrate was detected (spectra not shown), so that the coating was primarily intact. About 2 at% Pb was detected in these areas. However, in Area 3, some Fe was detected, with a corresponding increase in Pb concentration (i.e., 5 at%).

An SEM micrograph of a bearing tested under similar conditions, but allowed to run for 17.8 million cycles without failure, is shown in Figure 7b. Areas 1 and 2 represent intact H-DLC coating (the Fe detected in Area 1 is from Fe-containing debris). In both these areas, there was again only ~2 at% Pb detected (see Figure 8), in contrast to the higher cycle test for an uncoated bearing, where greater Pb-based tribofilms had formed (see above). In Area 3, breach of the coating had occurred (Pb determination was complicated in this case by the presence of a Si peak arising from an adhesion-enhancement layer).

FTIR spectra are shown in Figure 9 for residual lubricant obtained from both uncoated and H-DLC-coated thrust bearings after testing for varying numbers of cycles. This region is highly sensitive to the chemical state of the Pbnp in the oil. There are some subtle differences in the peak shapes, indi-

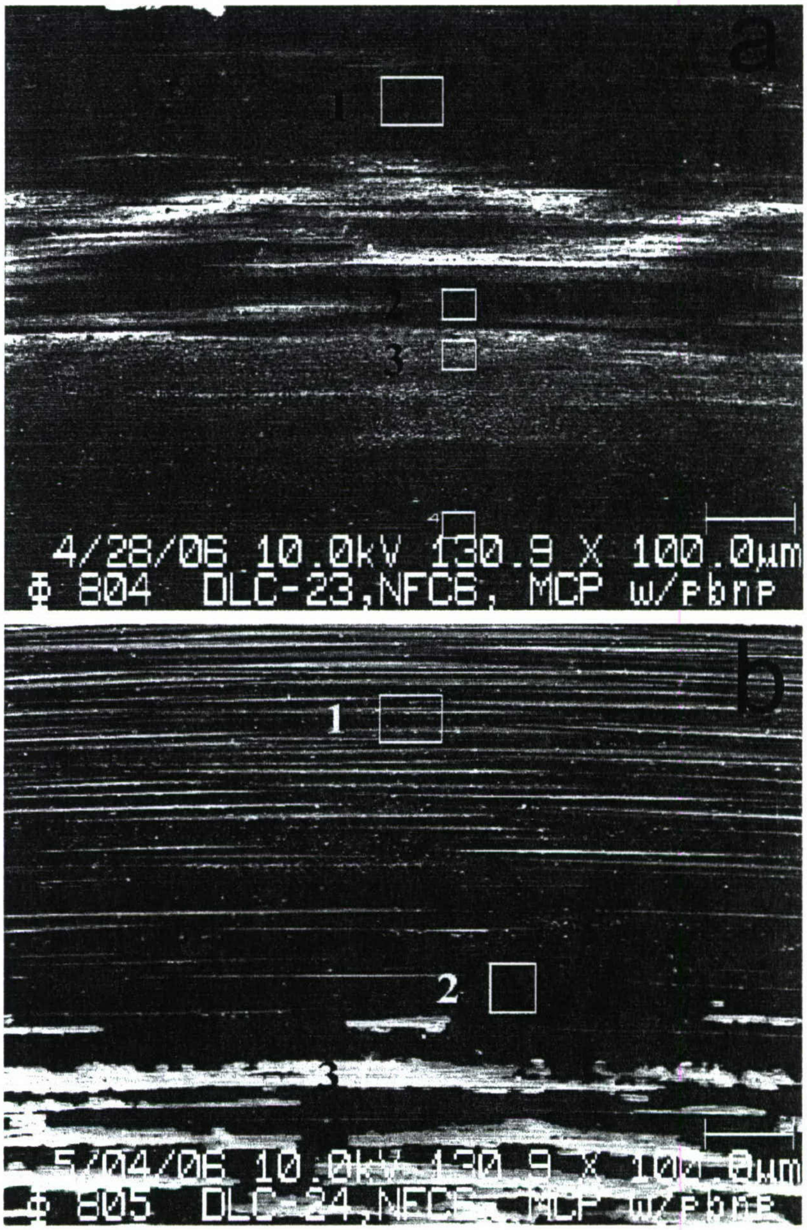

Figure 7. Scanning electron micrograph of the surface of a race of an H-DLC-coated steel thrust bearing lubricated with MAC/Pbnp oil after running for (a) 2.2 Mcycles without failure, and (b) 17.8 Mcycles without failure. The area shown is near the center of the wear track.

