

COMBINED DATA OF LIFETIME TESTS AND OUTGASSING EXPERIMENTS FOR LIQUID LUBRICATED BALL BEARINGS

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ABSTRACT

Two types of tests have been performed in NAL. Firstly, lifetime tests have been carried out in vacuum for liquid lubricated ball bearings. Three different PFPE oils and one SHF oil were impregnated in a polyimide retainer as a sole source of lubrication for these ball bearings. The torque, temperature and vacuum level were registered during the tests. Mass spectra were also recorded on a weekly basis. Secondly, the outgassing performance of these four oils -- either impregnated in a retainer or simply poured in a small metallic container -- was examined more accurately with a mass spectrometer in short-term experiments.

For each of the oils and for the retainer material, the outgassed species formed a «signature» which has been identified. The evolution of the mass peaks, as a function of temperature (in the case of the short-term tests) and as a function of time (in the case of the ball bearing tests) is presented. The analysis of the torque and the temperature, and also the comparison of the outgassing data, highlight the different steps in both the lubricant and the bearing life evolution. The results of the ball bearing surface analysis and the oil analysis (FTIR, gas chromatography) are discussed.

1. INTRODUCTION

Liquid lubricants are more and more widespread in space applications (Zaretsky, 1990). The increase of the satellite lifetime, the will to limit both noise and vibrations generated by rolling elements are the main reasons which can explain this situation. Nevertheless, this kind of lubrication may also exhibit some drawbacks such as outgassing pollution collected on optical surfaces, degradation of PFPE oils or lubricant starvation due to some creeping and/or non-wetting phenomena.

The aim of this study was to characterize the outgassing properties of four potential or recognized space lubricants as well as to determine the lifetime of ball bearings lubricated by those lubricants.

2. OUTGASSING EXPERIMENTS

2.1 Test with oil contained in a retainer

The outgassing experiments have consisted in heating ball bearing retainers, either dry or impregnated by oil, and registering the partial vapor pressure of exhausted

elements. These tests were conducted in a bell-jar vacuum chamber in which the pumping was assumed by a sorption pump firstly (down to 10^{-1} Pa), then relayed by a turbomolecular pump and a ion pump. The vacuum level at the beginning of the heating cycle was around $2-5 \cdot 10^{-5}$ Pa. The temperature increase was obtained by radiation emitted by a hot metallic ribbon. The temperature was controlled via the voltage imposed in the metallic ribbon. Two thermocouples measured the temperature in two different places. The first one (A) was entrapped in a small cylinder of copper narrowly adjusted in one retainer pocket whereas the other (B) hanged simply over the retainer which was laying flat.

The temperature cycle was divided in three steps. The first one was a ramp from ambient temperature up to $100-110^{\circ}\text{C}$ when measured by the thermocouple A ($\approx 140^{\circ}\text{C}$ by the thermocouple B). This temperature was maintained at this level during 45 minutes (second step) before stopping the heating. At regular intervals (≈ 5 minutes), an acquisition of the partial pressures of the gaseous species was completed by a mass spectrometer for the mass numbers ranging from 1 to 199. The final outgassing data were collected at temperatures near 50°C , more or less 3 hours after the beginning of the heating. After cooling, the test was repeated once.

The retainers are made of polyimide ($\text{HN}(\text{CO})_2$). The inner diameter of these retainers is 30.6 mm and their width is 13 mm.

Four oils, three perfluoropolyethers and one synthetic hydrocarbon, have been used to impregnate the retainers. Their main properties are summed-up in the table 1.

