Hybrid Lubrication of PFPE Fluids and Sputtered MoS$_2$

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Abstract

We present an overview of the recent activities performed by the European Space Tribology Laboratory (ESTL) into the potential of hybrid lubrication of PFPE fluids (Fomblin Z25 & Braycote 601EF) and sputtered MoS$_2$. Test campaigns were performed using a spiral orbit tribometer (SOT), pin-on-disc tribometer (PoD), and at spur-gear level.

Results demonstrated mixed behavior of hybrid lubrication. In the best case the lifetime is extended beyond that predicted by the individual constituent lubricants, with no elevation in friction coefficient. In the worst case the application of a grease to the sputtered MoS$_2$ appears to inhibit the favorable tribological behavior of the solid lubricant film, reducing the lifetime and elevating the friction/torque.

The degree of success of hybrid lubrication appears to be related to the physical properties of the applied fluid lubricant (film thickness, viscosity), rather than the tribo-chemical lifetime. A model is proposed by which this behavior occurs.

Introduction

For a mechanism engineer lubricant selection often comes down to a trade-off between solid and fluid lubrication, with merits and disadvantages to both solutions. Solid lubricants are typically chosen in situations where temperature restraints preclude the use of fluid lubricants (due to evaporative losses or viscous torque increases), as well as applications for which contamination is a major consideration (e.g. involving optics). Fluid lubrication is typically selected for applications operating at high speeds over medium-to-long periods (high duty). Fluid lubricants also typically display lower torque noise and higher thermal conductance.

Solid Lubrication – Sputtered molybdenum disulphide

Molybdenum disulphide (MoS$_2$), deposited as a thin film via physical vapor deposition (PVD), is commonly used as a solid lubricant within high vacuum and spacecraft mechanism applications. Such films yield very low friction and relatively long lives when operated under high vacuum conditions [1]. 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 moist 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 oxidizes and, as a result, wears at a much more rapid pace than would be the case in vacuum. Thus, operation in moist 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 (Figure 1).

Following operation in air, friction coefficient values can return to their low vacuum-running values, but often only after a period of high friction. The magnitude and duration of this increased friction upon

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subsequent vacuum running has been shown to be related to the in-air dwell period and the extent of moist air running (Figure 2) [6], and is more severe if the sputtered MoS$_2$ is exposed to in-air heating [7].
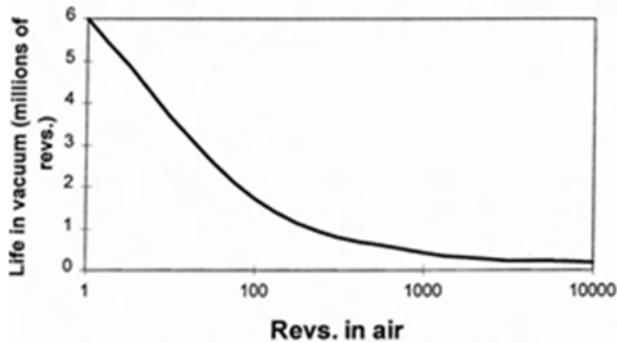
Figure 1. Low-torque life of MoS$_2$ lubricated ED20 bearings in vacuum as a function of in-air operation prior to vacuum testing [4]
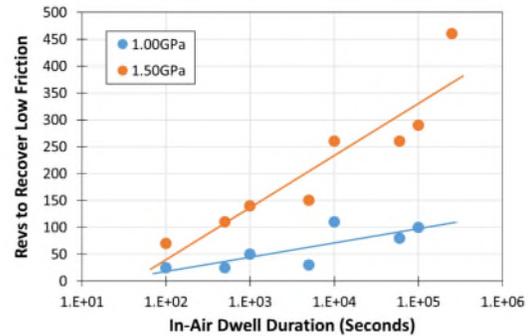
Figure 2. PoD revs required to achieve low friction performance of sputtered MoS$_2$ under vacuum following moist air dwell period [6]

This deleterious effect on subsequent lifetime under vacuum is of concern for space mechanisms applications, where demonstration of a successful deployment on ground is often demanded as part of the qualification program. Whilst the use of a protective dry nitrogen environment can theoretically be employed to protect the sputtered MoS$_2$ lubricated components during 'on ground' operation, this may have practical limitations especially at spacecraft level.

Numerous attempts have been made to improve the lubricating performance of sputtered MoS$_2$ films in moist air, including doped variants. However, this paper concentrates only on improving the performance of the existing film.

Fluid Lubrication – PFPE oils
Perfluoropolyether (PFPE) type fluids are well suited for applications in space due to their low vapor pressures, low pour points, resistance to radiation and atomic oxygen, good tribological properties, and being highly chemically inert [8]. Z-type PFPEs (such as Fomblin Z25, Brayco 815Z, and greases based upon these oils such as Braycote 601EF) are constructed from linear polymer chains and have been employed extensively as lubricants in spacecraft mechanisms for many decades [9]. However, under boundary conditions such lubricants are susceptible to chemical degradation, resulting in increased friction coefficients, material wear, and eventually component failure [10, 11]. This tribo-chemical degradation occurs primarily through the reaction between the polymer chains and chemically active sites in the substrate steel [12]. Nevertheless Z-type PFPE fluids are commonly used in spacecraft mechanisms.

PFPE fluid lubricants also offer the advantage that their tribological performance in vacuum is not compromised by prior operation in moist air, as displayed by sputtered MoS$_2$. In fact, lifetimes of PFPE fluids (and multiply alkylated cyclopentanes) are shown to be extended in moist air in comparison to vacuum [13, 14].