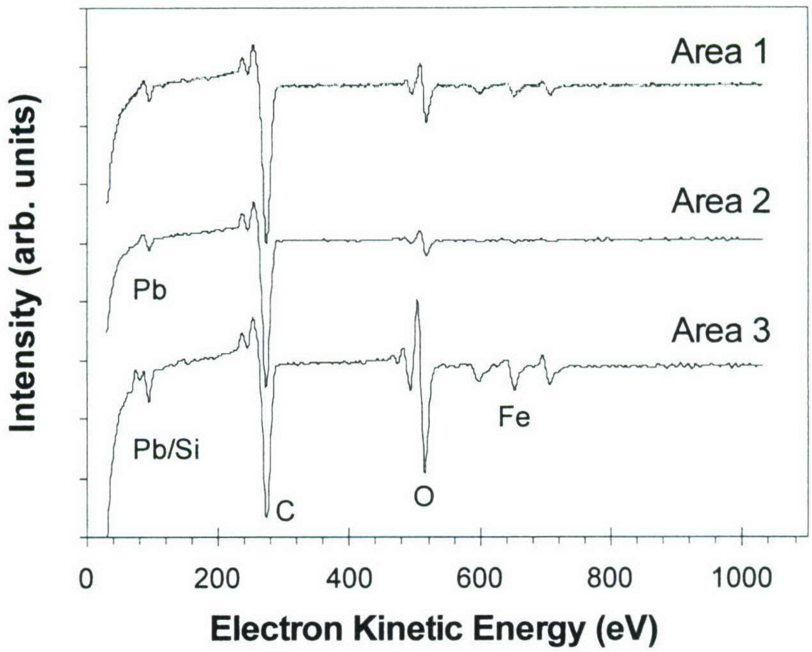

Figure 8. AES analysis of the surface of a race of an H-DLC-coated steel thrust bearing lubricated with MAC/Pbnp oil after running for 17.8 Mcycles without failure. Areas are shown in the SEM photomicrograph in Figure 7b.

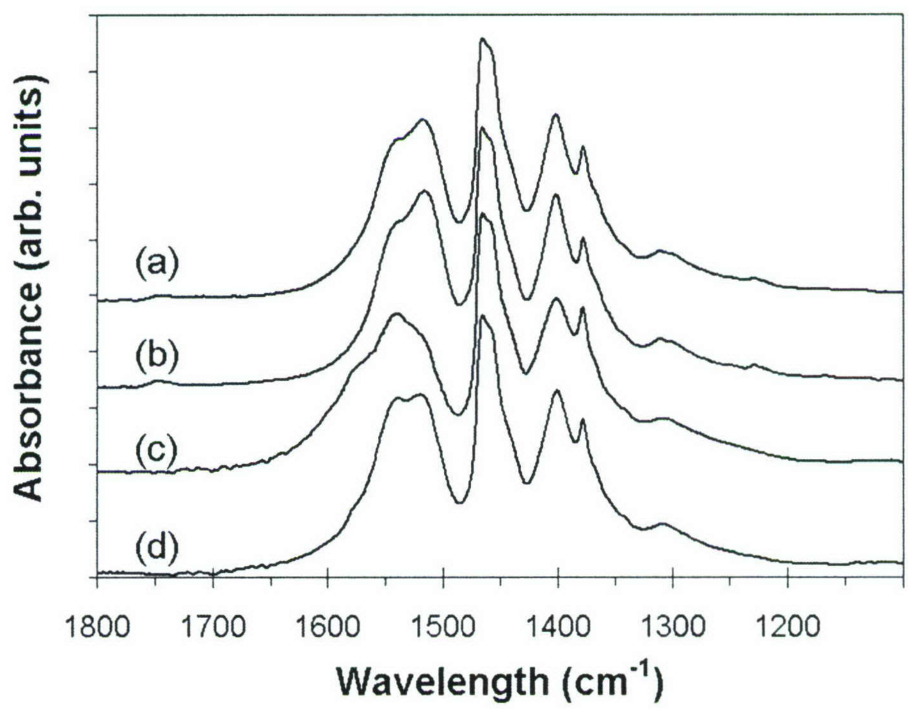

Figure 9. FTIR spectra of residual MAC/Pbnp oil from thrust bearings. Spectra are shown for uncoated steel thrust bearings tested for (a) 2.4 Mcycles and (b) 0.5 Mcycles, and for H-DLC coated bearings tested for (c) 17.8 Mcycles and (d) 2.2 Mcycles.