Name	PFPE Z	PFPE D	PFPE L	SHF
Molecular weight (gr/mol)	12800	8400	9500	910
Vapor pressure at 20°C (Pa)	$>10^{-10}$	$6.7 \cdot 10^{-9}$	$3.9 \cdot 10^{-10}$	$1.3 \cdot 10^{-10}$
Vapor pressure at 100°C (Pa)	$3 \cdot 10^{-8}$	$1.3 \cdot 10^{-5}$	$1.3 \cdot 10^{-6}$	$5.3 \cdot 10^{-5}$ (125°C)
Density at 20°C kg/m^3	1850	1894	1841	847
Viscosity at 20°C Pa.s	1.11	0.97	0.54	0.27

Table 1 : Physical properties of lubricants

They exhibit exceptional low vapor pressure and at this account have been extensively used or tested for space applications. A little less than 500 µl of lubricant was impregnated in each retainer, whatever was the lubricant. This corresponded to a saturated state for the retainers which impregnation rate was 23.5%.

2.2 Test with oil contained in a metallic recipient

It has sometimes revealed itself difficult to determine exactly what was the signature of the oils and what was the contribution of the cage. So, in a second time, outgassing tests in which the oil was poured in a small stainless steel cupel, have been performed. By doing so, we limited the sources of volatile components present in the vacuum chamber. In this kind of experiments, the temperatures were registered with a thermocouple placed above the oil (1-2 cm) and an other one inserted in a small hole bored in the wall of the recipient. The volume is not the main significant parameter in the case of outgassing (Sicre, 1995). The free surface of the oil sample mainly determines the outgassing flow. So, we reproduced for this cupel both conditions of volume and surface of a retainer by manufacturing a flat cylinder with the appropriate dimensions.

The same temperature cycle was applied in this case and in the experiments with the retainers.

2.3 Comments on the global pressure

All the results concerning the maximum pressure in the

chamber, the maximum partial pressure of the main element detected, and the evolution between the two tests are summed-up in table 2. The vacuum chamber and the various equipment it contained constituted a large trap for the water vapor. Thus, during the first outgassing tests with the empty chamber, the maximal pressure raised up to 20 times the initial pressure, due to the release of residual water vapor from the chamber walls (table 2). However, by repeating a similar test after the whole series of tests with the retainers, a lower initial pressure before test and a lower maximal total pressure were obtained. This proves that the chamber has been baked during the experiments, at least locally. It is noteworthy that in the second case, the main species detected was the mass 44 (CO₂) and no more 18 (H₂O). Nevertheless in the following test with the metallic cupel this time, it was the water peak which prevailed again. Between these two tests, the initial pressure was almost similar but the maximum has been reduced by a factor 4. This means that not only the metallic cupel didn't outgass but also that the multiplication of the temperature cycles lowered the content of water vapor pressure in the chamber. This was made possible due to the permanent positive pressure difference established in the chamber by an nitrogen flow after each aperture and during manipulation phases.