Hybrid Lubrication

The suggestion is occasionally made within the space mechanisms community that a form of hybrid lubrication may circumvent the restriction on in-air operation of MoS$_2$, through the application of a controlled quantity of PFPE fluid lubricant to a component lubricated in the conventional way with sputtered MoS$_2$, thus 'protecting' the MoS$_2$ from the moist environment. The suggestion states that the fluid lubricant will provide low friction during operation in moist air and will subsequently be lost (either through evaporation or tribo-chemical degradation depending upon the fluid) under vacuum, allowing the

(hopefully) uncompromised MoS$_2$ film to provide low friction and long life for the remainder of operation under vacuum.

The potential advantage of synergistic behavior between the fluid and solid lubricating constituents also exists, where performance of the whole lubrication solution is greater than the sum of its parts. Essentially this occurs where the presence of one lubricant constituent prolongs the operational performance of the other, and vice-versa. This behavior may occur on a physical and/or chemical level.

This paper details the recent testing campaigns at ESTL to understand and characterize the potential of hybrid lubrication of PFPE fluids and sputtered MoS$_2$ with respect to the advantages stated above, performed at both tribometer and component level. These testing campaigns shall be discussed individually.

Spiral Orbit Tribometer Experimental Campaign

SOT Phase One – Feasibility Study
A feasibility demonstration of hybrid lubrication was performed using a Spiral Orbit Tribometer (SOT). The SOT is essentially a rolling tribometer, where a solid or low volume of fluid lubricant can be assessed under representative conditions of an angular contact bearing operating within the boundary regime. Details of the SOT are described elsewhere [15].

SOT test balls of 52100 steel were first lubricated with sputtered MoS$_2$. These same test balls were then lubricated with ~50 µg of PFPE oil Fomblin Z25 via solvent plating, providing hybrid Z25/MoS$_2$ lubrication. Hybrid lubricated balls were inserted into the SOT test chamber and rotated in moist air for a defined duration and subsequently under high vacuum to failure. Test conditions were 2.25-GPa peak contact stress, 100-RPM rotation speed, and 23°C. Failure of the hybrid lubricant was defined as an increase in friction coefficient to ≥ 0.28.

Initial feasibility results demonstrated encouraging behavior, with indications that a small volume of fluid lubricant acts to protect the sputtered MoS$_2$ from elevated wear rates in moist air, and some degree of extension in life is observed under vacuum. In addition, the friction coefficient of the hybrid lubrication was found as 0.02 (Figure 3), identical to the value for MoS$_2$ alone in vacuum [16]. Given that Z25 provides a friction coefficient of 0.1 under vacuum on the SOT, we can say with some confidence that the MoS$_2$ was providing lubrication at this time. A more detailed account of this initial feasibility study is provided elsewhere [17].
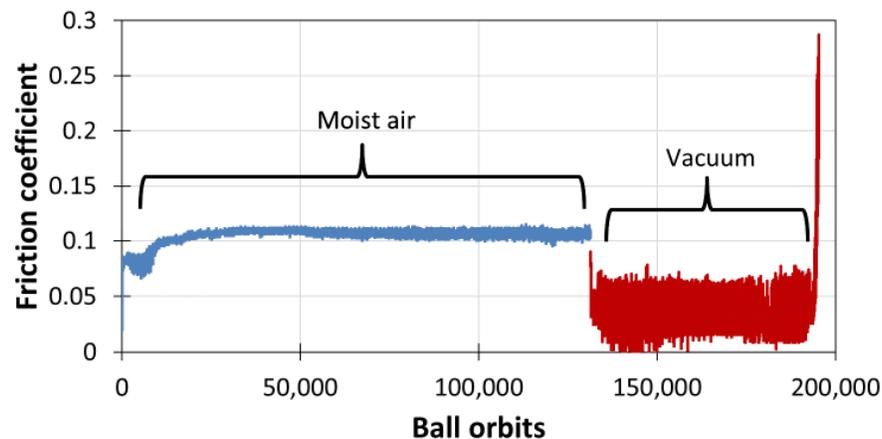
Figure 3. Hybrid Z25/MoS$_2$ rolling in air (blue) and vacuum (red). Elevated frictional noise during vacuum running is an artifact introduced by the analysis software and is not real

Given the encouraging performance of the hybrid Z25/MoS$_2$ lubrication, additional SOT tests were performed to further investigate this behavior.

SOT Phase Two – Detailed SOT Study
Phase Two of SOT testing was performed in an equivalent manner to Phase One, with the following alterations.

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

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

- In-air running durations defined as percentages of in-vacuum MoS$_2$ life.

Prior to hybrid lubrication testing, the lifetimes of MoS$_2$ under vacuum at the above contact stress was assessed. From these lifetimes the in-air durations required for the Phase Two hybrid testing can be calculated. The required in-air running durations are given in Table 1, with L_x being the in-vacuum MoS$_2$ life at a given contact stress S_X.

Table 1. Required in-air running for SOT Phase One

Peak stress	S$_1$ (3.00 GPa)	S$_2$ (2.25 GPa)	S$_3$ (3.00 GPa)
Required in-air operation	0.0005 L_1	0.0005 L_2	0.0005 L_3
	0.005 L_1	0.005 L_2	0.005 L_3
	0.05 L_1	0.05 L_2	0.05 L_3
	0.5 L_1	0.5 L_2	0.5 L_3

Results of Phase Two SOT testing on hybrid lubrication are presented in Table 2.

Except for S1-D, all tests passed the in-air duration of running without displaying evidence of lubricant failure, with S1-D displaying failure after operating for 98% of the required orbits. These results allow us to state that the application of ~50 µg of 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. Vacuum lifetimes show that in almost all cases vacuum lifetimes were extended in comparison to sputtered MoS$_2$ alone.

