THE SYNERGISTIC EFFECTS OF MOS_2 AND LIQUID LUBRICATION

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ABSTRACT

We present an overview of a three-stage program on the potential for hybrid lubrication of MoS_2 and PFPE fluids (Fomblin Z25 & Braycote 601EF) performed at the European Space Tribology Laboratory (ESTL).

Tests were performed using a spiral orbit tribometer (SOT) and a pin-on-disc tribometer (POD), demonstrating encouraging results. Hybrid lubrication allows for extended periods of in-air running of MoS_2 with no detrimental effect to the subsequent in-vacuum lifetime. In addition, hybrid lubrication was shown to be synergistic, with the lifetime of the hybrid fluid/MoS_2 lubrication extended in comparison to the individual constituents, with no detriment to the friction

1. BACKGROUND

Sputtered coatings of molybdenum disulphide (MoS_2) yield very low friction and relatively long lives when operated under high vacuum conditions [1]. In addition, these tribological properties are maintained over a wide range of temperatures [2, 3]. As such MoS_2 coatings are used routinely to lubricate spacecraft mechanisms. However, when operated in laboratory air, the coatings adsorb water molecules and this affects their shear properties which in turn causes the friction to increase (by up to an order of magnitude) [1].

Furthermore the coating oxidises and, as a result, wears at a much more rapid pace than would be the case in vacuum. Thus operation in air severely reduces the subsequent in-vacuum life of the coatings [4, 5]. This reduction in life is shown to be dependent upon running duration in-air, with even short running periods producing dramatic reductions in subsequent in-vacuum life (Fig. 1).

Friction coefficient values can return to their low vacuum-running values, but often only after a period of high friction. The upper value and duration of this increased friction upon subsequent vacuum running has been shown to be related to the amount of in-air running [6], as well as if the MoS_2 is exposed to in-air heating [7].

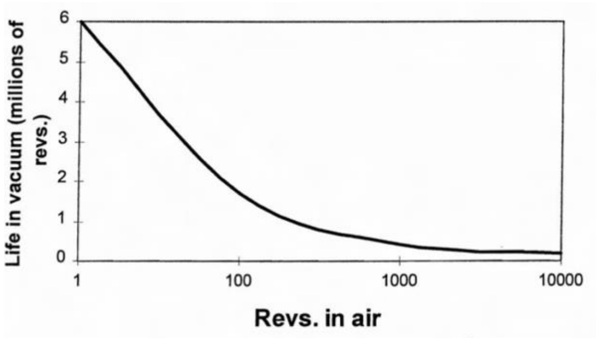

Figure 1. Low-torque life of MoS_2 lubricated ED20 bearings in vacuum as a function of the amount of in-air operation prior to vacuum tests [4]

This behaviour nearly always precludes the testing of components and mechanisms in air when lubricated by MoS_2. The advice proffered by the Space Tribology Handbook is 'avoid air testing at all costs' [4]. This restriction is hugely frustrating for mechanism engineers who often want to carry out testing 'on the bench' in a normal clean-room environment.

1.1. Hybrid Lubrication

One suggestion is that a form of hybrid lubrication may circumvent this problem, through the application of a small quantity of oil to a component lubricated in the usual way with sputtered MoS_2. The concept requires that the amount of oil be just sufficient to provide fluid lubrication for the required period of in-air testing; subsequently when the component is operated in vacuum any oil remaining should be lost (either by evaporation or by tribo-chemical wear processes), thus leaving the MoS_2 coating to provide the required low-friction, solid lubrication.

To test this theory a multi-stage program was performed at ESTL using both spiral-orbit and pin-on-disc tribometry.

2. APPARATUS

2.1. Spiral Orbit Tribometer

The Spiral Orbit Tribometer (SOT) is essentially a thrust bearing, with an individual ball held between two interchangeable flat plates, located within a vacuum

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chamber. A load is applied to the top plate via a spring-loaded linear translator. The lower plate rotates via a motor located outside the chamber, causing the ball to move in a spiral path with a radius ~21mm.

This configuration causes the ball to spiral outwards, and a fixed guide plate is positioned to keep the ball within the flat plates and to produce a repeatable orbit. A force transducer behind the guide plate measures the force exerted by the ball onto the guide plate. From this a friction coefficient value is found, once per orbit. The SOT is controlled using a LabVIEW-based data acquisition program.