cating differences in chemical structure. To better understand these changes, FTIR difference spectra were obtained that elucidated differences in bearings tested for differing amounts of time. Figure 10a shows an FTIR difference spectrum for H-DLC-coated bearings, with the spectrum for the 2.2 million

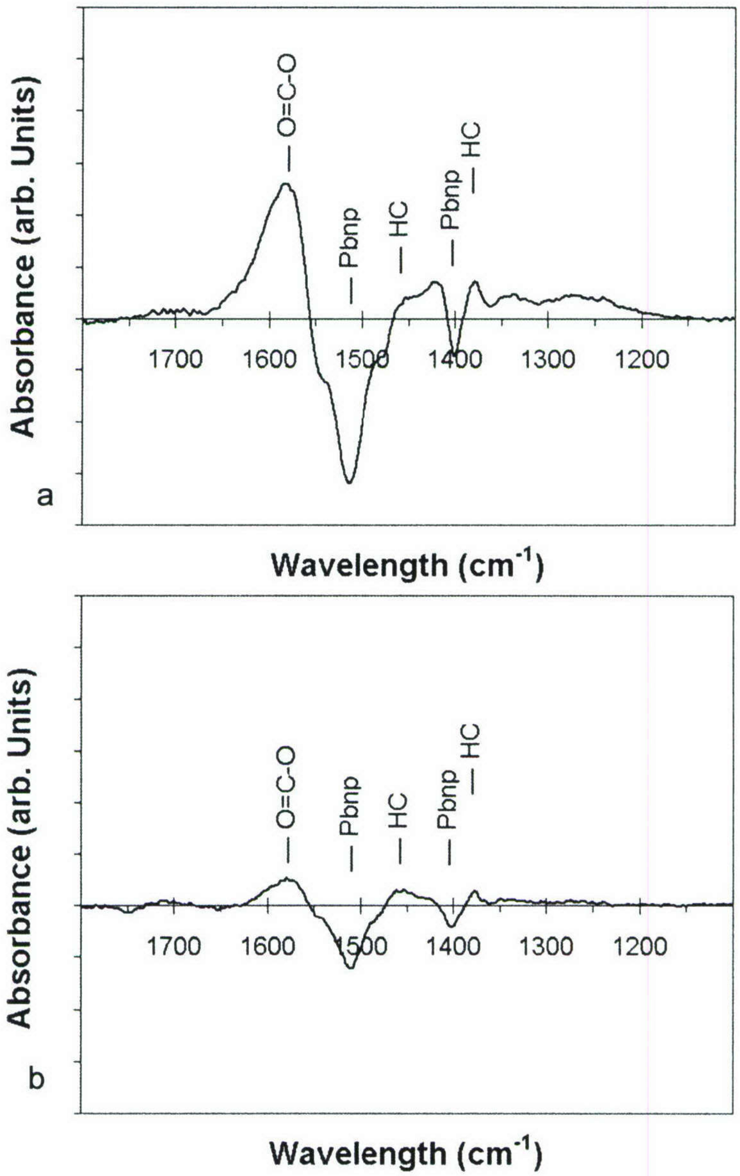

Figure 10. FTIR difference spectrum of residual MAC/Pbnp oil from (a) H-DLC-coated thrust bearings tested for 17.8 Mcycles and 2.2 Mcycles, and from (b) uncoated steel thrust bearings tested for 2.4 Mcycles and 0.5 Mcycles. The two spectra were rescaled and subtracted so that the net areas above and below the zero line approximately cancel out. Functional group assignments are shown above the difference spectrum. Positive intensity indicates a relative increase in the functional group for the 17.8 Mcycle test, while negative intensity indicates a corresponding decrease.

cycle test subtracted from that for the 17.8 million cycle test. The difference spectrum indicates a reduction in the amount of unreacted Pbnp additive, with a corresponding increase in the amount of a different carboxylate species. This type of reaction has been shown to occur in steel test bearings with Pbnp-containing hydrocarbon oils, and indicates chemical reaction of the oil additive.[18,19]

A similar difference spectrum is seen in Figure 10b for uncoated thrust bearings, comparing tests for 2.4 and 0.5 million cycles. The shape of the spectrum is similar to that shown in Figure 10 for the H-DLC-coated bearings. These results indicate that degradation of the additives occurs with both H-DLC-coated and uncoated bearings, although tribofilm deposition seems to occur only for uncoated bearings.