Table 2. Moist-air and subsequent normalized vacuum lifetimes of hybrid Z25/MoS$_2$ lubricated SOT tests

ID	Peak Stress (GPa)	Required in-air operation	In-air Pass/Fail	Subsequent vacuum life / MoS$_2$-only life	Total life / predicted vacuum life
S1-A	3.00	0.0005 L_1	Pass	2.199 L_1	2.176
S1-B	3.00	0.005 L_1	Pass	1.501 L_1	1.484
S1-C	3.00	0.05 L_1	Pass	2.106 L_1	2.130
S1-D	3.00	0.5 L_1	Fail (98%)	0.000 L_1	0.486
S2-A	2.25	0.0005 L_2	Pass	1.521 L_2	1.463
S2-B	2.25	0.005 L_2	Pass	3.644 L_2	3.550
S2-C	2.25	0.05 L_2	Pass	2.456 L_2	2.399
S2-D	2.25	0.5 L_2	Pass	0.002 L_2	0.482
S3-A	1.50	0.0005 L_3	Pass	1.210 L_3	1.103
S3-B	1.50	0.005 L_3	Pass	4.686 L_3	4.291
S3-C	1.50	0.05 L_3	Pass	2.976 L_3	2.833
S3-D	1.50	0.5 L_3	Pass	2.392 L_3	2.648
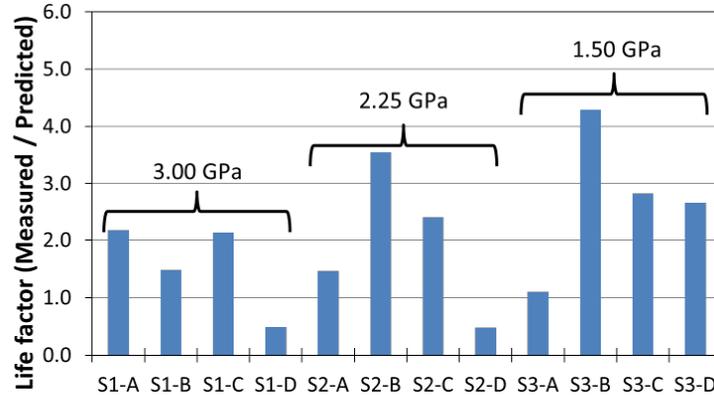
Figure 4. Measured lifetimes of hybrid Z25/MoS₂ lubrication as factor of predicted life

Using the predictions of fluid lifetimes taken from [16], 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 SOT tests (except for S1-D and S2-D), the total hybrid lubrication lifetime is longer than that of the individual lubricant constituents (Figure 4). That is to say there is a synergistic lubrication effect.

Life of PFPE/MoS₂ lubrication > Life of PFPE lubrication + Life of MoS₂ lubrication

In addition, the steady state friction coefficient in during the vacuum stage of testing was significantly below 0.1 in all cases. This suggests that the MoS_2 was providing the lubrication during the in-vacuo stage of all hybrid tests throughout the extended life.

Post-test inspections of the test pieces showed mixed regions of MoS_2 debris captured within the degraded PFPE oil, displaced to the edges of the ball tracks. No dusting of loose MoS_2 debris was observed, in contrast to the typical post-test observations of MoS_2 alone on the SOT.

Pin-on-Disc Tribometer Experimental Campaign

PoD Phase One – High volume fluid lubrication
To assess the potential for hybrid lubrication in a pure sliding environment a series of Pin-on-Disc (PoD) tests were performed under the following test conditions.

- Discs lubricated with Braycote 601EF grease only.

- Discs lubricated with sputtered MoS_2 only.

- Disc lubricated with both sputtered MoS_2 and Braycote 601EF. Where possible the same test disc was used as for the MoS_2-only test.

Grease lubricant was applied following standard ESTL procedure recommending 10 mg/cm^2 to a disc of surface area 4.4 cm^2, amounting to 44 mg onto the surface of each test disc. The grease was applied by syringe and then distributed using a ISO class-5 wipe to the necessary volume of grease, measured using a microbalance.

All tests were performed under vacuum, 0.6 ms^{-1} sliding speed, on standard 52100 steel PoD test pieces. Contact stress and temperature was varied as detailed below. Failure of the lubricant was defined as an increase in friction coefficient to ≥ 0.3. Friction and lifetime results are provided in Table 3.

Table 3. Tribological behaviors of PoD test campaign of hybrid Braycote 601EF/MoS₂ lubrication. Lifetimes are normalized to behavior of MoS₂-only, RT, 1500 MPa

Test ID	Lubricant	Peak stress (MPa)	Temp	Normalized Lifetime	Friction coefficient
PoD.A1	Braycote 601EF	850	23	0.001	0.20
PoD.B1	MoS$_2$	850	23	8.591	0.03
PoD.C1	Braycote 601EF/MoS$_2$	850	23	0.253	0.06
PoD.A2	Braycote 601EF	1500	23	0.005	0.15
PoD.B2	MoS$_2$	1500	23	1.000	0.02
PoD.C2	Braycote 601EF/MoS$_2$	1500	23	0.272	0.04
PoD.A3	Braycote 601EF	2000	23	0.000	0.17
PoD.B3	MoS$_2$	2000	23	0.060	0.06
PoD.C3	Braycote 601EF/MoS$_2$	2000	23	0.070	0.04
PoD.A4	Braycote 601EF	850	120	0.004	0.26
PoD.B4	MoS$_2$	850	120	1.867	0.10
PoD.C4	Braycote 601EF/MoS$_2$	850	120	0.413	0.15
PoD.A5	Braycote 601EF	1500	120	0.012	0.26
PoD.B5	MoS$_2$	1500	120	0.194	0.06
PoD.C5	Braycote 601EF/MoS$_2$	1500	120	0.008	0.16
PoD.A6	Braycote 601EF	2000	120	0.014	0.26
PoD.B6	MoS$_2$	2000	120	0.007	0.06
PoD.C6	Braycote 601EF/MoS$_2$	2000	120	0.039	0.04

Considering hybrid lubrication, the Braycote 601EF/MoS₂ tests are disappointing in comparison to the SOT testing campaign, with the following conclusions drawn (demonstrated in Figure 5).