The arrangement of the SOT allows the ball to experience rolling, sliding and pivoting – all motions experienced by a ball in an angular contact bearing. This allows for a more representative testing of a lubricant than conventional pin-on-disc testing, which only recreates sliding motion.

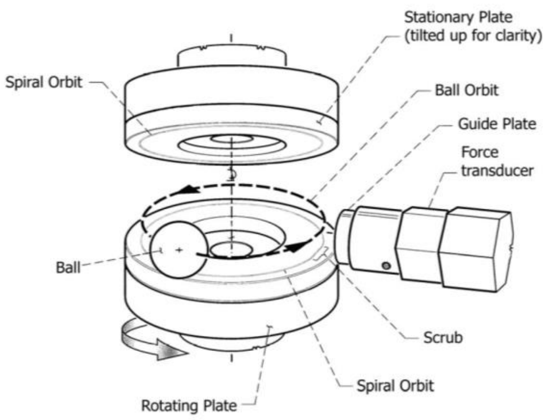

Figure 2. Internal arrangement of SOT

Lifetimes are given in units of oribts/micrograms, allowing for normalization of results should varying lubricant amounts be used.

2.2. Pin-On-Disc Tribometer

The pin-on-disc tribometer (POD) is shown below, and consists of a pin mounted on a balanced arm, and loaded against the disc by a deadweight. The disc is rotated by a motor positioned outside the vacuum chamber. The frictional force is measured via the deflection of the arm, and recorded using a PC-based data acquisition system. Calibration was checked using a pulley system to apply known loads to the tribometer arm.

Figure 3. Vacuum pin-on-disc tribometer

In addition, an AccuQuad Kurt J. Lesker residual gas analyser (RGA) was attached to the POD to allow identification of gaseous species within the chamber during tests. The RGA was operated only up to 140AMU with a resolution of 0.1AMU. The RGA was operated in analogue scan mode, with a full scan recorded every ~57 seconds. Prior to each test the RGA was degassed, re-calibrated, and tuned to maximise the signal-to-noise of each test.

2.3. Samples

SOT flat samples were manufactured from non-passivated 440C steel, polished to a roughness of R_a<0.05 micron. Balls used were manufactured from 52100 steel, of 7.14mm diameter.

POD test samples were standard test washers type WS 81102, manufactured of SAE 52100 steel. Discs are annular shaped with an OD of 28 mm and ID of 15mm. 52100 steel balls of 7.14mm diameter were used as pins.

Prior to lubrication, all balls and plates were solvent cleaned using Lenium ES solvent in accordance with standard ESTL procedure.

2.4. Lubrication procedure

Solid lubrication (MoS_2) on SOT balls and POD discs was provided using ESTL's sputtered coating rig. Coatings were applied with a target thickness of 1 micron for discs and 0.5 micron for balls, this being the typical thickness of MoS_2 in an application.

SOT fluid lubrication was achieved through the preparation of a solution of lubricant diluted in an appropriate solvent to give a known concentration. This solution was applied directly to a rotating ball. The solvent was allowed to evaporate from the ball's surface, leaving the desired lubricant amount. It was confirmed through POD testing that the selected solvents did not adversely affect the adhesion of the MoS_2 coatings.

This method of lubrication allows for the application of very small lubricant amounts, typically 50µg. This minuscule amount of oil allows for reduced test times, and ensures all tests take place under boundary conditions.

For POD samples the grease lubrication was applied evenly to the surface of the discs using a class 100 wipe. The total lubricant amount was measured using a 6-point microbalance before and after lubrication. It is acknowledged that this method does not provide an accurate measurement of the amount of lubricant available to the contact zone, and that a significant proportion of the applied grease will be distributed away from the running track, and will play no part in the tribology.

3. PHASE ONE – FEASABILITY DEMONSTRATION

3.1. Phase one test plan

An initial feasibility demonstration was performed on the SOT. The procedure for these tests is as follows;

a) Application of oil to a sputtered MoS_2 lubricated ball

b) Installation of hybrid lubricated ball in the SOT

c) Running of ball for pre-determined duration in air ($MoS_{2\ Air}$), 2.25GPa peak, 100 RPM, 23deg.C

d) Without removing the ball, evacuate test chamber to 1×10^{-6} mbar

e) Once pressure is achieved, immediately begin motion under the same loading and speed conditions

f) Run test until lubricant failure ($\mu \geq 0.3$)

$MoS_{2\ Air}$ was defined as the lifetime of the MoS_2 assessed in air on the SOT under identical load and speed conditions.