3.2 Testing of H-DLC-Coated and Uncoated Thrust Bearings with NSO2001 Oil

A series of tests was conducted for H-DLC-coated and uncoated thrust bearings that were lubricated with NSO2001 oil (i.e., MAC with an aryl phosphate additive mixture). Figure 11 shows the endurance results for a series of different types of H-DLC coatings, as well as uncoated steel. There is significant scatter in the results. Most of the bearings (both coated and uncoated) failed in the range of 1 to 7 million cycles, although one H-DLC bearing lasted the full 20 million cycle test duration. The results indicate that under the conditions of our test, the use of aryl phosphate esters as additives provides lower wear protection than does the Pbnp additive. The proven success of aryl phosphate esters in high-quality angular contact bearings indicates that the high roughness and corresponding greater boundary interactions in these tests are better mitigated by the Pbnp additive. However, the observation that one test lasted much longer indicates that H-DLC coatings can potentially provide mitigation of this lowered endurance.

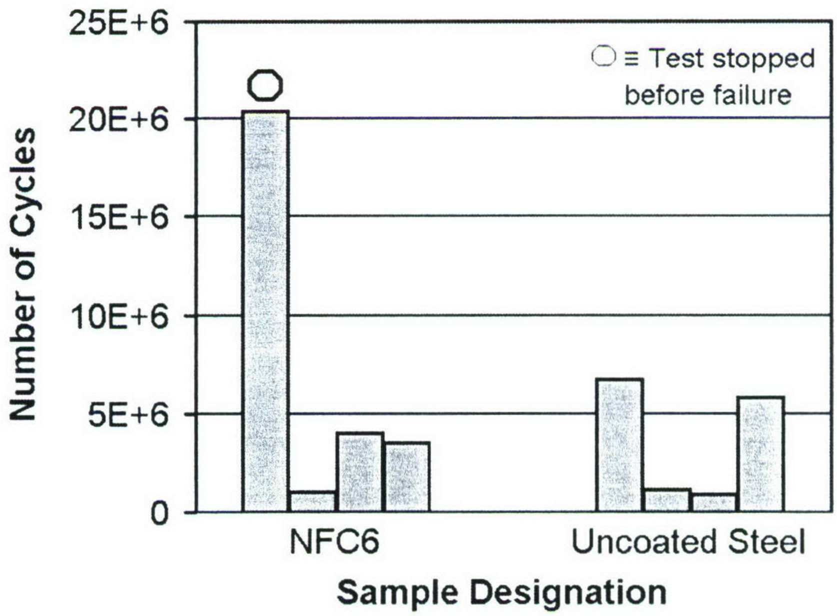

Figure 11. Summary of endurance testing of thrust bearings lubricated with NSO2001 oil (i.e., MAC formulated with phosphate additives). Tests were allowed to run until either failure occurred, or 20 million cycles was reached. H-DLC coatings were tested along with uncoated steel.

Figure 12a is an SEM micrograph of the H-DLC-coated thrust bearing that ran for 20.4 million cycles without failure. Similar to the MAC/Pbnp-lubricated bearings, there is evidence of loss of the H-DLC coating in some sections of the wear track, in the lighter regions seen in the micrograph. Figure 12b shows a magnified view of this region, and AES (Figure 13) confirmed that the coating has been removed in these regions (i.e., Fe is detected). P is only detected in the regions where the coating has been removed, i.e., 0.2–0.5 at% P in Areas 1 and 3. This is similar to the amount of P detected on an uncoated thrust bearing surface after testing, i.e., 0.3 at% (spectra not shown). In contrast, there is virtually no P detectable in areas where the coating is intact (i.e., in Area 2).