- The addition of MoS₂ to a grease lubricated surfaces will increase the lifetime significantly.

- The addition of grease to MoS₂ lubricated surfaces will not increase the lifetime significantly.
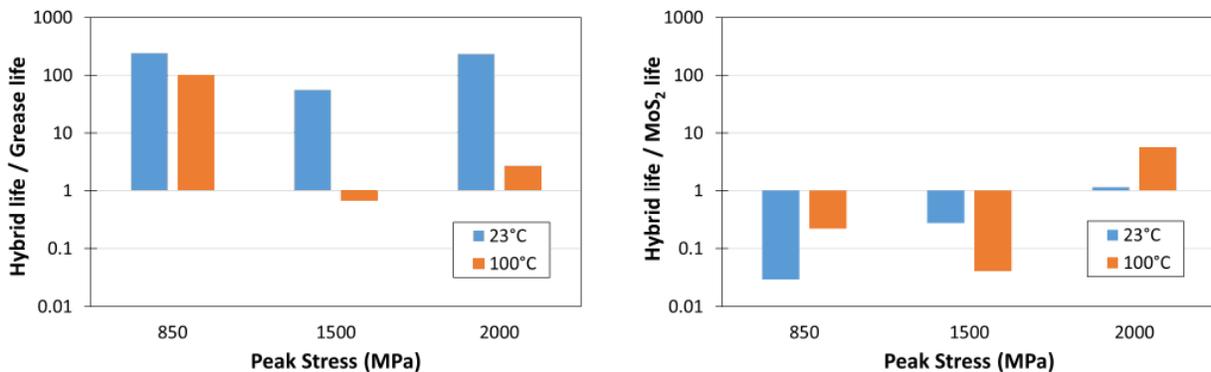
Figure 5. Hybrid Braycote 601EF/MoS₂ lifetimes as a factor of Braycote 601EF-only (left) and sputtered MoS₂ only (right) lifetimes

In addition, the friction coefficient of the Braycote 601EF/MoS₂ tests is, in most cases, elevated in comparison to MoS₂. Other observations are made from the test data above.

- The test results demonstrate that under vacuum conditions the lifetime of MoS₂ decreases with increasing temperature and contact stress. This is in line with previous data and expectations.

- As a general guide the lifetime of grease lubrication under vacuum increases with increasing temperature. This is seemingly counter-intuitive and not in line with our expectations given the known dependence of PFPE lubricant degradation and tribological lifetime upon temperature [11].

It is clear that the grease lifetimes in these PoD tests are dictated by some physical limitations of the lubricant, rather than tribo-chemical degradation. This can be conclusively demonstrated using a RGA system, where no evidence of residual gas by-products of the PFPE degradation were observed in any of the above tests, including those with hybrid Braycote 601EF/MoS_2 lubrication. Failure of the grease is therefore likely caused by the physical displacement of the fluid lubricant away from the contact zone, in contrast to the SOT tests, which may also contribute the poor hybrid performance during these PoD tests.

The improved performance of the grease at 200°C also leads to the suggestion that lower viscosity of the fluid selected may be helpful for the hybrid effect, due to an improved rate of lubricant flow into the contact zones. This can be demonstrated through repeating of a small selection of tests detailed in Table 3, utilizing the PFPE oil Fomblin Z25 as a replacement to Braycote 601EF. All other test conditions including the application method were held identical.

Results demonstrated that the Fomblin Z25 oil performed significantly better at PoD level than Braycote 601EF, with RGA data showing shear-induced tribo-chemical degradation of the lubricant under test. Hybrid Z25/MoS_2 tests were also improved in comparison to Braycote 601EF/MoS_2 (Table 4) but showed elevated test variability.

Table 4. Performance of hybrid Z25/MoS_2 under vacuum on PoD.
Tests performed at RT, 1500 MPa

Normalized lifetime		
Fomblin Z25	MoS_2	Hybrid Z25/MoS_2
2.723	1.000	0.365

Whilst Fomblin Z25 performs better than Braycote 601EF as a hybrid lubricant, it is clear that the highly encouraging performance observed at SOT is not replicated in the sliding environment of the PoD. This is potentially due to the significantly lower fluid volumes, and/or fluid film thicknesses, of the SOT testing campaign in comparison to the PoD tests (for context, the volume of fluid in a typical SOT test is three orders of magnitude less than the PoD). To explore this possibility, a second phase of PoD testing was performed utilizing a reduced lubricant volume, applied following the solvent-plating technique employed on the SOT onto MoS_2-lubricated discs.

PoD Phase Two – Low volume fluid lubrication
Three levels of Fomblin Z25 lubrication were achieved for low volume fluid lubrication tests.

- Discs lubricated with sputtered MoS_2 exposed to a PFPE solvent bath – To confirm that the solvent used for the oil plating technique (PF5060) does not adversely influence the tribological performance of the solid lubricant.