Name	Test No	Pressure before test(Pa)	Maximal total Pressure (Pa)	Increase by	Main Element / maximal partial pressure (relative intensity)
Vacuum chamber only	1	2.3 10 ⁻⁵	5.3 10 ⁻⁴	21	H ₂ O (18) / 3 10 ⁻⁹
Polyimide retainer	1	3.8 10 ⁻⁵	5.6 10 ⁻⁴	15	ClC ₂ H (60) / 1.5 10 ⁻⁸
	2	2.3 10 ⁻⁵	5.7 10 ⁻⁵	2.5	ClC ₂ H (60) / 1.1 10 ⁻⁹
Polyimide + SHF	1	3.5 10 ⁻⁵	1.9 10 ⁻³	55	H ₂ O (18) / 7.0 10 ⁻⁸
	2	3.2 10 ⁻⁵	5.7 10 ⁻⁴	18	H ₂ O (18) / 1.0 10 ⁻⁸
Polyimide + PFPE Z	1	4.5 10 ⁻⁵	4.0 10 ⁻³	89	H ₂ O (18) / 1.2 10 ⁻⁷
	2	2.6 10 ⁻⁵	2.6 10 ⁻⁴	10	H ₂ O (18) / 4.5 10 ⁻⁹
Polyimide + PFPE D	1	4.9 10 ⁻⁵	3.2 10 ⁻³	65	H ₂ O (18) / 6.0 10 ⁻⁸
	2	1.7 10 ⁻⁵	2.0 10 ⁻⁴	12	H ₂ O (18) / 4.0 10 ⁻⁹
Polyimide + PFPE L	1	5.0 10 ⁻⁵	1.2 10 ⁻³	24	H ₂ O (18) / 1.6 10 ⁻⁸
	2	1.6 10 ⁻⁵	2.2 10 ⁻⁴	14	H ₂ O (18) / 2.6 10 ⁻⁹
Vacuum chamber only	1	0.8 10 ⁻⁵	6.7 10 ⁻⁵	8.4	CO ₂ (44) / 1.6 10 ⁻⁹
Empty metallic cupel	1	0.85 10 ⁻⁵	1.85 10 ⁻⁵	2.2	H ₂ O / 4.8 10 ⁻¹⁰
SHF oil in the cupel	1	2.2 10 ⁻⁵	4.4 10 ⁻⁵	2	H ₂ O (18) / 8.9 10 ⁻¹⁰
	2	1.4 10 ⁻⁵	2.4 10 ⁻⁵	1.7	H ₂ O (18) / 7.3 10 ⁻¹⁰
PFPE Z oil in the cupel	1	2.2 10 ⁻⁵	7.0 10 ⁻⁵	3.2	H ₂ O (18) / 3.3 10 ⁻⁹
	2	1.4 10 ⁻⁵	2.8 10 ⁻⁵	2	H ₂ O (18) / 4.8 10 ⁻¹⁰
PFPE D oil in the cupel	1	1.8 10 ⁻⁵	12.2 10 ⁻⁵	6.8	H ₂ O (18) / 7.1 10 ⁻⁹
	2	1.6 10 ⁻⁵	2.1 10 ⁻⁵	1.3	H ₂ O (18) / 5.4 10 ⁻¹⁰
PFPE L oil in the cupel	1	1.4 10 ⁻⁵	4.1 10 ⁻⁵	2.9	C ₂ F ₅ (118) / 1.1 10 ⁻⁹
	2	1.4 10 ⁻⁵	2.0 10 ⁻⁵	1.4	H ₂ O (18) / 5.6 10 ⁻¹⁰

Table 2 : Typical pressures during outgassing experiments

2.3.1 Retainers

Heating up to 140°C (thermocouple B) has constantly led to a considerable elevation of the pressure in the first series of tests. The maximum pressure in the chamber rose of two orders of magnitude for the first test and one for the second. One notable exception was the case of the non-impregnated retainer, for which the heating has brought about an increase of pressure by more than 15 times during the first test but only by 2-3 times in the second test : the outgassing of the retainer was easier without oil. The main species detected were also different in case of impregnated or non-impregnated retainers. For the first ones, the main mass number detected was 18 (water). For the bare polyimide, the main outgassing elements detected were 60, 95, 97, 49, 35 and then only 18 (figure 1). These peaks didn't come from polyimide itself. In fact, it seems that these peaks corresponded more or less to dichloroethylene (60, 95, 97) and chlorine (35). This reveals that the solvent (trichloroethylene) used during the cleaning process was still remaining in the retainer pores at the moment of the test. These peaks were also detected in the case of impregnated retainers even if with the supply of hydrogen atoms of the SHF oil, the peaks 95 and 97 became 94 and 96. The solvent peaks remained weak during the first series of test and vanished in the second one. Moreover they have never been the main peaks detected for the impregnated retainers. Thus, it seems that it was more the phenomena occurring at the interface oil-retainer which might explain the massive outgassing during the first exposition to vacuum. Some air might have been trapped in the porous material during the impregnation phase taking place in primary vacuum and air. Under high vacuum, this air escaped, leading to the formation of bubbles at the surface of the impregnated retainers. These bubbles were visible to the naked eye. As a consequence of this massive bubbling and its subsequent pumping, the maximal vapor pressure decreased from one order of magnitude during the second test compared to the first one.