For this phase three oils were selected: one space oil expected to degrade during in-vacuum rolling, and two non-space oils expected to evaporate during evacuation of the chamber.

Table 1. Selected fluid lubricants for phase one – feasibility demonstration

Fluid	**Fomblin Z25**	**Synalox 100-D020**	**Millube-5**
Description	PFPE space oil	Terrestrial polyether	Terrestrial hydrocarbon
VP @ 20°C	1.6×10^{-13} mbar	< 1 mbar	1 – 0.01 mbar

3.2. Phase one results

For Fomblin Z25 running in air the friction coefficient was maintained at 0.1 for the majority of the test duration, with no indication of failure of the coating (Fig. 4). This result tentatively suggests that the Z25 oil was providing the lubrication during in-air running, and was protecting the MoS_2. It is also noted that, although the lifetime of Fomblin Z25 is extended in the presence of moisture [8], this extension alone is not significant enough to account for the observed performance.

When restarting the test under vacuum, we see high frictional noise. However this high noise is typical for MoS_2 tested under vacuum in the SOT [9], and is caused by false high friction coefficient values being outputted by the SOT logging software for occasional orbits. These false high values are a result of the amplitude of the ‘ringing’ in the guide plate force profile (the method in which the SOT measures friction) becoming significant in relation to the frictional force.

From the raw SOT data a true value of friction can be found as 0.02, identical to the value for MoS_2 alone in vacuum [9]. Given that Z25 gives a friction coefficient of 0.1 under vacuum, we can say with some confidence that MoS_2 was providing lubrication at this time. This low friction was maintained for ~60,000 orbits before rapid failure

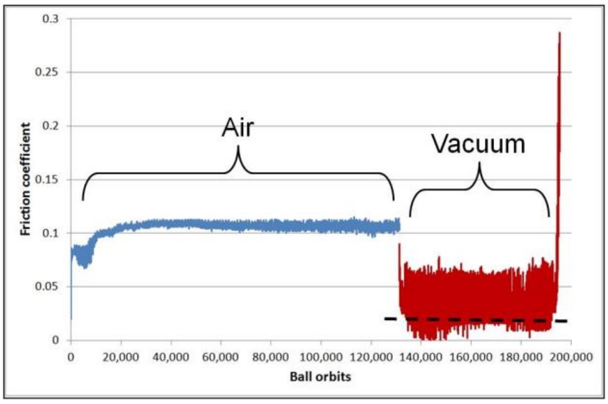

Figure 4. Z25/MoS_2 rolling in air (blue) and vacuum (red). Dashed line shows the true vacuum friction

Post-test inspection of the Z25/MoS_2 samples showed evidence of degradation of both lubricants, and evidence that mixing of the solid and liquid had occurred. In comparison to MoS_2 alone very little MoS_2 debris was found thrown away from the ball track. Instead the ball tracks appeared similar to tests on the oil alone, with dark degraded material within the ball track itself, and lighter material pushed to the side. This lighter material appeared a dull grey in colour, and is suspected to consist of MoS_2 particles captured within the oil. No evidence of oxidised MoS_2, a product of running this lubricant in air, was observed.

These observations tentatively lead us to the following explanation. In air the contact is initially lubricated by the Z25 oil, with MoS_2 degradation occurring in the later part of the test. Under vacuum the MoS_2 provides the lubrication, apparently immediately, but due to the wear/oxidation experienced during the air test its lifetime is somewhat shorter than for MoS_2 alone. As the MoS_2 is lost from the ball it is trapped by the remaining wet oil, and pushed to the side of the ball track.

For the Synalox oil in air, a friction coefficient of 0.1 was maintained for ~100,000 orbits. Beyond this the friction coefficient began to rise, suggesting that the MoS_2 was nearing the end of life (Fig. 5).

On restart under vacuum the tests ran for an average of ~14,000 orbits. This is significantly reduced in comparison to the Fomblin Z25, and indicates that more significant degradation of the MoS_2 may have taken place in air prior to commencing the vacuum test.