- Discs lubricated with sputtered MoS_2 and low mass of Fomblin Z25 (~40 µg).

- Discs lubricated with sputtered MoS_2 and high mass of Fomblin Z25 (~400 µg). It should be made clear that this 'higher' mass is still two orders of magnitude less than the recommended amount for a fluid lubricated component.

All tests were performed under vacuum, 0.6 ms^{-1} sliding speed, 900-MPa peak contact stress, on standard 52100 steel test components. Multiple repeat tests were performed under each condition. Failure of the lubricant was defined as an increase in friction coefficient to ≥ 0.3. Mean friction and normalized lifetime results are presented in Table 5.

Table 5. Performance of varying levels of hybrid Z25/MoS$_2$ lubrication under vacuum on PoD. Tests performed at 900 MPa, 23°C. Lifetimes normalized to MoS$_2$-only life at room temperature

Lubricant	Life / MoS$_2$ life (RT)	Friction coefficient
MoS$_2$ only	1.000	0.03
MoS$_2$ with solvent bath	0.998	0.03
MoS$_2$ with Z25 (~40 µg)	1.271	0.03
MoS$_2$ with Z25 (~400 µg)	1.848	0.03

Results demonstrate no tribological influence from the solvent bath, demonstrating that a PF5060 solvent immersion does not influence the lifetime of the sputtered MoS$_2$ film in a measurable manner.

Hybrid lubricated tests show an increase in sliding lifetime in comparison to MoS$_2$, with the higher mass of the applied fluid lubricant increasing the success of the hybrid lubrication (Figure 6). In addition, the friction coefficient was not compromised by the addition of the oil onto the sputtered MoS$_2$.
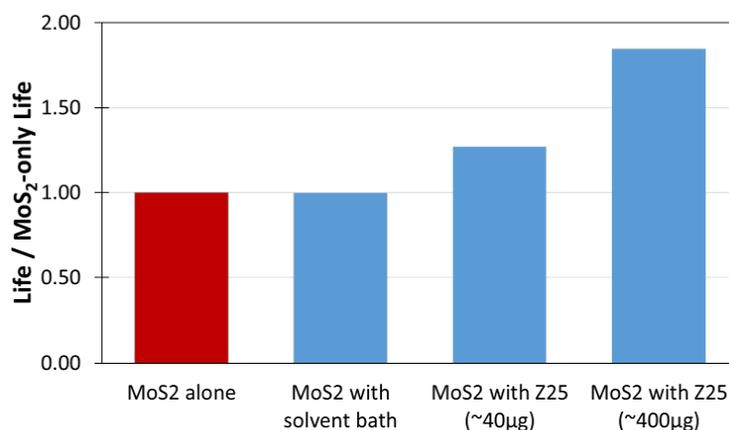
Figure 6. Normalized performance of Z25/MoS$_2$ lubrication under vacuum

The above tests were then repeated under vacuum at elevated temperature (200°C), displaying greater sliding lifetimes than those achieved at room temperature, again with no increase in friction coefficient (see Table 6). This is a surprising observation given that the degradation lifetimes of both the sputtered MoS$_2$ films and Fomblin Z25 oil are known to dependent upon operating temperature.

Table 6. Comparison of hybrid Z25/MoS$_2$ lubrication at room temperature and 200°C under vacuum

Lubricant	Temp (°C)	Life / MoS$_2$-only life (RT)	Friction coefficient
MoS$_2$ with Z25 (~400 µg)	23	1.848	0.03
MoS$_2$ with Z25 (~400 µg)	200	2.261	0.03

Using data from the Phase One PoD testing (Table 3) we observe that the sliding lifetime of sputtered MoS$_2$ films under vacuum at 120°C is reduced to ~10 – 20% of the lifetime achieved at room temperature. Although the nature of this reduction in life is not fully known (i.e. to what level the increased reaction between oxygen/moisture with the MoS$_2$ film, and the softening of the substrate steel factor into this reduction), and the dataset is not sufficient enough to produce a confident life vs. temperature relationship, it is clear that the sliding life of MoS$_2$ at 200°C is predicted to be <10 – 20% of the room temperature lifetime for a given contact stress.

A similar relationship exists for Fomblin Z25, where the rolling lifetime of the fluid lubricant at 100°C is reduced to ~30 – 50% of the lifetime achieved at room temperature on the SOT, due to an increase in the

degradation rate of the PFPE [11]. It is also known that, when not experiencing shearing, spontaneous degradation of Fomblin Z25 occurs between 190 – 250°C [11]. Together these indicate that the lifetime of the oil should be severely reduced when operating at 200°C (assuming the lifetime is dictated by the chemical degradation of the fluid).

It is therefore suggested that the increased lifetime of the hybrid $Z25/MoS_2$ at elevated temperature is again a result of temperature-related viscosity changes within the fluid lubricant, allowing 'reflow' of fluid into the running track to occur more easily. This is highly surprising given the low volumes of lubricant employed during these low fluid volume PoD tests, the fact that the low coefficient of friction suggests that the lubrication between the contacts is still predominantly provided by the MoS_2 film, and the extent of the predicted MoS_2 lifetime reduction. By whatever mechanism hybrid lubrication occurs (see below), it is clearly influenced by operating temperature.

Spur Gear Experimental Campaign

In parallel to the SOT and PoD test programs, a small series of spur gear tests was performed to determine if the hybrid lubrication effect could be reproduced at component level. These gear tests form part of an ongoing campaign to evaluate and characterize the performance of solid, fluid, and hybrid lubricated gears under vacuum and are reported in detail elsewhere [18].