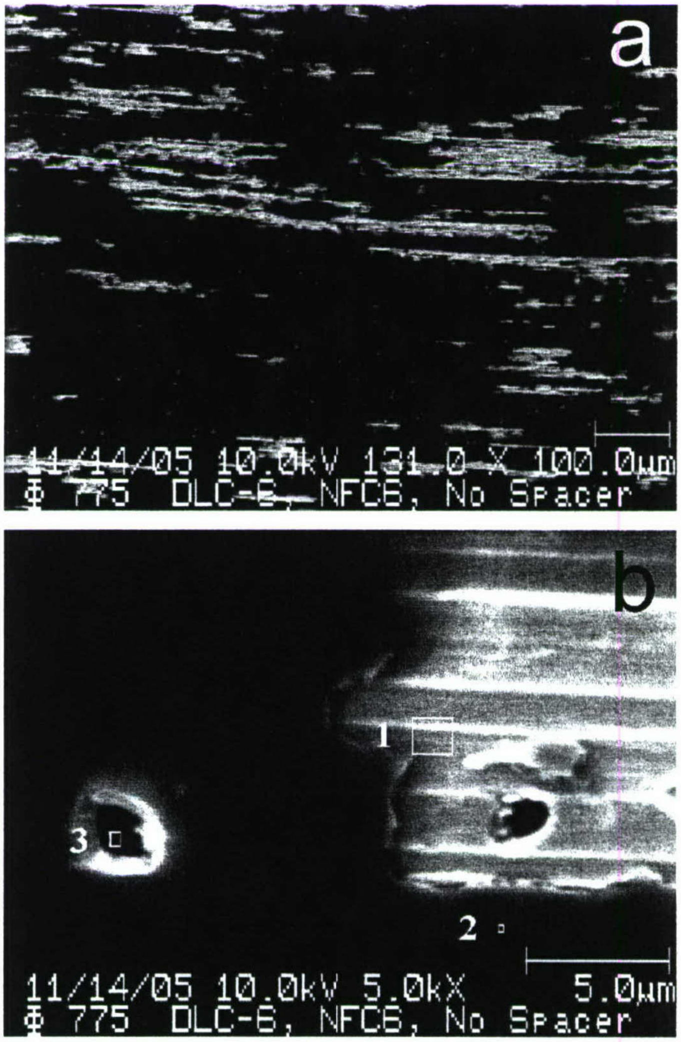

Figure 12. Scanning electron micrograph of the race of an H-DLC-coated thrust bearing lubricated with NSO2001 oil (i.e., MAC/phosphate) after running for 20.4 Mcycles without failure. The micrographs are of areas near the center of the wear track, and are shown at magnifications of (a) 131X and (b) 5000X.

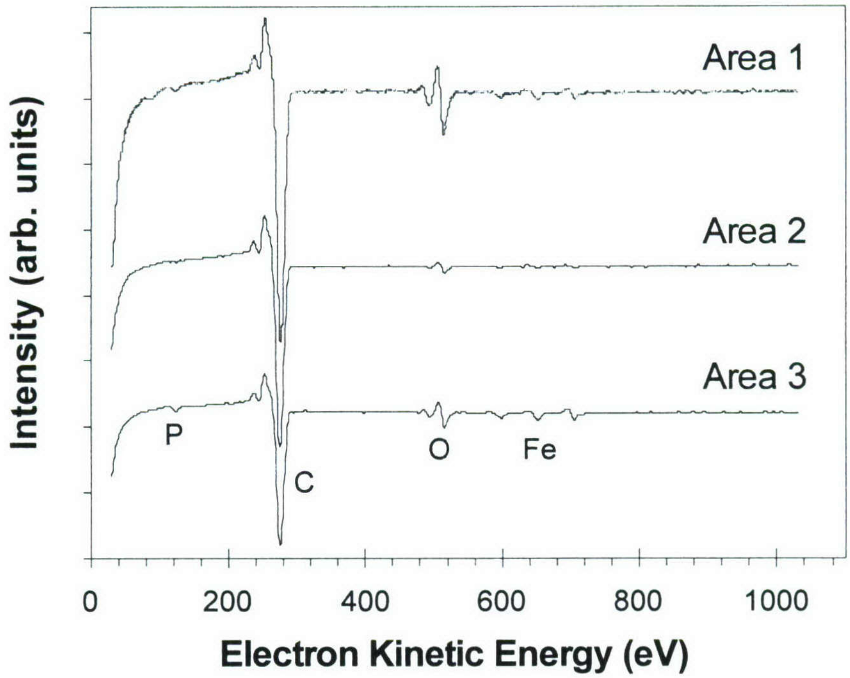

Figure 13. AES analysis of the surface of the race of an H-DLC-coated thrust bearing lubricated with NSO2001 oil (i.e., MAC/phosphate) after running for 20.4 Mcycles without failure. Areas are shown in the SEM photomicrograph in Figure 12b.

An SEM micrograph near the edge of the wear track is shown in Figure 14. In this region, the wear of the coating is much lower than in the more central part of the wear track represented by Figure 12. The P detected on both the unworn and worn H-DLC coating is below the detection limit of AES (see Figure 15).