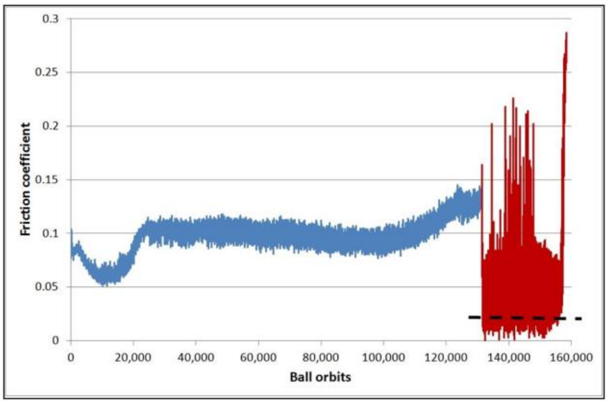

Figure 5. Synalox/MoS_2 rolling in air (blue) and vacuum (red).

Post-test inspection of the Synalox/MoS_2 samples were similar to that of the Z25/MoS_2, with the exception of a small amount of oxidised MoS_2 debris, again suggesting that, at least to some degree, MoS_2 degradation had taken place in air. A larger amount of loose MoS_2 was also observed, perhaps indicating there was less oil available at the time of MoS_2 degradation under vacuum to 'capture' the loose MoS_2 particles.

It is not clear exactly how the MoS_2/Synalox combination has acted to lubricate in these tests. Although the Synalox did prevent failure of the MoS_2 during the air test, it also appears that some degree of MoS_2 degradation did take place whilst in air, resulting in the reduced lifetimes when continuing the test under vacuum. Whether the Synalox reached the end of its lubricating lifetime in air, or if it was unable to protect the MoS_2 from wear is not clear from these results.

The Millube-5 oil produced similar results to the Synalox, with a similar increase in friction towards the end of the in-air running, but even shorter in-vacuum life.

The results of the three tested oil/MoS_2 combinations are summarised in Tab. 2 below.

Table 2. Summary of phase one results

Oil lubricant	**Passed $MoS_{2\ Air}$?**	**Subsequent in-vacuum life**
Fomblin Z25	Yes	60,000
Synalox 100-D020	Yes, with μ increase	14,000
Millube-5	Yes, with μ increase	1,200

Given its encouraging performance, Fomblin Z25 was therefore selected for phase two of this activity.

4. PHASE TWO – DETAILED STUDY

4.1. Phase two test plan

Phase two of testing was performed in a similar manner to phase one, with the following alterations;

a) All testing on Fomblin Z25 oil

b) Tests performed at three contact stresses (3.00GPa, 2.25GPa & 1.50GPa peak)

c) Tests performed with varying durations of in-air running prior to vacuum

d) In-air running durations defined as percentages of in-vacuum MoS_2 life

In-air running durations are given in Tab. 3 below, with L_x being the in-vacuum MoS_2 life at a given contact stress S_X.

Table 3. Test matrix of in-air running for phase two

Contact stress	**S_1 (3.00 GPa)**	**S_2 (2.25 GPa)**	**S_3 (1.50 GPa)**
In-air operation	$0.0005L_1$	$0.0005L_2$	$0.0005L_3$
	$0.005L_1$	$0.005L_2$	$0.005L_3$
	$0.05L_1$	$0.05L_2$	$0.05L_3$
	$0.5L_1$	$0.5L_2$	$0.5L_3$

Prior to hybrid lubrication testing, the lifetimes of MoS_2

under vacuum at the above contact stress was assessed. These results demonstrated an expected decrease in rolling lifetime with increasing contact stress. From these lifetimes the in-air durations required for the phase two hybrid testing can be calculated (see Tab. 5).

Table 4. Rolling lifetimes of MoS_2 in vacuum

Contact stress (GPa)		Life (orbits)	
3.00	S_1	165,000	L_1
2.25	S_2	312,000	L_2
1.50	S_3	773,000	L_3

4.2. Phase two results

The results of the in-air stage of phase two are shown below.

Table 5. In-air rolling results for in-air stage of phase two hybrid lubrication testing

ID	Stress	In-air operation		Pass/Fail
S1-A	3.00	$0.0005L_1$	83	Pass
S1-B	3.00	$0.005L_1$	826	Pass
S1-C	3.00	$0.05L_1$	8,256	Pass
S1-D	3.00	$0.5L_1$	82,562	Fail (81,433)
S2-A	2.25	$0.0005L_2$	156	Pass
S2-B	2.25	$0.005L_2$	1,557	Pass
S2-C	2.25	$0.05L_2$	15,584	Pass
S2-D	2.25	$0.5L_2$	155,840	Pass
S3-A	1.50	$0.0005L_3$	386	Pass
S3-B	1.50	$0.005L_3$	3,863	Pass
S3-C	1.50	$0.05L_3$	38,627	Pass
S3-D	1.50	$0.5L_3$	386,271	Pass

As can be seen, all tests with the exception of S1-D passed the in-air duration of running, with S1-D running for 98% of the required time. This allows us to state that the application of ~50µg Fomblin Z25 oil can allow balls lubricated with sputtered MoS_2 to run in-air for 50% of their in-vacuum lifetime without displaying evidence of failure.