Selected gears were BS4582 class B (DIN867 Q7) (hobbed) precision, manufactured in 17-4PH steel (Condition A) without further surface treatment. Gears were 0.5 modulus, the pinion having 40 teeth (face width 5 mm) and the wheel 120 teeth (face width 2.5 mm). It should be noted that the gears do not have a "hunting tooth" ratio, such that the same teeth contact repeatedly in each revolution.

Whilst the 17-4PH material was selected for similarity with known applications, its relatively low hardness was chosen to permit both a comparison with earlier work on non-hardened steels and a subsequent evaluation of the beneficial impact of more typically hard surface treatments (Condition A results in a minimum hardness of 35 HRC (~333 Hv)). The relatively low gear precision class and surface finish (hobbed rather than ground gears) were selected for reasons of similarity with earlier test campaigns, and to permit these production factors to become a variable in the wider context of the larger test campaign. A unique gear set was employed for each test.

Spur gear tests were performed under vacuum at 23°C, with a preload of 7.5 Nm, equivalent to a peak stress of 1000 MPa. Sputtered MoS_2, Braycote 601EF grease, and hybrid Braycote 601EF/MoS_2 provided the lubrication, with the recommended 10-mg/cm^2 volume of grease applied directly to the gear teeth. Rotation speed was 100 RPM for the fluid and hybrid lubricated gears, 50 RPM for the MoS_2 lubricated gears. Failure of each test was defined as an increase of the long-term torque measurement to 1.25x the steady-state value. The steady-state torque was determined by averaging the torque after an initial running-in period.

Given these selected conditions and ratio we might expect these results to provide a demanding (even perhaps a kind of "worst") test case for the lubricants used.
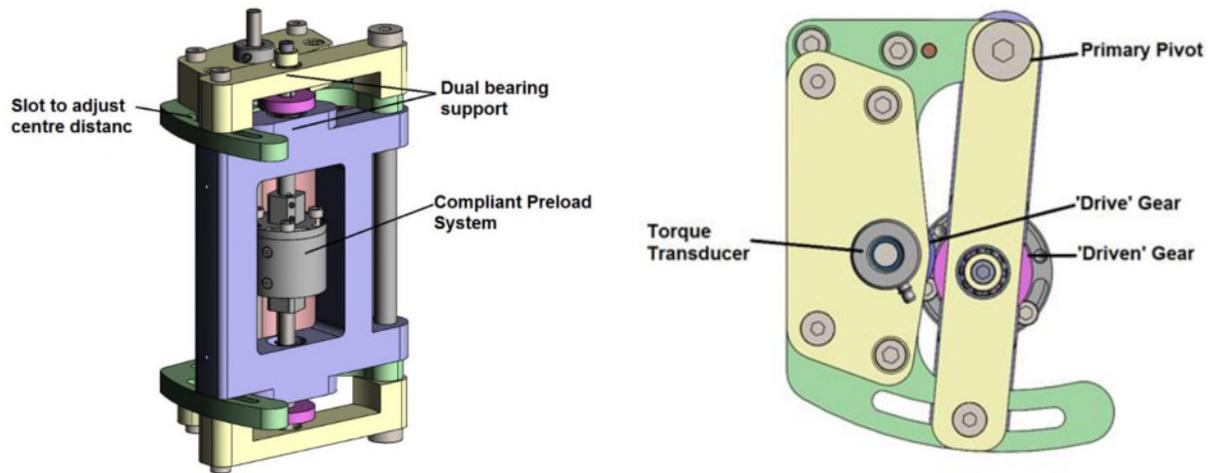
Figure 7. Conventional gear test setup for ESTL's miniature gear/pinion testing rig

Tests were performed using ESTL's miniature gear/pinion testing rig, utilizing the 4-square principle with compliant gear preloading (Figure 7). The test gears are supported on both sides by rolling element bearings to avoid any misalignment/stiffness issues. The compliant preload system maintains axial alignment between the gear shafts by supporting the shafts with plain bushings and locating the two shafts with a recess and boss. The torque was assessed via a transducer.
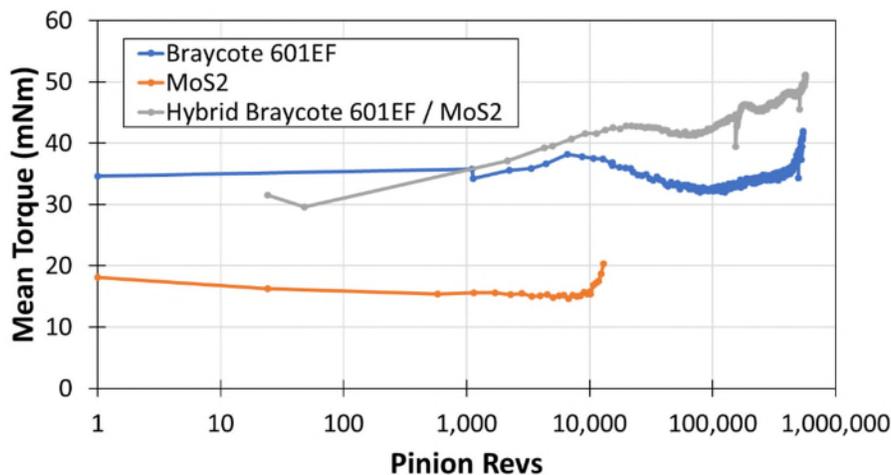
Figure 8. Mean torque behavior of spur gear tests (parasitic torques removed)

Results from the gear testing was unfortunately inconclusive. Whilst true that the hybrid lubricated test lasted longer than the sum of the lifetimes for the solid and fluid lubricated gears individually, this lifetime extension is extremely minimal and likely falls within the margin of error. The elevated torque value is also disappointing, suggesting that for the hybrid lubricated gears the lubrication was predominantly being provided by the fluid, and not the MoS_2.