2.3.2 Tests with a metallic cupel

The tests in the metallic cupel confirmed the conclusions on the total pressure i.e. the interface oil-retainer is of major concern to explain the high outgassing rates. Between the first and the second test, the drop of the total pressure varied from a factor of less than 2 (SHF oil) to 6 (PFPE D oil) and no more from 3 (SHF oil) to 16 (PFPE D oil) like in the case with the retainers. The interface oil-cupel offered fewer possibilities to trap bubbles. In the series of tests with the metallic cupel, the main species detected during the tests was also H₂O, except for the notable exception of the first test with the PFPE L, for which the peak 118 (C₂F₅ compound) was the highest. As a possible satisfactory explanation, we formulate the hypothesis that a droplet of oil could have fallen on the heater ribbon, and that an over-heating could have led to the release of the fluorinated compound C₂F₅ (N=119). The test before this one was conducted with SHF oil, so it is improbable that the pump could have released this compound. This hypothesis would also explain the

detection of the peak (N=69) in the same test. The oil on the ribbon may have been cracked by an intensive heating, releasing some elements of decomposition which didn't appear for the other impregnated retainers.

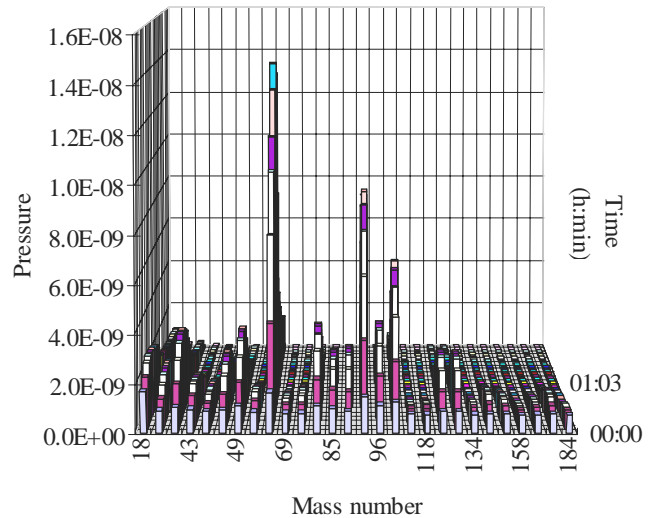


Figure 1 : Polyimide retainer outgassing spectra.

2.4. Oil by oil characterization

Twenty eight elements with detectable levels have been selected to follow the evolution of the lubricants outgassing performances. Ten of these peaks appeared only when the lubricants were introduced in the chamber : they contain the signature of the lubricants. Obtaining such a signature in the case of an impregnated retainer is not immediate as the outgassing peaks are masked by the main peak (usually H₂O), which level can be up to two orders of magnitude greater than the oil ones, and also by the chlorine by-products peaks. The technique adopted to reveal the oil peaks has consisted in subtracting the bare retainer spectrum from the spectrum of the impregnated retainer. By doing so, the significant peaks appeared. The results for each oil are summarized in table 3.

OIL	PEAKS
SHF	57, 111, 158, 184
PFPE L	47-49 (CFO), 83-85 (CF ₃ O)
PFPE Z	47-49, 83-85, 118 (C ₂ F ₅), 134 (CF ₂ OCF ₃)
PFPE D	47-49, 83-85, 118, 168 (C ₃ F ₇)

Table 3 : Significant peaks detected for each oil

The tests with the metallic cupel have furnished a good confirmation of the tests with the impregnated retainers. The peaks identified as coming from the oils were found again.

2.4.1 SHF oil signature

The synthetic hydrocarbon oil was probably the easiest to characterize with peaks of mass number 57, 158, 184, and in a less measure 111, appearing on the spectra shown in figure 2. These peaks may correspond to some hydrocarbon elements. 57 may characterize the alkane C₄H₁₀ (mass 58), may be in an unsaturated version :

C₄H₈. By a similar way, it is possible to imagine other hydrocarbon compounds which would have the masses detected, but when the molecule structure is examined, no cleavage appears obvious.