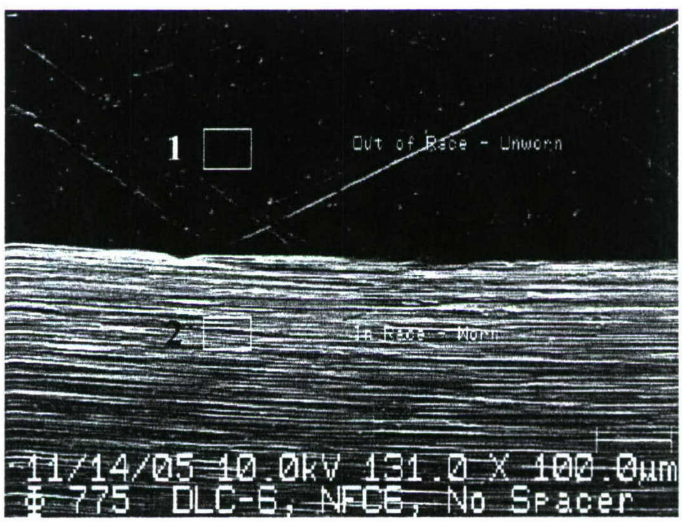

Figure 14. Scanning electron micrograph of the edge of a race of an H-DLC-coated thrust bearing lubricated with NSO2001 oil (i.e., MAC/phosphate) after running for 20.4 Mcycles without failure. Areas noted on the micrograph were chosen for AES analysis.

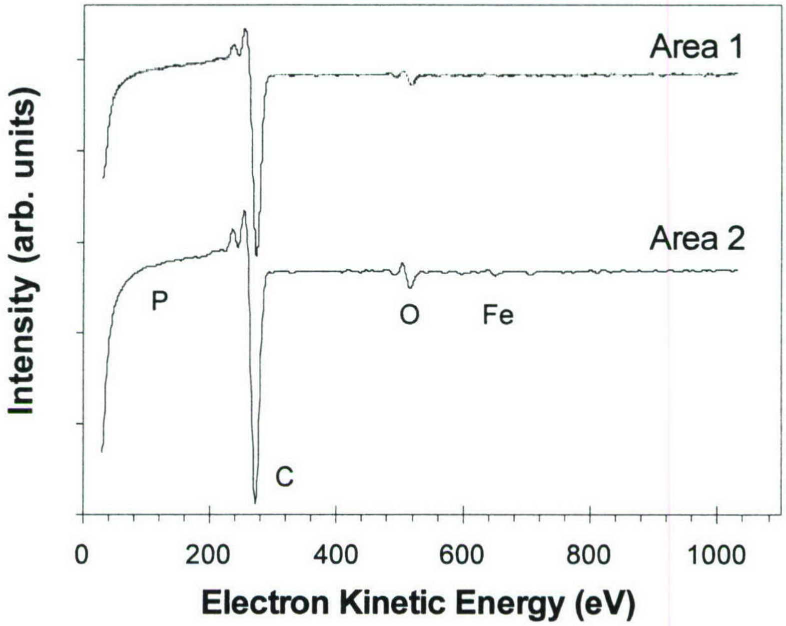

Figure 15. AES analysis of the edge of the race of an H-DLC-coated thrust bearing lubricated with NSO2001 oil (i.e., MAC/phosphate) after running for 20.4 Mcycles without failure. Areas are shown in the SEM photomicrograph in Figure 14.

FTIR analysis was conducted on residual NSO2001 oil obtained from three uncoated thrust bearings and three H-DLC-coated bearings. Figure 16 shows spectra from a number of thrust bearing samples

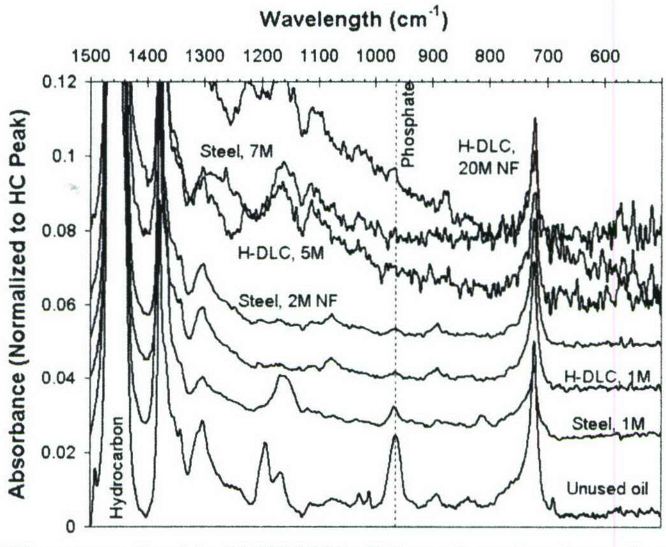

Figure 16. FTIR spectra of residual NSO2001 oil from thrust bearings. Spectra are shown for both H-DLC-coated and uncoated steel thrust bearings, tested from 1 million cycles up to 20 million cycles. Tests that were stopped before failure have the notation "NF" at the end of the label. A spectrum for unused oil is shown for comparison. The spectra were normalized so that the hydrocarbon peaks at 1450 cm^{-1} are the same height. The phosphate peak at 960 cm^{-1} represents the nondegraded aryl phosphate ester additives in the oil.