Table 7. Summary of spur gear test behavior

Lubricant	Lifetime (revs)	Steady-state torque (mNm)	End-of-life torque (mNm)
Braycote 601EF	545,545	33.4	41.8
MoS_2	12,525	15.2	19.0
Braycote 601EF / MoS_2	567,490	42.3	52.9

Post-test inspection of gears showed clear evidence of lubricant failure, with varying degrees of scuffing and pitting of the teeth leading to metallic wear.

It is recognized that despite the similar fluid lubricant volumes employed, significant differences exist between the gear and tribometer testing which may influence the lubricant behavior, and hence the success of hybrid lubrication. The PoD tribometer is an assessment of the lubricant behavior in a pure sliding regime only, whilst the motion on the SOT is predominantly rolling. In contrast, the spur gears experience a combination of rolling motion during gear meshing at the pitch point, combined with sliding as the mesh moves from the pitch point. This meshing action of the gears can act to redistribute the fluid lubricant in the contact, bringing about an extension in life for the fluid lubricated gears, and also potentially altering the behavior in the hybrid lubricated case in a way not comparable to the PoD (or SOT).

Discussion of Results and Theory of Hybrid Lubrication

From these experimental test results the commonality can be drawn that the key factor in the success of hybrid PFPE/MoS_2 lubrication appears to the mass and/or viscosity of the fluid layer applied to the sputtered MoS_2. This observation shall now be discussed in the context of three proposed models for the hybrid lubricating behavior.

It has been shown that hybrid lubrication has the potential for two attractive tribological behaviors.

- Hybrid lubrication can act to protect the sputtered MoS_2 film from elevated degradation/wear/oxidation when operating in moist air.

- Hybrid lubrication provides a vacuum lifetime greater than the vacuum lifetime of its constituent parts (i.e. it is synergistic), with no subsequent increase in friction/torque.

The mechanism by which these behaviors arise is not clear, but three general models are proposed.

- **Model 1) Protection of PFPE fluid from degradation** – It is known that the lifetime of a PFPE lubricant can be extended through physical protection of the fluid from reaction with the substrate steel, in this instance provided by an MoS_2 film. However, if this model were to dominate we would expect to observe a higher friction coefficient, more akin to fluid lubrication than MoS_2, when observing an extension in life. This is not the case during SOT and PoD testing.

- **Model 2) Protection of sputtered MoS_2 film from degradation** – An alternative model has been proposed whereby the layer of PFPE fluid upon the surface of the MoS_2 film acts to protect the solid lubricant from reacting with the environment, prolonging the wear life of the MoS_2. This model is attractive to explain the protection seen by the MoS_2 film when rolling in moist air but is less applicable to explain the extension of life under vacuum, where the presence of moisture/oxygen is severely reduced.

- **Model 3) MoS_2 transfer film establishment** – It is understood that the production of a 3rd-body transfer film is vital for successful lubrication of MoS_2 [19]. Given that this 3rd-body transfer film is produced from what is essentially 'wear debris' of the MoS_2, it is proposed that the physical presence of a viscous fluid within and around the contact zone helps to retain this MoS_2 debris, and is advantageous to the formation/protection of a 3rd-body layer.

It is clear that the success of hybrid lubrication is sensitive to the film thickness of the applied oil (or ratio of fluid film thickness to MoS_2 film thickness). If we assume an equal distribution of oil, the fluid film thickness achieved during the Phase Two PoD tests ranged from 0.05 to 0.55 µm, applied onto a ~1-µm sputtered MoS_2 film. SOT tests described above employed a similar fluid film thickness of ~0.17 µm over

a 0.5-µm MoS$_2$ film, assuming fluid distribution over the ball only. These test cases demonstrate an extension in life provided by the hybrid lubrication, where the fluid film thickness is 5-50% of the originally deposited MoS$_2$ film.

In instances where a significantly larger mass of fluid lubricant is applied to a sputtered MoS$_2$ film, typical of the mass applied to a grease-only lubricated component (e.g. Phase One PoD tests and spur gear tests), the fluid film thickness is closer to 50 µm, and the positive effect of hybrid lubrication is essentially lost. This demonstrates that a threshold may exist above which the application of additional fluid lubricant onto the surface of the sputtered MoS$_2$ film is detrimental to the performance, rather than an improvement.