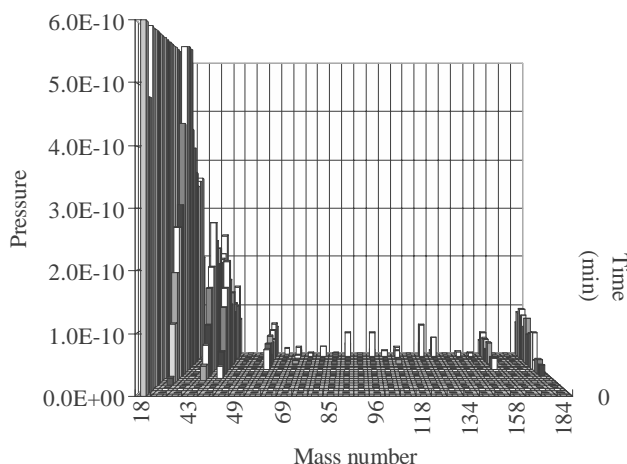


Figure 2 : Spectrum of a SHF-impregnated retainer.

2.4.2 Perfluoropolyether oils signature

Perfluoropolyether oils were difficult to differentiate through their outgass products. However, PFPE Z was slightly different from the two others. Gaseous products of mass numbers 118 and 134 formed peaks that were missing for the two other oils. They probably revealed the presence of CF₂OCF₃ (N=135) and C₂F₅ (N=119) compounds. As for the two other lubricants, peaks 83-85 (CF₃O) and 47-49 (CFO) were also detected. These are gaseous products which have been usually observed during tribological tests with these oils (Mori and Morales, 1989). We can notice that the peak of mass number 69 (CF₃), which usually appears during catalytic degradation, was not detected here except for the case already discussed.

3. BALL BEARING TESTS

3.1 General description

Tested bearings were angular contact ball bearings with a bore size of 20 mm (7204 type, 10 balls). They were made of 440C stainless steel and lubricated by an impregnated retainers containing one of the lubricants previously described. The figure 3 shows a schematic view of the ball bearing tester.

A pair of bearings were mounted on a shaft which was driven by a motor through a magnetic coupling. A thrust load was imposed by a coil spring. Due to the friction torque, a small angle shift occurred between the drive shaft and the driven shaft. This angle measured by photo-sensors allows the determination of the friction torque. The vacuum chamber was evacuated using an ion-sputter pump. Pressure at the beginning of the tests is less than 10⁻⁶ Torr. The rotational speed was 2000 rpm, the load was 200 N which corresponded to a contact pressure of 0.71 and 0.76 GPa for the outer races and the inner races respectively.

Two tests have been conducted for PFPE D, with either wiped retainer (seemed dry to naked eye) or non-wiped

(wet to naked eye). Aside from this case, the retainer with SHF oil was also initially wet. The criterion to stop a test was a high torque value.

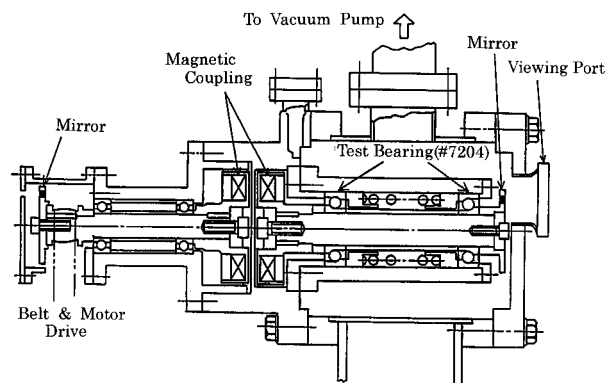


Figure 3: UHV Ball bearing Tester

3.2 Life time results

Oils displayed very different performances in this test from one oil to the other. The results are summed-up in the table 4.