Following in-air testing each sample was run until failure under vacuum conditions. It was decided that despite failing the in-air running duration, test S1-D would also be continued to vacuum. Results are shown below (Fig. 6).

From the vacuum results it is clear that, with the exception of S1-D and S2-D, extended lifetimes were observed for all tests. In all instances these extensions in life were greater than the lifetime of MoS_2 alone in vacuum (Tab. 4). This effect is more pronounced at lower contact stresses, as the lifetime of Fomblin Z25 oil is much more sensitive to loading than MoS_2 (Tab. 6) [9].

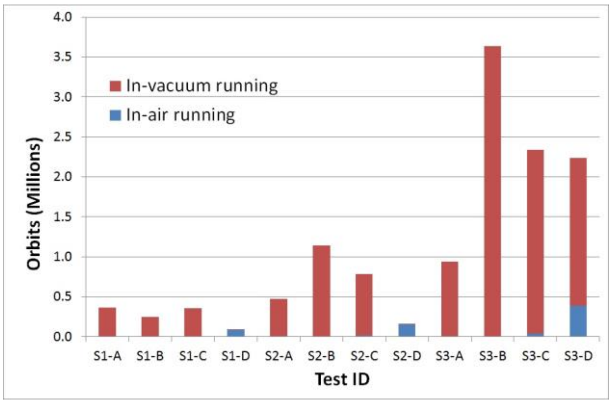

Figure 6. Rolling lifetime of phase two SOT tests

Table 6. Factor rolling lifetime increase from 3.00 -> 1.50GPa contact stress under vacuum on the SOT [9]

Lubricant	Lifetime increase
Fomblin Z25	32
Sputtered MoS_2	5

This may imply that, in the case of the S3 tests performed at 1.50GPa, the contribution of the Fomblin Z25 oil to the total life is proportionally greater than for higher contact stresses, resulting in lower MoS_2 wear during the in-air running phase of this test, and subsequently producing an extended in-vacuum lifetime.

Using the predictions of fluid lifetimes taken from [9], we can calculate the individual contributions from the fluid and solid components of these tests under vacuum (assuming no prior in-air running). Such calculations demonstrate that for all phase two tests (with the exception of S1-D and S2-D), the total 'hybrid' lubrication lifetime is longer than that of the individual lubricant constituents. That is to say there is a synergistic lubrication effect.

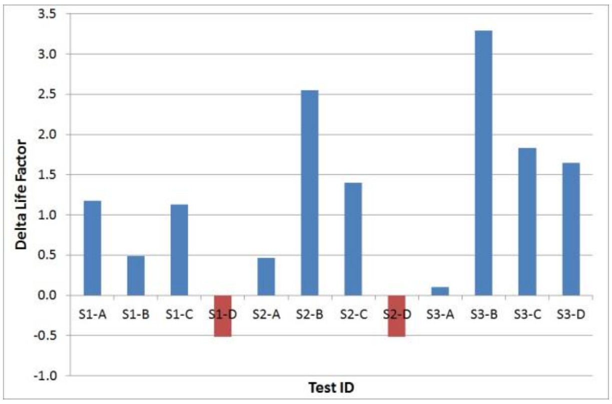

Figure 7. Delta-life extensions of 'hybrid' lubrication in comparison to predicted lifetime of the lubricant constituents

This extension can be summarised with the following statement.

Life of PFPE/MoS_2 lubrication > Life of PFPE lubrication + Life of MoS_2 lubrication

For all tests the steady-state friction coefficient during the in-vacuum stage of testing was significantly below 0.1, strikingly different to the profile shape observed during PFPE oil tests. This suggests that the MoS_2 was providing the lubrication during the in-vacuo stage of all hybrid tests throughout the extended life.