run from 1 to 20 million cycles. They were normalized to have equal hydrocarbon peak heights, and are compared to a spectrum for unused oil. The phosphate peak at 960 cm^{-1} represents phosphate functional groups present in the aryl phosphate lubricant additives. This peak is shown to decrease in height with increasing numbers of cycles, irrespective of whether they were coated or uncoated, or whether they had failed or were stopped before failure. This indicates a mechanism that is not surface mediated. Lack of surface chemistry as the cause is reinforced by the different amounts of phosphate-derived tribofilms on the surface detected by AES, as discussed above. Evaporation might explain the peak lowering: the more volatile phosphate esters in the oil could have been lost because the pumped chamber is an open system (unlike, for example, sealed spacecraft hardware). These more volatile components comprise ~70% of the total phosphate additive content in the oil,[20] and therefore could lower the FTIR phosphate peak height so that it is comparable to the noise level. Unfortunately, we have not identified a reacted phosphate peak, as we have done for Pbnp (see Subsection 3.1).

3.3 Testing of H-DLC-Coated and Uncoated Thrust Bearings with NSO2001A Oil

Endurance results for a number of H-DLC-coated and uncoated thrust bearings lubricated with NSO2001A oil (i.e., unformulated MAC) are shown in Figure 17. The Figure shows that there is a statistically significant improvement in bearing endurance for the H-DLC coated bearings, although one of them lasted approximately the same amount of time as the three uncoated bearings. This improvement is likely due to the H-DLC coating preventing tribochemical attack of the bearing steel by the lubricant.

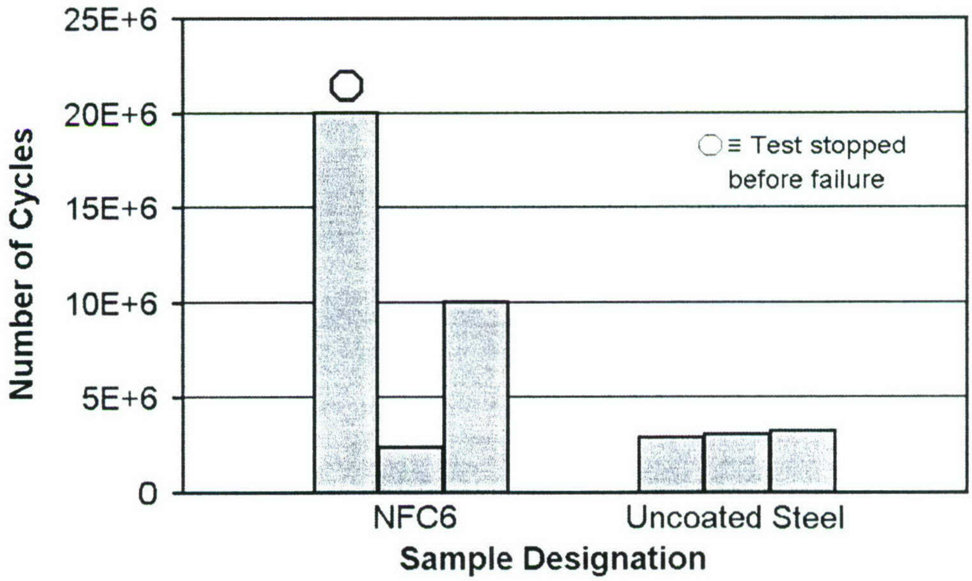

Figure 17. Summary of endurance testing of thrust bearings lubricated with NSO2001A oil (i.e., unformulated MAC). Tests were allowed to run until either failure occurred, or 20 million cycles was reached. H-DLC-coated bearings were tested along with uncoated steel.

4. Discussion

The roughness of the bearings prior to coating, with the resultant spalling of the H-DLC coating after millions of cycles of wear, provides the opportunity of comparing H-DLC/additive surface chemistry to steel/additive surface chemistry on the same bearing. For oils with Pbnp additives, there appears to be minimal interaction of the H-DLC with the Pbnp additive in the oil in the wear track. This is illustrated by the similar amounts of Pb detected on both worn and unworn H-DLC surfaces, indicating that the Pbnp is only physisorbed or chemisorbed on the surface with minimal decomposition of the Pbnp molecule, and no significant tribofilm formation. In contrast, on uncoated steel bearings, and in regions of coated bearings where the H-DLC coating has spalled off, there are significantly higher amounts of Pb detected. For testing of H-DLC-coated bearings with the phosphate-containing oil (NSO2001), we see similar results. Specifically, there is no detectable P on either the worn or unworn H-DLC surfaces. In contrast, we detect small amounts of P on bare steel surfaces that had been subjected to wear, i.e., worn uncoated steel bearing surfaces, and in regions where H-DLC has spalled off of coated bearings.