Impregnating oil	Duration of the test
PFPE L	7848 hr
PFPE D	916 hr
PFPE D (wet)	1051 hr
PFPE Z	245 hr
SHF (wet)	10099 hr*

* : stopped before failure

Table 4: Lifetime of the different bearings

With more than 10000 hours of running time (test intentionally stopped after this milestone was reached even if no problem was noticed) the SHF oil outmatched the PFPE oils performances which were ranging from a poor lifetime of 245 hours (PFPE Z) to 7848 hours (PFPE L). An important quantity of black debris was found for each of the PFPE oil-lubricated bearing in a post test examination. In contrast to this observation, very little wear debris was found for the SHF oil-lubricated bearing.

Previous tests performed in the same operating conditions with a bearing containing a PTFE composite retainer and a MoS₂ sputtered film on balls and races, were stopped after about 1500 hours : SHF and PFPE L lubricated bearings had a much longer life than the solid lubricated ones, yet no precaution (shield, anti-creep coating) to retain the oil within the bearing was taken. This might have led to a shortage of oil supply and thus a shorter life. We indeed observed some oil droplets on the housing surface after the tests.

3.3 Outgassing evolution

Outgassing data have been recorded during these tests on the basis of one acquisition per week. For each bearing, the main species detected at the beginning of the test is water (18). No peak corresponding to mass numbers 60, 95, 97 or 35 (chlorine by-products) was detected in these experiments for which the pumping

process started several days before the running period.

3.3.1 SHF oil

In the case of the SHF oil, water (18) has remained the main peak during the whole test and has masked almost each of the twenty seven other investigated peaks. The principal secondary peaks were 44 and 28 (CO_2 , CO) and then 57, which was found in the previous tests to be the main gaseous product emitted by the SHF oil (figure 4).

During the initial pumping, the level decreased slowly until the test (rotation of the bearings) was started after about 800 hours. At that time, a burst of outgassing species was detected, and especially water for which the peak level reached a level ten times bigger than before operation. After this start, a second stage which lasted around 1000 hours characterized itself by a high outgassing rate. Following this period, the level of the outgassed species was roughly stabilized until the test was stopped.

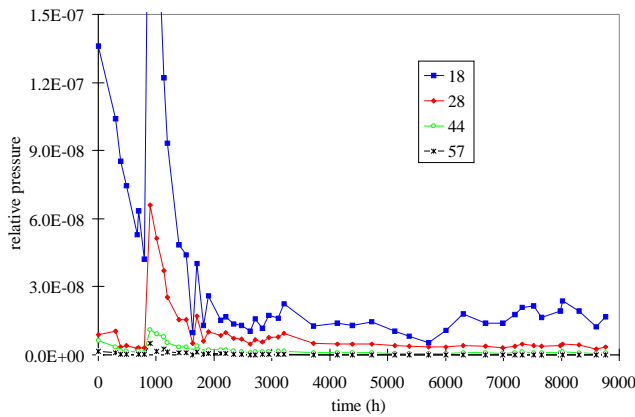


Figure 4 : Evolution of the main outgassing peaks.

The evolution of the total pressure is displayed in the figure 5 for which the time is referenced from the moment the bearings were rotated. The torque, the temperature have remained quite stable all the test long.

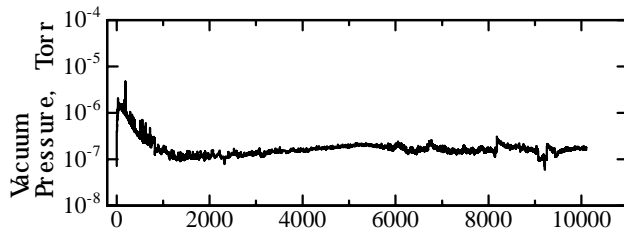


Figure 5 : Evolution of the pressure for a SHF oil lubricated ball bearing.

3.3.2 PFPE oils

In the case of the PFPE oils, the peaks appearing due to the lubricant degradation became predominant when the failure was approaching. These peaks were slightly different in case of PFPE Z or L and PFPE D.