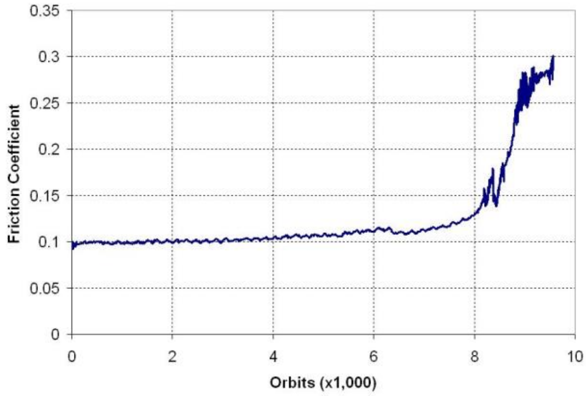

Figure 8. Example friction trace of Fomblin Z25 in vacuum [9]

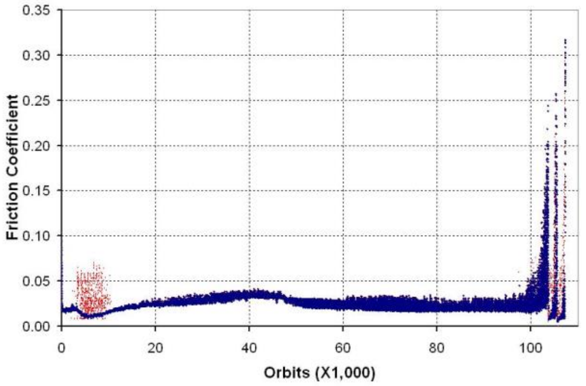

Figure 9. Example friction trace of sputtered MoS_2 in vacuum [9]

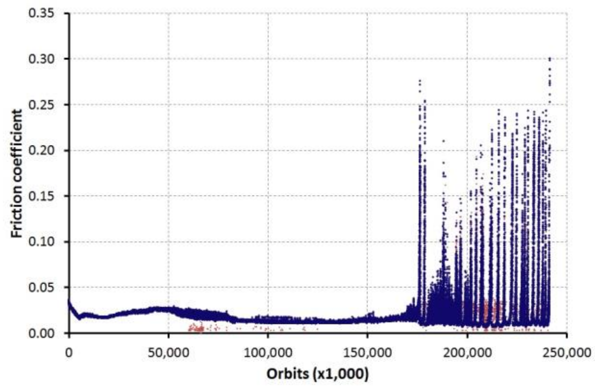

Figure 10. Friction trace from in-vacuo stage of S1-B, clearly showing similar behaviour to 'dry' MoS_2

5. PHASE THREE – POD STUDY

5.1. Phase three test plan

In parallel to the SOT test program, a series of POD tests were performed to verify the above results for Braycote 601EF grease in a sliding regime. Three families of POD tests were performed

a) Discs lubricated with Braycote 601EF grease only

b) Discs lubricated with sputtered MoS_2 only

c) Discs lubricated with both sputtered MoS_2 and Braycote 601EF

All tests were run under vacuum, 0.90 GPa peak contact stress, 0.6ms^{-1} sliding speed, 170°C.

5.2. Phase three results

Lifetime results show clear separation in the lifetimes of the lubricant combinations, with the Braycote/MoS_2 lifetimes again being significantly extended in comparison to the individual lubricant constituents (Fig. 11).

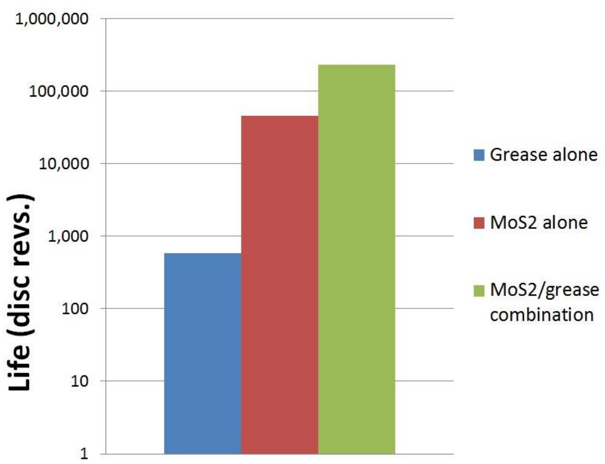

Figure 11. Mean lifetimes for Braycote 601EF and MoS_2, separately and in combination, during phase three of testing

Considering friction, the Braycote 601EF/MoS_2 tests produced marginally higher and noisier friction than the MoS_2 alone. This behaviour is suspected to be due to the effect of viscous losses of the lubricant, a notion supported by the observation of a reduction in sliding friction during a temporary rotation speed reduction performed during the tests.