It is not surprising that there are higher P concentrations on the steel surfaces of bearings tested with NSO2001, compared with Pb concentrations seen on corresponding surfaces tested with MAC/Pbnp oils. This is in agreement with previous studies of surface chemistry of additives on bearing surfaces.[21] This observation does not necessarily indicate that the tribofilm thickness on steel is smaller for phosphate additives compared with Pbnp additive. In fact, the high C AES intensity indicates that there are thicker carbon-containing tribofilms on the steel surfaces tested with NSO2001 oil (e.g., compare Figure 6 with Figure 13).

For uncoated steel thrust bearings, there is a clear wear life improvement for Pbnp-containing oil compared with oil containing aryl phosphate esters. However, conditions in our tests are very different than for typical spacecraft angular contact ball bearings. A big difference is the high roughness in our bearings compared to high-quality spacecraft bearings; the Pbnp may provide better boundary protection at the asperity contacts.

For both the coated and uncoated bearings, the variation in the endurance results clearly indicate that the bearing roughness—specifically, asperities on the race surfaces—is controlling the operational lifetime of the bearings. In the case of the H-DLC-coated bearings, several of the coatings last for millions of cycles with the coatings intact, but eventually exhibit localized spalling in the center of the wear track. This spalling is likely initiated at asperities, where the local Hertzian contact stress is significantly higher than that averaged over the contact region. Otherwise, we would see a larger, more uniform area of coating wear located at the center of the wear track.

When testing with unformulated oil (NSO2001A), there is a statistically significant endurance enhancement for H-DLC-coated bearings compared to uncoated bearings when tested with unformulated oil. Although the bearing roughness does affect the variability of the endurance results, the coatings clearly are providing boundary protection during testing. In contrast, there appears to be (statistically) poorer performance of H-DLC-coated bearings when used with NSO2001 oil, which contains aryl phosphate additives. The cause of this phenomenon cannot be elucidated based on the results of the current study, but could be due to a weakening of H-DLC/substrate adhesion by the presence of the additive.

Future studies should help to clarify the results presented here, including the high variability of bearing endurance, as well as the apparent performance degradation for the H-DLC-coated bearings caused by the presence of aryl phosphate additives. In particular, we are conducting follow-on studies with higher quality bearings, i.e., that exhibit considerably smaller surface roughness. However, the results of the present study do indicate that there is a wear-life enhancement due to the use of H-DLC coatings. This is highlighted by the observation that, for bearings tested with NSO2001 or NSO2001A oil, the only thrust bearings enduring longer than 7 million cycles are bearings with H-DLC coatings.

5. Summary

In this study, steel thrust bearings were tested with and without H-DLC coatings, using three different multiply-alkylated cyclopentane (MAC) lubricant formulations (NSO2001A, NSO2001, and MAC with Pbnp). A primary goal was to investigate additive/surface chemistry to determine whether heritage additives could provide wear life enhancement when using H-DLC coatings. The results were compared with data obtained using unformulated MAC oil (NSO2001A). Post-test analysis of the bearing surfaces was conducted using electron microscopy and Auger electron spectroscopy, while analysis of the residual lubricant was conducted using Fourier-transform infrared analysis. For both Pbnp and the aryl phosphate esters, minimal additive-based tribofilm formation was detected on the surfaces of the H-DLC coatings in the wear tracks (relative to that on steels). The results indicate that additives suitable for steels may not be appropriate for H-DLC coatings. The high roughness of the bearings used in this study contributed to local spallation of H-DLC at asperities in the wear track after millions of cycles. Nevertheless, several H-DLC-coated samples tested with NSO2001 and NSO2001A oils endured for more than ten million cycles, longer than any uncoated thrust bearings evaluated with these oils. All coated and uncoated bearings tested using Pbnp-formulated MAC oil lasted the full length of the test without failure (i.e., >17 to 20 million cycles). In general, the roughness and roughness variation of the bearings contributed to statistical uncertainty regarding potential endurance enhancement of H-DLC coatings. Future studies planned with higher quality bearings should provide more definitive results.

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