Despite a shortage of data for PFPE Z, peaks 47 (CFO), 69 (CF_3), 168-169 (C_3F_7), and 118-119 (C_2F_5) revealed, 60 hours before the end of the test, a beginning of a

degradation process.

In the PFPE L case, the peak 69 (CF_3) was the main outgassed species detected with 47 (CFO). The $\text{C}_2\text{F}_3\text{O}$ peak (97) was also important. The evolution of the outgassing spectra was very perturbed by many outgassing bursts which affected the total pressure. The torque was also very noisy, expressing the fact that the oil degradation is a tribo-chemical process. The temperature increased slowly from 33°C to 39°C at the end of the test.

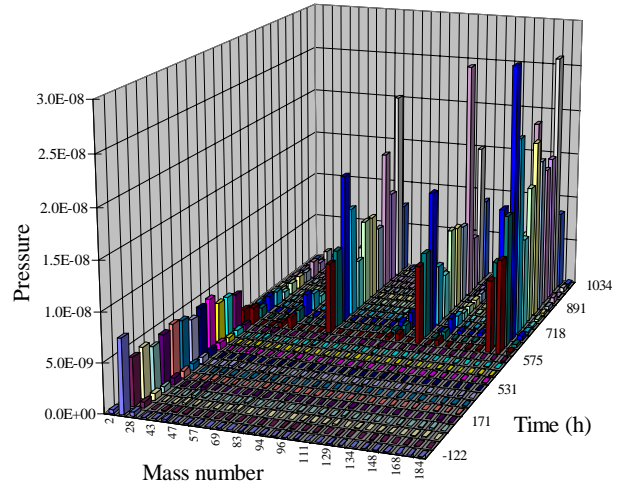


Figure 6 : Outgassing spectra of PFPE D bearing test.

For PFPE D, the situation was almost the same for wet and dry retainers. The figure 6 displays the evolution for the test with the wet retainer. As long as the running was normal, almost only the peak of H_2O (18) was significant, but after 600 hours, the oil began to be deteriorated. Peaks 168-169 (C_3F_7), 118-119 (C_2F_5) and 69 (CF_3) became clearly predominant. At the same time, the total vacuum pressure rose as well as the torque which became high and erratic (figure 7).

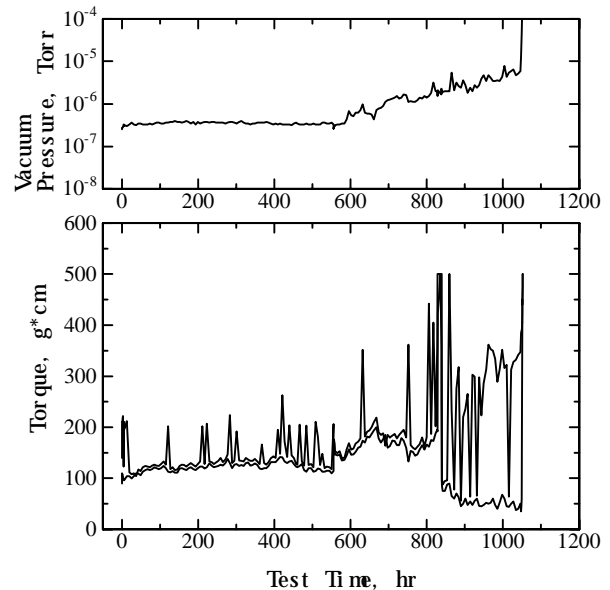


Figure 7 : Torque and pressure for PFPE D bearing test (wet retainer).

3.4. Post-test chemical analysis

SHF oil - Gas chromatography didn't reveal any degradation product in the oil after the test. This was confirmed by FTIR as no new absorption line was detected. The absorption degree was found to be lower for the test oil than for a new one.