A single dwell test on the hybrid lubrication was performed in which the motion was paused for 10-days at 100°C under vacuum. Following this dwell the temperature was raised to 170°C for the duration of the life test. No evidence of continued degradation of the grease, the so called auto-catalytic effect, was observed, and the full lifetime of test fell well within family for the Braycote601EF/MoS_2 tests.

In-situ monitoring of the volatile gas species within the chamber showed evidence of PFPE (i.e. grease) degradation during the entire duration of the Braycote 601EF/MoS_2 tests. This suggests no clear transition from grease-dominated lubrication to MoS_2-dominated lubrication, but rather that the lubrication is mixed throughout.

6. RESULTS AND DISCUSSION

It is clear from the above that the application of a small amount of fluid lubricant can act to reduce the detrimental effect of in-air running on the MoS_2, and provide subsequent long lifetime in vacuum. However it is not so clear why this extension is occurring.

One theory which may explain how the addition of a small volume of fluid oil would extend the lifetime of MoS_2 is that the fluid oil acts provides a hydrophobic surface to the MoS_2, resulting in a barrier to MoS_2 from moisture attack.

Optimal thin film MoS_2, including ESTL's MoS_2 coating are known to grow in a columnar structure with the Mo – S planes parallel to the substrate surface. This provides a small area of exposed Molybdenum at the sides of the columns which suffer from reactions with moisture present in the test environment. It is theorised that the fluid oil will fill the surface voids between the columns of MoS_2 and intermix with sliding MoS_2 during testing, offering continued protection through sliding wear.

6.1. An Alternative View-Point

During this paper the view has been taken that this extension in life of the hybrid lubrication is a result of the fluid protecting the MoS_2 from failure, and thus extending its lifetime. However these results can also be interpreted in a different fashion –that the MoS_2 layer is preventing the tribo-chemical degradation of the PFPE fluid.

It is well understood that PFPE lubricants degrade due to tribochemical reactions with iron within the substrate steel [10]. It is also true that the rate of this degradation is related to multiple variables, including load, temperature & environment [8, 9, 11]. It is also known that if the steel is coated in such a way to prevent contact with the fluid, then lubricant lifetimes are generally extended, for example as seen with TiC ball coatings [12].

It is proposed that a similar behaviour is occurring during these tests, with the MoS_2 layer acting to retard the degradation rate of the PFPE fluid.

Evidence to back up this idea is given by the RGA data from phase three, in which evidence of constant PFPE degradation was seen during the entire hybrid tests. As this sliding lifetime way exceeds the lifetime of the grease alone, we can state that the rate of PFPE tribo-chemical degradation was severely reduced, potentially by the presence of MoS_2. A similar behaviour may be occurring in phase two to also produce the extended lifetimes.

It is suggested that the observed extension in life seen for the hybrid lubrication cases is a combination of the two processes detailed above. In this way we can say that MoS_2 and liquid lubrication has the potential for synergistic behaviour.

Irrespective of the reasons, these results are encouraging, and suggest that hybrid lubrication is a viable and potential lubricant solution for vacuum applications. It is also worth stating that these extensions in life (for the SOT) were achieved using much smaller lubricant volumes than would be conventionally applied for fluid lubricated applications (~50µg).

Life of PFPE/MoS_2 lubrication > Life of PFPE lubrication + Life of MoS_2 lubrication

However it is of course recognised that many applications preclude the use of fluid lubrication, for reasons of contamination (for example optical applications). Therefore the above statement is true for non-contamination sensitive applications only.

7. CONCLUSIONS

The main conclusions of the work presented here are:

- Hybrid lubrication of MoS_2 is demonstrated as feasible.
- Hybrid lubrication allows for extended periods of in-air running with no detrimental effect to the subsequent lubricating properties of the $MoS_{2.}$
- Hybrid lubrication of Fomblin Z25/Braycote 601EF and MoS_2 can be synergistic, with the lifetime of the hybrid fluid/MoS_2 lubrication extended in comparison to the individual constituents, with no detriment to the friction.

7.1. Further Work

Future work is anticipated in this field, likely in the following areas;

- Further investigation of this effect when sliding.
- Demonstration of effect at component level (gears & bearings).

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