Figures 9 and 10 suggests a mechanism by which this process may occurs, whereby a thicker fluid film is proposed to dislodge the lightly adhered MoS$_2$ transfer film from the contact via viscous drag effects (assuming Model 3). This threshold for fluid lubricant volume would appear to exist at a significantly lower volume than the amount prescribed by the standard grease lubrication procedure.
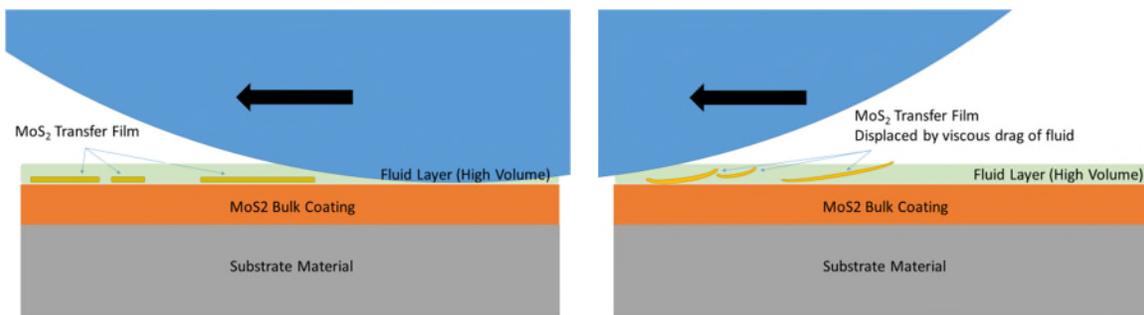
Figure 9. MoS$_2$ transfer film formation (3rd body) of high fluid volume hybrid lubrication
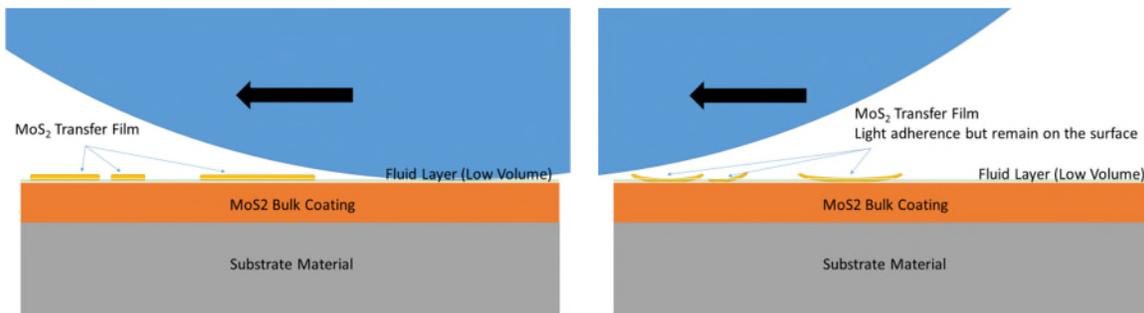
Figure 10. MoS$_2$ transfer film formation (3rd body) of low fluid volume hybrid lubrication

The improved performance of hybrid lubrication at elevated temperatures suggests that the viscosity of fluid also is a factor in determining the success of hybrid lubrication, with the reduced viscosity of the Fomblin Z25 at 200°C potentially allowing for greater re-flow of the lubricant within the contact zones, producing longer life. It is known that the physical lifetime of Fomblin Z25 is longer than the more viscous grease Braycote 601EF when assessed on a PoD tribometer, despite their tribo-chemical degradation lives behaviors identical [16], due to reflow effects. Given the above it is likely that a relationship exists whereby the success of hybrid lubrication is governed by the lubricant film thickness, and the physical properties (i.e. viscosity) of the fluid itself (as well, potentially, as the surface roughness of the substrate).

The true mechanism of hybrid lubrication of PFPE/MoS$_2$ is likely to be a combination of these above models.

Non-PFPE fluids in Hybrid Lubrication

The above hypothesis states that the success of hybrid lubrication depends primarily upon the ability of the fluid constituent to retain the 3rd-body MoS_2 transfer film within the contact zone, a phenomenon related to related to film thickness (or ratio of film thickness to MoS_2 thickness) and viscosity of the fluid. If this is the case, the tribological properties the fluid itself may be second order effects compared to the physical properties of the fluid (vapor pressure, viscosity), potentially allowing for the selection of more favorable fluids to achieve hybrid lubrication.

For instance, the lower viscosity offered by the MAC fluid Nye 2001a may prove advantageous in a hybrid regime. However, this fluid is characterized by a relatively higher vapor pressure, and so potentially is not so attractive for elevated temperature operations. In such cases the ultra-low vapor pressure fluid Fomblin Z60 may be a viable candidate, despite its poorer tribo-chemical lifetime [16].

Table 8. Vapor pressures and viscosities for potential candidate fluids for hybrid lubrication

	Fomblin Z25	Nye 2001a	Fomblin Z60
Vapor pressure (mbar)			
20°C	2.13×10^{-13}	1.53×10^{-11}	1.47×10^{-16}
38°C	4.13×10^{-12}	1.34×10^{-10}	7.33×10^{-15}
100°C	3.73×10^{-09}	4.74×10^{-08}	5.47×10^{-11}
Kinematic viscosity (cSt)			
20°C	263	297	600
40°C	157	108	355
100°C	49	14.6	98
Viscosity index	358	137	360

An SOT testing campaign to explore the potential of other fluids in a hybrid lubrication regime is planned to take place at ESTL in 2018.

Conclusions

The work presented here (and elsewhere by ESTL) demonstrates the potential for hybrid lubrication, but also the limitations. Under the right conditions the lifetime of a sputtered MoS_2 film can be significantly extended under vacuum and protected from a reasonable degree of moist-air operation, with no consequential increase in friction coefficient. In other cases, however, attempts at hybrid lubrication have resulted only in the loss of the good tribological properties of the sputtered MoS_2, resulting in shorter operational lifetimes.

These results would suggest that the physical properties of the applied fluid lubricant are potentially more important to the success of hybrid lubrication than their tribo-chemical properties (which more dictate their performance alone). For successful hybrid lubrication a layer of fluid lubricant of sufficient volume is required to protect the MoS_2, but this layer must be sufficiently thin, mobile and/or fluid to ensure it does not disrupt the formation of the MoS_2 transfer film or provide a fully fluid lubrication regime over the MoS_2 (i.e. the friction/torque retains the characteristic of solid lubrication).

Component and tribometer level testing activities are continuing at ESTL to verify this hypothesis, and to demonstrate the potential of hybrid lubrication at angular contact bearing level.

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