The main elements detected by XPS on the balls surface were C (carbon) and O (oxygen) and then Fe (iron) and Cr (chromium). O was found in a chromium oxide Cr_2O_3 whereas very few iron oxide was detected. The carbon had two sources, on the surface, the C of the hydrocarbon C-H liaison and deeper, the C of the steel cementite.

PFPE oils - Wear debris analyzed by SEM+EPMA were found to be constituted of Fe and Cr i.e. materials that have been detached from the bearing surface.

By observing the balls or races surfaces with XPS, a small thickness of oil was firstly detected. Under this oil, some metal fluoride was found by monitoring the intensity of the fluorine peak. According to the Fe peak evolution, this fluoride could be FeF_3 also called Lewis acid. A small amount of chromium oxide was also detectable. It is noteworthy that we didn't detect the same by-products in the oil taken, on one hand, on the balls or the races surfaces and on the other hand, in the oil taken at the surface of the retainer, except for the only case of the wet retainer of PFPE D. Actually, gas chromatography (sensitivity better than 1ppm) of PFPE D and PFPE Z samples taken on retainers revealed several compounds with a boiling point in the range 100-180 °C and 100-200 °C respectively. The mass chromatography performed on the same samples unveiled peaks 118, 146, 168 for PFPE D and 119, 147, 169 for PFPE Z, peaks that have already been detected during the tests with the exception of the peak 146 (C_3F_5O). One of the possible explanations for the absence of these small fluorinated compounds on the balls and races (yet the place where the initial decomposition should take place) could be this one : we may have, in accordance with the hypothesis described by Carré 1985-1986, an interaction between the iron of the steel and the PFPE to form the Lewis acid, followed by the catalytic cleavage of the PFPE oil by FeF_3 which would result in the formation of low molecular weight compounds but we probably had also a third phase in which these compounds kept on reacting with the metallic fluoride or metallic surface until their total disappearance. In case of the oil taken from the retainer, this phase couldn't occur due to the absence of metallic contacts. We don't have further evidence to support this hypothesis. We may also consider a different evaporation rate depending of the substrate nature, porous like the polyimide retainer or less absorbent like the balls and races surface.

3.5 Geometrical analysis

After wiping the debris and cleaning the bearings, the deviation from the circular form or roundness (table 5), the rotation accuracy and the wear amount in a cross section profile have been measured.

In accordance with its behavior in the lifetime tests, the SHF lubricated bearings were the less deteriorated. The

rolling elements and the rings were still in a condition that would have allowed a continuation of the tests according to the rotation accuracy criteria adopted by the bearing maker. No measurable amount of wear was found on a cross section profile in the rolling tracks of the inner and outer races.

On the other hand, all the bearings lubricated by one of the PFPE oils were damaged. Some bearings (specially N° 4, 5 and 8) have encountered a severe deterioration of the rotation accuracy (incompatible with the acceptance criteria); the wear amount was also very important in these three bearings and was not negligible for the three others (N° 3, 6 and 7).

Lubricant	Bearing	Inner Race	Outer Race	Balls
SHF	1	0.30	0.40	0.078
SHF	2	0.25	0.65	0.065
PFPE L	3	0.50	0.89	0.310
PFPE L	4	1.04	1.34	0.709
PFPE Z	5	2.26	4.50	1.340
PFPE Z	6	0.86	1.39	0.825
PFPE D	7	0.57	1.56	0.283
PFPE D	8	2.22	1.93	0.145

Table 5: Roundness of the bearings after tests (μm).

CONCLUSIONS

-Ball bearings lifetime experiments have revealed an excellent behavior of a SHF oil, much better than three PFPE oils, but also than MoS_2 lubricated bearings.

-The quantity of outgassed products during the ball bearing tests remained low as long as no degradation occurred (PFPE oils case only). Then, a much larger amount of gas was detected.

-The nature of the small weight compounds released by the oils during a normal running of the ball bearings has been identified more accurately in short term experiments with a mass spectrometer.

-As a possible continuation of this study, further testing should be performed to evaluate the effects of the UV rays and the atomic oxygen on the aging of the compounds identified here.

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